Materials Horizons: From Nature to Nanomaterials

Vimal Katiyar
Tabli Ghosh

Nanotechnology in Edible Food Packaging

Food Preservation Practices for a Sustainable Future

Springer

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Preface

The current book demonstrates the systematic overview of nanotechnology-assisted edible food packaging with an in-depth discussion on biopolymeric nanostructures, inorganic nanostructures, associated fabrication strategies of versatile nanomaterials, and edible films and coatings, multiphase nanosystems including biopolymer nanocomposite, nanoencapsulation, and nanoemulsions for edible packaging, and other magnificent areas. In line with recent advances in edible food packaging assisted by nanosystem, the multifarious packaging technology including bioactive compounds aided edible packaging, nanosystem-assisted antimicrobial edible packaging, secondary packaging, modified atmospheric packaging, controlled atmospheric packaging, smart packaging, and active packaging has been outlined in this book. To provide a comprehensive understanding directing the learners and researcher of the edible food packaging field, the book also focuses to deliberate the basics of edible food packaging including a selection of suitable biopolymers for a specific category of food products, characterization techniques, and experimental procedures associated with the processing of nanostructures, nanosystem-aided edible food packaging and further discusses the characterization approaches to study the storage life of edible food-packaged products. Specifically, the fundamental mechanism of edible food packaging facilitated by nanomaterials has a potential demand in present-day scenario, and for technological progression, upcoming consequences, and for ultimate integration into industrial sector for being sustainably worthy. Based on this, the book collection has been emphasized for the use of nanotechnology in edible films or coating directing the researcher and audience of this field to accumulate a wide-ranging spectrum of understanding in the remarkable field, which is a developing area in the current global food packaging market. More precisely, the present book collection consists of four parts and distinctly fifteen chapters that are focused to discuss targeted biopolymeric and inorganic nanostructured materials in edible food packaging; multiphase nanosystems in edible food packaging; advances in edible food packaging and targeted food products and related characterization techniques. To move with edible food packaging in a strategical way and delivering the versatile health beneficial food products to end users has become essential to combat the implications of petroleum-derived packaging materials and to reduce food loss globally.

Chapter 1 begins by addressing several features of edible food packaging (edible films or edible coating) which is considered as an eco-friendly food preservation technique and delivers biodegradable packaging materials similar to the food products and acts as an alternative to available conventional and sustainable non-edible packaging materials. The chapter deciphers the current need of developing edible food packaging as a synergistic approach along with other food preservation techniques for highly perishable food products as an alternative to the available packaging materials. In this chapter, the global market analysis of edible packaging including status on targeted materials, end-users, and key players in the edible packaging market has been discussed. The available commercialized edible packaging with existing noteworthy traits and ongoing research and development of edible films and coatings at a lab scale has been discussed elaborately. The chapter further provides a brief overview and schematic illustration of the current book *Nanotechnology in Edible Food Packaging* for several sectors such as biopolymeric nanostructured materials, synthesis strategies, multiphase nanocarriers, recent advances, and others.

Chapter 2 introduces the various biomaterials including polysaccharide, proteins and lipids, and others which are targeted to be used in the field of edible food packaging. The use of eatable material such as fruit peel and renewable waste materials in developing edible coating can be a strategic way to convert "waste into wealth." The chapter also introduces the use of existing nanosystem-based biomaterials related to biopolymeric nanomaterials (cellulose, chitosan, starch, protein, and lipid-based nanostructured materials) and inorganic nanomaterials, polymer nanocomposite, nanoencapsulation, and others for developing tailored-made edible food packaging. The fabrication methodologies of edible films and coatings have also been discussed in Chap. 2. The synthesis strategies in developing edible coating include dip coating, spraying-based edible coating, electrospraying, fluidized bed drying, and others, whereas the synthesis strategies for edible films are solution casting and extrusion techniques and others, which has been discussed elaborately in this chapter.

Chapter 3 demonstrates the fabrication of cellulose nanostructured materials assisted edible food packaging in terms of edible films and coating for several food products. The chapter also provides a brief overview of the application of cellulose and nanocellulosic materials in food applications including the fabrication of tailored edible food packaging. Several types of nanocellulosic materials are extracted using various techniques such as acid hydrolysis, mechanical methods, enzymatic hydrolysis, ionic liquid treatments, steam explosion, supercritical water hydrolysis, and others, which have been focused to discuss in this chapter. The chapter also focuses to detail the several traits of nanocellulose-aided edible food packaging materials in terms of barrier property, mechanical property, thermal property, optical property, and others. The use of nanocellulose-based edible food packaging with the storage study and tunable packaging property has also been discussed based on the past, present, and future perspectives.

Chapter 4 focuses on the use of chitosan-based nanostructured materials as a component in developing edible films and coatings for food products. The chapter details the various fabrication approaches for chitosan nanomaterials in the form of nanocrystals, nanofibers, nanomaterials, and others using ionotropic gelation, microemulsion, emulsification solvent diffusion, polyelectrolyte complexes, reverse micellar, solvent evaporation, co-precipitation, complex coacervation techniques. The several features of chitosan nanoparticles such as biocompatibility, antimicrobial property, antioxidant property, non-toxicity, and others have made it a noteworthy candidate in edible food packaging. In this chapter, the different applied areas of chitosan nanostructures aided edible films and coatings for fruits and vegetables, fish products, and other food sectors have been demonstrated further.

Chapter 5 begins by discussing the components, categories, and available sources of starch. The starch is an important component in the various food systems and edible food packaging, which is discussed elaborately in the current chapter. The chapter also aims to discuss the global industrial starch market based on region, source, application, and industry. The fabrication of starch nanomaterials for edible food packaging can be obtained using acid hydrolysis and enzymatic hydrolysis, regeneration, and cross-linking, and mechanical treatment/microfluidization, which will be discussed in this chapter. The modification strategies of starch and its derivatives for edible food packaging including chemical modification, physical modification, enzymatic modification, development of biocomposites and blends, and addition of bioactive compounds has been detailed in the present chapter. Additionally, the use of various forms of nanostarch materials for improved product life has also been discussed at a laboratory-scale level.

In Chap. 6, the various features of available protein-based nanostructured materials to be used in edible food packaging has been discussed elaborately. The protein and associated nanomaterials having various noteworthy attributes such as hydrophobicity, improved mechanical property, antibacterial properties are targeted to be used in developing edible food packaging materials. The chapter also discusses the use of protein-based materials for both solid (fresh-cut fruits, beans, and cashew nuts) and liquid food products (vegetable oils). The addition of protein-based nanostructured materials in edible food packaging can improve the properties in terms of aggregation and shelf life improvement, which are noteworthy traits.

Chapter 7 focuses on the use of various lipid-based nanostructured materials in edible food packaging including nanoemulsion, nanoliposomes, nanostructured lipid carriers, and others. The application of lipid nanostructured materials in edible food packaging for improved product life and quality of food products such as microbiological property and other food properties has been detailed in this chapter. The chapter also provides an overview about the several lipids based materials such as synthetic and natural waxes, fatty acids and alcohols, lacs, acetylated glycerides, essential oils and extracts, and cocoa-based compounds and their derivatives for targeted lipid-assisted edible food packaging.

Chapter 8 focuses to discuss about the use of inorganic nanomaterials in edible food packaging. The targeted inorganic nanomaterials in edible food packaging including titanium dioxide, silicon dioxide, zinc oxide, iron oxides, and others have been demonstrated in this chapter. The inorganic nanomaterials are used in addition to polymer composites to improve the efficiency of the packaging materials in terms of fortified and nutraceutical-added food products.

Chapter 9 focuses to discuss opportunities and application of biopolymer nanocomposites in edible food packaging. The chapter provides an overview of using biopolymer nanocomposite in edible food packaging, various food sectors, and others. The chapter also discusses various features of biopolymer composites such as physical properties, barrier properties, functional attributes, antimicrobial properties, and surface properties to be used in edible food packaging including the recent advances of biopolymer nanocomposite in edible food packaging. The modification strategies of biopolymer nanocomposites including binding agents, plasticizers, crosslinkers, functional additives, and compatibilizers have been detailed in this chapter. Additionally, effective properties and benefits of using biopolymer nanocomposite in edible food packaging have been detailed elaborately with the existing applications as binary and ternary edible food packaging films and others.

In Chap. 10, the approach and significance of nanoencapsulation for several food components have been discussed. The chapter introduces the targeted core materials and carrier materials for the fabrication of nanoencapsulates of bioactive compounds for increased bioavailability and stability against the adverse conditions. The research and development in nanoencapsulation of food components using various techniques such as physical methods (spray drying, freeze-drying, fluidized bed coating, supercritical liquid method, etc.), physicochemical methods (spray cooling, hot melt coating, ionic gelation, solvent evaporation method, coacervation, etc.), and chemical methods (interfacial polycondensation, in situ polymerization, interfacial polymerization, and interfacial cross-linking) has been detailed in this chapter. The different case studies on nanoencapsulation of bioactive compounds and related characterization techniques such as morphology, physicochemical property, surface characterization, thermal properties, and others have been stated.

At the foremost, Chap. 11 discusses the state of the global market and research insights on bioactive compounds followed by the available types of bioactive compounds for edible food packaging applications including carbohydrate, lipids, phenolics, alkaloids, terpenoids, etc. The current trend in edible food packaging with the aid of bioactive compounds such as disappearing packages, seaweed-based packages, potato waste-based packages, casein-based packages, and others has been addressed in this chapter. Additionally, the chapter also focuses to discuss the various medicinal properties of bioactive compounds such as antioxidant property, anti-inflammatory property, antitumor property, antimicrobial property, antidiabetic property, neuroprotection, and others. The various available nanocarrier systems to deliver bioactive compounds in edible packaging of food products such as nanoparticles, lipid-based nanocarrier system, nanoencapsulation, nanoemulsion, and others have been deliberated further.

Chapter 12 deals with nanotechnology-assisted antimicrobial edible food packaging which is considered as a remarkable candidate in prolonging the shelf life of perishable and other food products. The chapter details with the design and functions of antimicrobial packaging with different types of available antimicrobial packaging such as sachets or pads containing antimicrobial agents, antimicrobial packaging with direct incorporation of antimicrobial agents, natural antimicrobial polymers, polymers immobilized with antimicrobials through ionic or covalent linkage and polymer surfaces coated with antimicrobials. Additionally, the chapter also discusses the research and developments in nanotechnology-aided antimicrobial edible food packaging using organic and inorganic nanomaterials, and nanoemulsion-based antimicrobial packaging for various food products to protect against microbial degradation and others. The safety evaluation in nanotechnology-based antimicrobial edible packaging against pesticides, additives, drugs, foodborne pathogens, heavy metal reduction, allergens has also been addressed in the chapter.

The advanced packaging technology including modified atmospheric technology, controlled atmospheric packaging, active packaging, and smart packaging for improved delivery of edible packaged products has been discussed in Chap. 13. The chapter details the strategies in modified atmospheric technology for improved product life targeting various perishable and other food products including the gas exchange mechanism and effectivity of several factors in modified atmospheric technology. The use of active packaging including gaseous composition and moisture-retaining system in active packaging, antimicrobial agents in active packaging, and utilization of active components in active packaging has been discussed elaborately in this chapter. Additionally, the various packaging strategies in controlled atmospheric technology and smart or intelligent packaging has been overviewed in this chapter.

The various perishable, semiperishable, and non-perishable food products which are packaged using edible films and coatings are described in Chap. 14. The chapter deciphers the nutritional benefits, spoilage, and preservation approach for fruits and vegetables, minimally processed fruits and vegetables, meat and meat products, poultry products, seafood, and fish products, and dairy products. The chapter also discusses the available bakery products and fried products with strategies to obtain the improved property. The advances in using edible films and coating using different types of materials retain environmental sustainability. The chapter aims to provide an overview of the suitable use of edible polymeric materials for several categories of food products.

In Chap. 15, the key interest has been given to detail the experimental procedures and characterization techniques for nanostructured materials, edible packaging, and food products. The chapter will provide a piece of basic knowledge to the readers to start working with edible packaging. The elaborate discussion on the required characterization techniques for analyzing nanostructured materials such as X-ray diffraction, X-ray absorption spectroscopy, small angle X-ray scattering, X-ray photoelectron spectroscopy, dynamic light scattering spectroscopy, transmission electron spectroscopy, Raman spectroscopy, Fourier transform infrared spectroscopy, atomic force microscopy, scanning electron microscopy, nuclear magnetic resonance, and vibrating sample magnetometer, and others has been made in this chapter. Further, a section of this chapter also focuses to discuss the characterization of edible films and coating materials such as film thickness, barrier properties, mechanical property, optical property, morphological analysis, thermal property, physicochemical property (water solubility, viscosity, moisture content, the stability of dispersion and rheological property), and others to evaluate the property of the materials. Specifically, the focused experimental techniques for shelf life analysis of food products such as physicochemical property, a study on respiration kinetics and mathematical modeling, sensory evaluation, texture property, migration study, and other properties have been discussed to proof the usability and commercial aspects of the developed edible packaging materials.

In brief, the book is focused on addressing the use of nanosystem-assisted edible food packaging with existing noteworthy properties and emerging application over existing packaging materials. Specifically, the book will provide systematic guidance to the researcher and industries to start working with edible food packaging for moving towards a sustainable future.

Guwahati, India

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Guwahati, Assam, India

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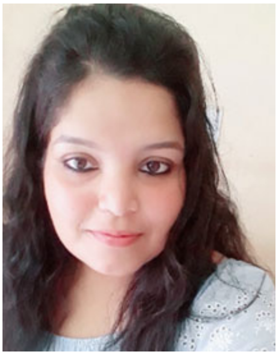
Dr. Vimal Katiyar is currently a Professor in the Department of Chemical Engineering at Indian Institute of Technology Guwahati (IIT), India. He is also an Honorary Senior Fellow to Kyoto Institute of Technology, Japan, Visiting Professor at GIFU University, Japan and has also been honored as Chair Professor at Numaligarh Refinery Limited and Hindustan Gums Co. Limited. He received his Ph.D. in Chemical Engineering from IIT Bombay, India. Currently, he is the coordinator for three Centre of Excellence at IIT Guwahati including Centre of Excellence for Sustainable Polymers funded by Department of Chemicals and petrochemicals, Government of India and the Joint Centre of Excellence for Biofuels and Biocommodities funded by Department of Biotechnology, Government of India and NRL-Centre of Excellence for Sustainable Materials at IIT Guwahati through industry intervention. His main area of research includes sustainable polymer development, its processing, and their structure property relationship, rheological aspects, migration studies, toxicological effects, polymer degradation, polymer based nanomaterials, food packaging, clean and green energy technologies. He is a co-inventor of 22 granted/filed patents. He has published more than 100 peer reviewed research articles in highly reputed journals and more than 250 conference papers and 40 book chapters.



Ms. Tabli Ghosh is currently a DST INSPIRE Fellow under the supervision of Prof. Vimal Katiyar in the Department of Chemical Engineering at Indian Institute of Technology Guwahati (IIT), India. Her work focuses on developing and evaluating the health impacts of edible medicinal coatings based on bioactive compounds facilitated bionanostructures for the enhanced shelf life of food products. She pursued her B.Tech. and M.Tech. from the Department of Food Engineering and Technology, Tezpur University, India. She was awarded the DST Inspire Fellowship for pursuing her Ph.D. She was a special research student at the United Graduate School of Agricultural Science, Gifu University, Japan. She has authored 18 book chapters, and 6 research articles in journals of national and international repute.

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Abbreviations

AFM	Atomic force microscopy
Ag	Silver
AgNP	Silver nanoparticle
AgNPs	silver nanoparticles
ALG	Alginate
ALT	Alanineaminotransaminase
AOT	Sodium bis(2-ethylhexyl) sulfosuccinate
AP	Antimicrobial packaging
AST	Aspartateaminotransaminase
ASTM	American Society for Testing and Materials
ATR	Attenuated total reflectance
ATR-FTIR	Attenuated total reflectance Fourier-transform infrared spectroscopy
Au	Gold
AuNPs	Gold nanoparticles
AV	Aloe vera
BC	Bacterial cellulose
BCNC	Bacterial cellulose nanocrystal
BCNF	Bacterial cellulose nanofibers
BEO	Basil leaf essential oil
BHA	Butylated hydroxyanisole
BHT	Butylated hydroxytoluene
BNC	Bacterial nanocellulose
BOPP	Biorientated PP
BTG	Benitaka Table Grapes
CA	Citric acid
CaO	Calcium oxide
CAP	Controlled atmospheric packaging
CAS	Controlled atmospheric storage
CCN	Curcumin–cinnamon EOs

CEO	Cinnamon essential oil
CFU	Colony forming unit
CGN	Curcumin–garlic EOs
CLSM	Confocal laser scanning microscopy
CMC	Carboxymethyl cellulose
CNC	Cellulose nanocrystal
CNCs	Cellulose nanocrystals
CNF	Cellulose nanofibers
CNW	Cellulose nanowhisker
CNWs	Chitin nanowhiskers
COX-2	Cyclooxygenase-2
CS	Chitosan
CSN	Curcumin–sunflower oil EOs
CSNP	Chitosan nanoparticle
CSNPs	Chitosan nanoparticles
Cu	Copper
CW	Cellulose whisker
DCP	Dicumyl peroxide
DHA	Cis-4,7,10,13,16,19-docosahexaenoic acid
DLS	Dynamic light scattering
DM	Degree of methylation
DNA	Deoxyribonucleic acid
DPPH	2,2-diphenyl-1-picrylhydrazyl
DSC	Differential scanning calorimetry
EAB	Elongation at break
EC	Ethylcellulose
EDTA	Ethylenediaminetetraacetic acid
EEAs	Essential amino acids
EELS	Electron energy loss spectroscopy
EFBP	Empty fruit bunch pulp
EFSA	European Food Safety Authority
ELISA	Enzyme-linked immunosorbent assay
EO	Essential oil
EOs	Essential oils
EPA	Cis-5,8,11,15,17-eicosapentaenoic acid
EU	European Union
EVA	Ethylene vinyl acetate
EVOH	Ethylene vinyl alcohol
EXAFS	Extended X-ray absorption fine structure
FAO	Food and Agricultural Organization
FBD	Fluidized bed drier
FCMs	Food contact materials
FDA	Food and Drug Administration
FEM	Finite element method
FESEM	Field-emission scanning electron microscopy

FTIR	Fourier transform infrared spectroscopy
GA	Gum arabic
GAPs	Good agricultural practices
GHP	Good hygienic practices
GI	Gastrointestinal
GMP	Good manufacturing practices
GRAS	Generally recognized as safe
GSE	Grape seed extract
GTR	Gas transmission rate
H_2SO_4	Sulfuric acid
H_3PO_4	Phosphoric acid
HACCP	Hazard analysis and critical control points
HBr	Hydrobromic acid
HCl	Hydrochloric acid
HDPE	High-density polyethylene
HO-MAP	High-oxygen modified atmosphere packaging
HPC	Hydroxypropyl cellulose
HPH	High-pressure homozenizer
HPMC	Hydroxypropyl methylcellulose
HRSEM	High-resolution scanning electron microscopy
HRTEM	High-resolution transmission electron microscopy
IC_{50}	Inhibitory concentration
ITO	Indium tin oxide
IU	International unit
JCPDS	Joint Committee on Powder Diffraction Standards
k-carr	k-carrageenan
LA	Lactic acid
LBG	Locust bean gum
LBL	Layer-by-layer
LC-MCC	Lipid-coated MCC nanoparticle
LDPE	Low-density polyethylene
LG	Lemongrass
LIBD	Laser-induced breakdown detection
LLDPE	Linear low-density polyethylene
LNs	Lipid nanoparticles
LPOS	Lactoperoxidase
LSPR	Localized Surface plasmon resonance
MA	Myristic acid
MAP	Modified atmospheric packaging
MC	Methyl cellulose
MCC	Microcrystalline cellulose
mgCNF	Magnetic cellulose nanofibers
MgO	Magnesium oxide
MgO-NPs	Magnesium oxide nanoparticles
MIC	Minimum inhibitory concentration

MLLC	Microemulsion Lyotropic Liquid Crystalline
MMT	Montmorillonite
M_r	Remnant magnetization
M_s	Saturation magnetization
NaCl	Sodium chloride
Nano-SiO_2	Nanosilicon dioxide
NC	Nanoencapsulated curcumin
NCC	Nanocrystalline cellulose
NCF	Nanocellulose fibers
NCS	Nanochitosan
NCs	Nanocrystals
NFC	Nanofibrillated cellulose
NFs	Nanofibers
Ni	Nickel
NLCs	Nanolipid carriers
NMR	Nuclear magnetic resonance
NOS	Inducible nitric oxide synthase
NP	Nanoparticle
NPs	Nanoparticles
OA	Oleic acid
OAN	Oleic acid nanoemulsion
OEC	Oxygen evolving complex
OEO	Oregano EO
OOR	Olive oil residues
OP	Oxygen permeability
OSHA	Occupational Safety and Health Act
OTR	Oxygen transmission rate
P (HB-co-HV)	Copolymer polyhydroxy (butyrate-co-valerate)
PA	Palmitic acid
PBAT	Butylene-adipate-co-terephthalate,
PBS	Poly(butylene succinate)
PBS	Polybutylene succinate
PCL	Poly(ε-caprolactone)
PDI	Polydispersity Index
PE	Polyethylene
PEG	Polyethylene glycol
PET	Polyethylene terephthalate
PF	Puncture Force
PHA	Polyhydroxyalkanoates
PHB	Poly(3-hydroxybutyrate)
PHV	Polyhydroxyvalerate
PLA	Poly lactic acid
PLA	Polylactic acid
PMBS	Potassium metabisulfite

PP	Polypropylene
PPI	Peanut protein isolate
PS	Pea starch
PU	Polyurethane
PVA	Polyvinyl alcohol
PVC	Polyvinyl chloride
PVDC	Polyvinylidene chloride
PVP	Poly(vinyl pyrrolidone)
QACCP	Quality analysis and critical control points
QELS	Quasi-elastic light scattering
REO	Rosemary essential oil
RFID	Radiofrequency identification tags
RH	Relative humidity
ROS	Reactive oxygen species
RTE	Ready-to-eat
SA	Sodium alginate
SAXS	Small angle X-ray scattering
SBN	Starch-butanetetracarboxylic acid dianhydride-N-hydroxysuccinimide
SDEY	Salted duck egg yolks
SEM	Scanning electron microscopy
SERS	Surface-enhanced Raman spectroscopy
SG	Sago
SHMP	Sodium hexametaphosphate
SiNPs	Silica nanoparticles
SiO_2	Silicon oxide
SLNs	Solid lipid nanoparticles
SLS	Static light scattering
SNC	Starch nanocrystal
SOS bags	Self-opening satchel bags
SPI	Soy protein isolate
SSOP	Sanitation Standard Operating Procedures
ST	Starch
STM	Scanning tunnelling microscopy
STNC	Starch nanocrystal
STNCs	Starch nanocrystals
STPP	Sodium tripolyphosphate
TA	Titratable acidity
TBA	Thiobarbituric acids
TBARS	Thiobarbituric acid reactive substances
TBC	Total bacterial count
TEM	Transmission electron microscopy
TEMPO	2,2,6,6-tetramethylpiperidine-1-oxyl
T_g	Glass transition temperature

TGA	Thermogravimetric analysis
TH	Thyme
TiN	Titanium nitride
TiO_2	Titanium dioxide
TiO_2NP	Titanium dioxide nanoparticle
TMA	Trimethylamine
TMAB	*Total mesophilic aerobic bacteria*
TMC	Total mesophilic count
TMR	Transparency Market Research
TNF-α	Tumor necrosis factor-α
TNTs	Titanium dioxide nanotubes
TP	Tea polyphenols
TPA	Texture profile analysis
TPC	Total plate counts
TPP	Tripolyphosphate
TS	Tensile strength
TSM	Total soluble matter
TSS	Total soluble solids
TTIs	Time-temperature indicators
TVB	Total volatile bases
TVB-N	Total volatile basic nitrogen
USDA	U.S. Department of Agriculture
USEPA	U.S. Environmental Protection Agency
USFDA	U.S. Food and Drug Administration
UV	Ultraviolet
UV-Vis	Ultraviolet–visible spectroscopy
VRS	Volatile reducing substances
VSM	Vibrating sample magnetometry
WAXS	Wide angle X-ray scattering
WG	Wheat gluten
WHO	World Health Organization
WI	Whiteness Index
WPC	Whey protein concentrate
WPI	Whey protein isolate
WPNF	Whey protein isolate nanofibrils
WS	Water solubility
WVB	Water vapour barrier
WVP	Water vapour permeability
WVPR	Water vapour permeability rate
WVTR	Water vapour transmission rate
XANES	X-ray absorption near edge structure
XAS	X-ray absorption spectroscopy
XPS	X-ray photoelectron spectroscopy
XRD	X-ray diffraction

YFT	Yellowfin tuna
YI	Yellowness index
ZEO	*Zataria multiflora* essential oil
ZN	Zinc oxide nanoparticle
ZnO	Zinc oxide

Chapter 1
Edible Food Packaging: An Introduction

Tabli Ghosh and Vimal Katiyar

1.1 Introduction

Edible food packaging is defined as a customized and sustainable packaging practice that can be consumed with the food product or can be removed before eating the edible packaged food, where the packaging materials have the characteristics features of biodegradability similar to the food materials. The current trend towards making a sustainable world, food processing industries are continuously developing the sustainable packaging materials as a replacement to available conventional packaging materials. The petrochemical plastics generate environmental pollution such as production of toxic components, increase in plastic-based waste. [1, 2]. In the USA, about one third of municipal wastes are generated from packaging and containers [3]. In this regard, several available biobased materials are continuously utilized for the upbringing of sustainable packaging due to their versatile nature such as non-toxicity, biocompatibility, biodegradability, renewability, easily available, degrade faster, no greenhouse gas emission, reduce carbon footprint. [1, 2, 4, 5]. Additionally, the food packaging is a very essential component for delivering the food products to targeted consumers in a safe condition. The benefits of using plastic packaging materials include flexible nature, versatility, lightweight, economic value, etc. Adversely, the drawbacks of using plastic as packaging materials include degradation of the environment, durability, generates harmful agents to the environment, etc. Thus, the plastic packaging materials are focused to be tailored for the various characteristics attributes, such as development of sustainable packaging materials in terms of edible or inedible packaging, which has an ability to reduce the generation of harmful agents to the environment.

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The use of edible films and coating started in the twelfth and thirteenth centuries [6] and now is one of the dominant food packaging materials throughout the world. This kind of packaging may be obtained in different forms and being developed from several substances. The development of edible packaging based on available materials generally selected targeting the food products. Further, the use of different edible materials should have the status of generally recognized as safe (GRAS) according to Food and Drug Administration (FDA). The most familiar use of edible packaging for consumers is found as the ice-cream cone, where waffles or sugar-based cones are utilized to carry ice-cream. The several innovations and development of edible ice-cream container [7], edible films for sundae ice cream cones (as moisture impermeable barriers) [8], combine edible cone and ice-cream [9] have put a remarkable impact in reducing the waste due to edible nature of the ice cream containers. Further, a fruit-like casing, known as WikiCell, has been developed by a company, viz. WikiFoods, which surrounds the foods, and further, the casing can be broken similar to the skin of the foods [10]. The WikiCell is a kind of film-like membrane having characteristics attributes of biodegradability, thin, soft, and held to carry a small portion of food products, which plays a role in replacing the plastic materials. The others include which are not available in the market are sugar casings, seaweed packaging, beeswax container, etc. The development of edible coffee cup using a hard cookie lined with a chocolate layer provides heat-resistant property. Further, cupcake wrappers and candy wrappers are also developed from starch (from potato fibers) and rice paper, respectively. The food-based industries such as grains, sugar, beverages, edible oils, fruits and vegetable processing industry, dairy industry, poultry processing industry, meat processing industry, fisheries, etc., are focused to utilize edible food packaging for reduced waste. Interestingly, the edible films and coatings as edible food packaging materials have a great deal of interest in both research and development section and industrial section because of the greenery and sustainable approaches. In this regard, the biobased polymers (as shown in Fig. 1.1) are thoroughly researched for the development of edible food packaging materials including cellulose, starch, chitosan (CS), gum arabic (GA), carrageenan, pectin, proteins (Sources: casein, whey, soy, zein, wheat gluten, cottonseed, collagen, egg white, wool keratin, collagen, etc.), lipids and waxes (fatty acids, acylglycerols, carnauba wax, beeswax, candelilla wax, rice bran wax, mineral oils, vegetable oils, paraffin wax, etc.), resins (wood rosin, shellac), etc. [11–29]. Ideally, the edible food packaging provides a remarkable opportunity in the innovations of food packaging materials due to their biodegradable nature.

However, the global market size of edible packaging is done based on (1) available sources such as plants and animals; between 2019 and 2025, among the two available sources, the plant-based sources are dominating the current market trends of edible packaging; (2) available input materials such as polysaccharides, proteins, lipids, waxes, plasticizers, and additives; (3) end use of developed materials such as pharmaceuticals, food packaging, beverages, edible cups,

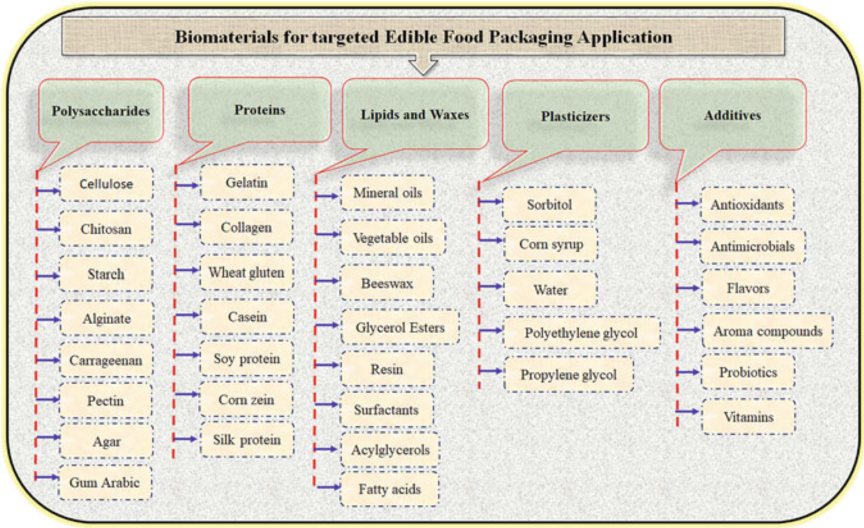

Fig. 1.1 Targeted biomaterials for Edible Food Packaging

cutlery items, fresh foods, baby foods, etc.; (4) targeted packaging application such as antimicrobial packaging, edible coatings, edible films; (5) regions such as North America, Asia-Pacific, Europe, South America, and others, etc.

Moreover, there are many benefits of using edible food packaging in regard to other available packaging materials such as the edible packaging are eatable, edible packaging have a biodegrading nature within a very short period of time, no waste cycling is required, provide health beneficial agents to human health, etc. Additionally, this kind of packaging is used as single-served food products. The limitation of edible packaging includes the water solubility of edible packaging, which may degrade the quality at humid climates; further, edible packaging needs another packaging material as secondary packaging for the transportation of the products due to hygiene concern. The edible food packaging should be compatible with consumers, where some of the edible packaging's may create allergies due to variations in components. The chapter will give an overview of the available food preservation techniques giving an emphasized focus on edible food packaging, related aspects including the use of nanotechnology in edible food packaging. Additionally, in this book, the emerging research fields related to nanotechnology in edible food packaging will be discussed.

1.2 Overview of Edible Food Packaging: History Outline, Classification and Current Prospects

1.2.1 Edible Coating: A Class of Edible Food Packaging

The edible coating is commonly used as a food preservation approach, where various edible materials are used to provide a thin layer of barrier on targeted food products. In the twelfth century, the edible coating was noted to be developed in China for the first time to coat citrus food products (oranges and lemons) using waxes to prevent water loss. In England, the lard or fats are used to develop a coating material on meat products during the sixteenth century, which was known as larding. During the 1930s, the citrus food products are coated with hot-melt paraffin waxes. In later days, the fresh fruits and veggies are coated with the aid of carnauba wax and oil-in-water emulsions. In the past days, the various beneficial properties of edible-coated food products were not well known by the consumers, which resulted in their unacceptability. However, the edible-coated food products obtained consumers' acceptance after they became familiar with the beneficial properties of edible-coated food products. The edible coating can be obtained by utilizing various kinds of approaches such as dip coating, spray coating, foaming, brushing, wrapping, dripping, fluidized bed coating, and panning, which help to create a barrier between the food product and the external environment. The inclusion of edible coating also helps to improve the appearance of food products with the aid of edible materials on the surface of food products. The edible food packaging in terms of coatings is used to give a semipermeable barrier against harmful environmental agents such as light, temperature, gaseous agents, microbial agents, etc. Further, this kind of postharvest preservation technique is extensively utilized for reduced respiration rate, maintaining weight of fruit products, total soluble solids, appearance, and others. The respiration of fruit and vegetables is also affected by storage temperature, time and gaseous conditions, etc. [30, 31]. The application of edible coating is utilized to coat cut pineapple using CS [32, 33], kiwifruits using aloe Vera [34]. The edible coating materials also include the widespread use of biopolymers including polysaccharides, protein, lipids and waxes, bioactive compounds, and others. The materials that are used for edible coating should be safe for human consumption, nutritionally rich, and are accepted by consumers. GA is another kind of polysaccharide used in food industry having several beneficial attributes such as antimicrobial, stabilizers, adhesiveness properties. [35].

The edible coating on food products is obtained by various processes, where dip coating of fruit product is one of the widely used processes. A detailed discussion about the available edible coating approaches has been made in Chap. 2. The dip coating is obtained by dipping the selected food product in selected coating solutions, and drying of the coated food products. In some cases, multiple layers of coating are also applied to obtain more effective food properties. The application of edible coating using dripping (applying the coating materials on the food products) is very cost effective, where the uniform coating can be obtained. Foaming-based edible coating is obtained by using a foaming agent, which is added to coating solution, where compressed air is further blown into the applicator tank. Spray coating involves the spraying of coating solution on the food products; this technique is used when a thin layer of coating is needed on food products. Besides biopolymeric materials, the components from fruits and vegetables are also used for the development of edible food packaging such as purees, pomaces, juices, active components, etc. [36]. The details of potential candidates used in edible packaging will be discussed in Chap. 2. The use of edible coating on food products has several aspects such as providing sweet flavor, enhanced texture of cereal products, reduce moisture loss of dried fruit (using mineral oil), improve appearance in fruits and chocolate candy, use as a carrier for active compounds, reduce mold growth in cheese, smoked fish, reduce fat uptake in fried products, etc. [37]. The critical concerns of edible coating materials include chemical safety, eatable food products, cost, barrier property, shelf life, food quality, nutritive value, environment, etc.

1.2.2 Edible Film: A Class of Edible Food Packaging

The edible films are another class of edible food packaging which have been an attractive packaging material in the current trend of packaging market with the immediate effect in commercialization of developed materials. The term "edible films" has two considerations, where the first term "edible" defines the designing of eatable materials and considered safe, non-toxic and the second term "films" defines about the film-forming properties of the materials similar to packaging materials [6]. Edible films are a thin layer of edible materials including polysaccharides, proteins, lipids and being used as pouches, films, wraps, and others on food products or between food components as sandwich materials. During fifteenth century, the development of edible films using soymilk, known as yuba film was done in Japan, which is the first free standing edible film. The yuba is developed by using denatured soy protein, and the film is used for ground meat, vegetables, and as a component in the soups [38]. The yuba film has gained a popularity in China and Japan having high nutritional and digestible property. Further, the edible films are developed using casting [39], extrusion, and compression molding [40], compression molding [41], and others which can further be used on or between food products. The proper application of edible food packaging depends on various characteristics such as selected food properties, selected materials, effect of materials, cost, etc. The properties of edible films are improved by adding various food additives such as natural food coloring agents, spices, antimicrobial agents, plasticizers, antioxidants, and others, which help in improving film properties such as optical properties, microbial properties, roughness, etc. The focused characteristics features of edible films include biodegradability, enhanced shelf life of food products, improved sensory properties, active functions of films, physical properties, optical properties, etc. The properties of food products are dependent on

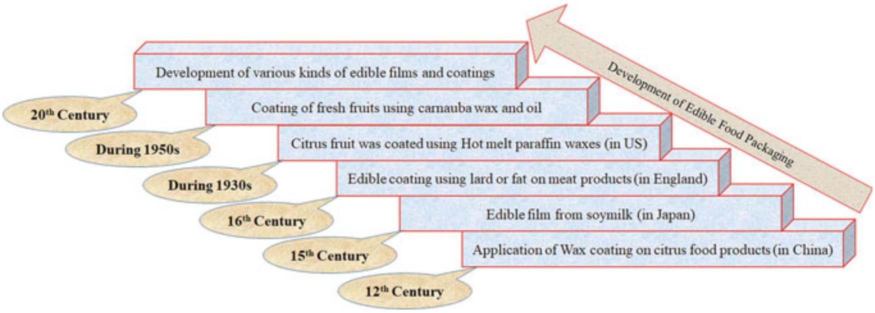

Fig. 1.2 Development of edible food packaging in terms of edible films and coatings

several factors such as film compositions, thickness of the film, transparency, etc. The versatile application of edible films includes as active packaging, oral-disintegrating films, edible oven bags, fruit and vegetable leathers, food wrappings, etc. The edible food packaging in terms of edible films and coatings provides various benefits for improved life of food products by reduced water leakage from food products, reduced gas diffusion, reduced solutes movement, improve appearance of food products, reduce microbial growth, reduce oil and fats movement, and others. The most crucial characteristics features of edible food packaged products are microbiological test, sensory properties, nutritional properties, wettability, mechanical properties, optical properties, etc. Additionally, the edible food packaging is also involved in the encapsulation of active compounds, aroma compounds, antioxidants, pigments, and others [42]. The development of edible food packaging in terms of edible films and coatings are displayed in Fig. 1.2.

1.3 Synergistic Use of Edible Packaging and Other Preservation Approaches

The synergistic use of several available postharvest approaches with edible food packaging helps to reduce the degradation of available food produces; however, packaging of food products may serve as a mean of protection, preservation, and promotion of food produces. The inclusion of preservation techniques (as shown in Fig. 1.3) such as thermal treatment, cold processing, use of preservatives, water controlling, and packaging (edible and inedible) can help to minimize the postharvest loss of fruit produces by reducing postharvest diseases, senescence, microbial count, reduced respiration rate, and others. The thermal processing of food products may be low-heat, medium-heat, or high-heat processing techniques, where the low-heat processing of food products is not effective toward thermoduric microorganisms. The selection of heat treatment generally depends on nature of

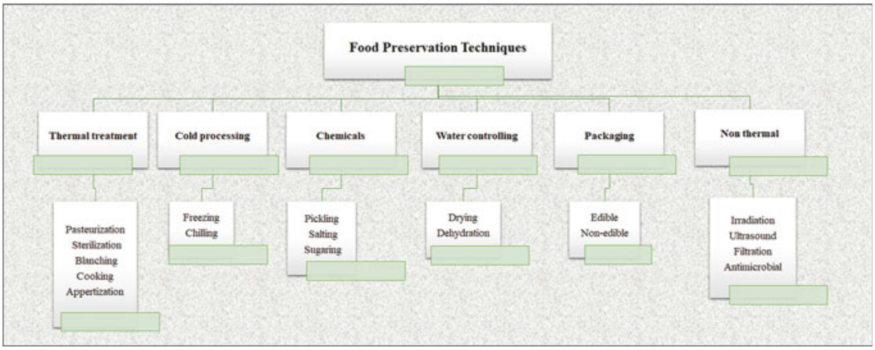

Fig. 1.3 Several food preservation techniques for improved products life

food products, nature of microorganism, nature of process, and others. In this regard, the use of several food preservation techniques reduces the food loss which in turn may help to meet the global food requirement. Additionally, the inclusion of several postharvest techniques help to make available seasonal food products and make easy transportation and exportation of food products. The extensively used conventional food preservation techniques include drying, dehydration, pasteurization, sterilization, pickling, freezing, edible films, and coatings, etc. The food product degradation occurs due to microbiological, enzymatic, chemical, physical, and mechanical reasons, where (i) the microbiological attack includes microbial growth, toxin production; (ii) enzymatic attack creates browning, color change, off flavor; (iii) chemical reasons are color loss, flavor loss, non-enzymatic browning, nutrient loss, rancidity; (iv) physical reasons are collapse, controlled release, crystallization, shrinkage, transport of component; and (v) mechanical reasons are bruising (due to vibration), cracking, damage due to pressure, etc. The traditional methods which are widely utilized for food preservation techniques are boiling, heating, drying, canning, cooling, freezing, salting, sugaring, smoking, pickling, etc. On the other hand, the industrial-based modern methods for food preservation includes vacuum packing, modified atmosphere, pasteurization, artificial food additives, irradiation, pulsed electric field electroporation, biopreservation, non-thermal plasma, etc.

The inclusion of pretreatment can improve the quality of food products such as edible coating and blanching as a pretreatment technique is a promising method for drying of pumpkins [43]. The formation of acrylamide (a procarcinogen) in banana chips can be reduced with the aid of pre-frying treatments such as blanching and pectin-based coating [44]. The application of coating materials based on starch and pectin for osmotic dehydrated and convective dried food products can effectively influence the drying attributes, where the drying characteristics also depend on the type of coating materials [29]. A report suggests that the use of edible coatings (whey protein isolate and pullulan) on freeze-dried Chinese chestnut can effectively improve the quality and shelf life of chestnut [45]. The growth of *Listeria* *monocytogenes* (a pathogen which causes foodborne infections) in roasted turkey (at chiller storage) can be effectively controlled with combined processing techniques such as edible antimicrobial coatings (pectin) with frozen storage [46]. In this regard, the application of freezing with edible coating can further minimize the risks of listeriosis (an infection) caused by the germ *L. monocytogenes*. A report further suggests that the application of pectin and green tea powder as edible coating materials can effectively improve the quality of irradiated pork patty (Irradiation at 0 and 3 kGy using cobalt-60 gamma rays) [47]. The edible-coated pork patties have a reduced count of total aerobic bacteria in comparison to uncoated pork patties. Further, in 2003, **Vachon et al.** have studied the effectiveness of edible coating (based on caseinate) and gamma-irradiation treatment on the keeping quality of fresh strawberry fruit products [48]. The application of both the treatment helps in delaying the mold growth and the irradiated caseinate is more effective in comparison to the unirradiated caseinate for obtaining improved storage life of strawberry. The combined application of gamma irradiation, ascorbic acid, and protein-based edible coating can help to improve the keeping quality of ground beef in terms of biochemical and microbial attributes [49]. Additionally, the edible films based on milk protein such as calcium caseinate and whey proteins cross-linked using radiative and thermal treatments can also provide tunable mechanical and structural properties [50]. The storage quality of fresh-cut pears can be maintained with the aid of pure oxygen pretreatment, CS, and rosemary extract-based edible coating [51]. The combined effect of the treatments has an ability in inhibiting polyphenol oxidase activity and further increases the beneficial properties of fresh-cut pears and reduces the browning effect, etc. There are various other pretreatments using additives (ascorbic acid, citric acid, sodium benzoate, and others), controlled atmosphere storage, and CS coating can improve the quality of fresh-cut jackfruit bulbs slices by retaining phenolic content, sensory attributes, and phenolic content, etc. [52]. A report suggests that the characteristics quality attributes of strawberries can be monitored during freezing when whey protein-based coating is used as a pretreatment method [53]. Thus, the inclusion of food preservation technique and edible food packaging has a beneficial property of improved product life as represented in Fig. 1.4. The available food preservation techniques are used for improved shelf life of food products, however, the inclusion of some preservation techniques may alter the taste and nutritional quality of specific food products due to different processing conditions and use of different agents. In this regard, edible food packaging in terms of edible films and coatings helps in maintaining the nutritional quality of food products. Further, the edible coating and films enhance the nutritional quality of food products by delivering active agents from available natural agents. The use of edible coating and films is different, where edible coating is developed on food products by developing a thin layer of edible material on food products and edible film is used as a layer of edible material between food products or onto food surface.

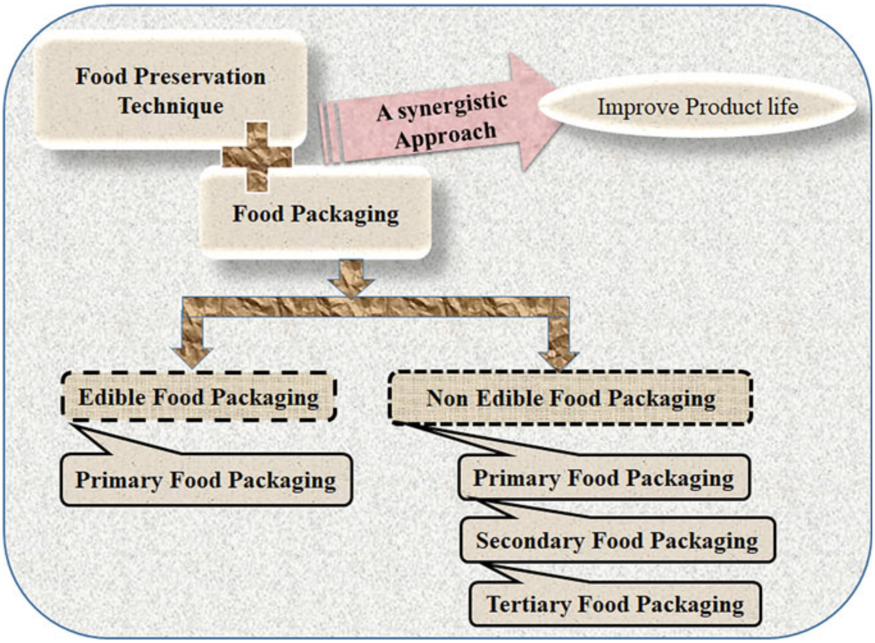

Fig. 1.4 Synergistic approach of several food preservation techniques and edible food packaging

1.4 Transportation of Edible Packaged Food Products

The food packaging is classified into three categories such as primary packaging, secondary packaging, and tertiary packaging. The primary packaging materials remain in contact with food products. The secondary packaging materials are used to transport the primary packaged food products such as carton boxes, paper boxes. On the other hand, the tertiary packaging materials are used to carry the secondary packaged food products. The secondary and tertiary food packaging materials can be recycled and reused if used properly. The edible food packaging materials are generally considered as primary food packaging materials, which are a potential candidate in preserving, transporting, and marketing of food products. The edible food packaging in terms of edible films and coatings is used to provide tailored food properties. The addition of various plasticizers and additives can be added to different biopolymeric materials to obtain the improved physical properties, surface functionality of the biopolymeric materials. The intermolecular forces within biopolymeric materials include electrostatic, hydrophobic, covalent bonds, ionic interaction, and others. The main difference in application of edible films and coatings is that edible films are applied in solid forms, and on the other hand, edible coatings are applied as liquid form and then get dried for easy transportation [54]. The use of edible materials in improving the shelf life of food products depends on some of the characteristics attributes of used materials such as availability, functional properties, cost, optical properties, barrier properties, sensory properties, safety of materials, etc. Edible coatings provide a passive modified atmospheric environment for the fruits and vegetables which help in reducing respiration rate in terms of oxygen and carbon dioxide, increasing the shelf life of food products. In this way, the use of edible food packaging with available inedible packaging materials as secondary and tertiary packaging can be utilized.

1.5 Global Overview of Edible Food Packaging in Research and Development

In this section, the past and existing trend in edible food packaging obtained by using continuous matrices of polymers, nanoparticles, and active agents, etc., will be discussed. Edible food packaging is one of the potential candidates utilized as a food preservation technique for the improved shelf life of food products. The global consumerization of edible food packaging as edible coating and films are increasing due to several advantageous features such as reduced plastic waste, use of naturally availability, biocompatibility of materials, renewable materials, non-toxicity, increase food value, ready-to-eat food products, etc. The improved food quality generally depends on several factors such as type of coating materials, processing condition, concentration of materials, storage condition, type of food products, etc. The existing biomaterials or biopolymers are used individually or in a combined form for maintaining the food property during storage. Based on the discussion, the present chapter will provide a general global status of edible food packaging materials.

The materials in edible packaging should be eatable both in the initial and in the final packaging forms [55]. The edible packaging is a remarkable candidate in using biobased packaging materials throughout the world. The first patent on edible coating was available in the year 1933 in the USA, where wax coating was mainly applied to citrus fruit products [55]. In later days, the many inventions have been done on edible coatings such as edible film-coated dried fruits [56]; coating of dehydrated foods [57]; meat [58]; frozen confection [59]; fruit pieces [60]; frozen fish [61], etc. Edible coatings provide a lot of beneficial properties in retaining food factors such as reduction of fat uptake in the deep fat-fried food products (meat, potatoes) based on hydrocolloids as edible coating with better nutritional quality [62]. In this regard, the edible oil barrier properties in edible films and coatings are very essential to obtain nutritional-fried products. The patents on edible films available are CS or mixture of quinoa protein-CS [63], gelatin [64]; films and edible food casings from carboxymethyl cellulose [65]; casein-based edible films [66], etc. The patents on edible films for their versatile use are also available such as transmucosal delivery of terpenes [67]. Further, the tunable film-forming properties of edible food packaging materials are attained via heating, enzymatic modification, salt addition, drying, cross-linkers, food additives, etc. In this way, the innovations in research and development are providing a new trend in the field of food packaging with many beneficial traits.

1.6 State of Global Market and New Trends in Edible Food Packaging

The global consumerization and market value of the edible food packaging market are increasing day by day. In this regard, according to the available report on Global Opportunity Analysis and Industry Forecast, 2017–2023, the edible food packaging market was marked at $697 million in 2016, which is forecasted to reach $1097 million by 2023. The popularization of edible food packaging market relates to the increase hygiene concern, reduced conventional packaging waste, etc. The plastic-based waste is a critical concern to the society which further increases the carbon footprint, global warming, which acts as a catalyst for market growth of edible food packaging. On the other hand, the manufacturing regulations, high manufacturing cost, safety concern may lead to a decrease the market value of edible food packaging. The main marketing features of edible food packaging include class of materials, targeted end users, and major market areas. The principal class of materials includes polysaccharide, proteins, lipids and waxes, biocomposites, blends, surfactants, active agents, etc. The targeted consumers of edible food packaging include food and beverages, pharmaceuticals, medicinal use, etc. The major market area of edible food packaging includes North America, Europe, Asia-pacific, etc. The global food packaging markets are WikiCell Designs Inc., MonoSol LLC, Tate & Lyle Plc, JRF Technology LLC, Safetraces, Inc., Bluwrap, Skipping Rocks Lab, Watson, Inc., and Devro plc., etc. The region-wise analysis of the global edible packaging market covers North America (US, Canada, Mexico), Europe (UK, Germany, France, Rest of Europe), Asia-Pacific (India, China, Japan, Rest of Asia-Pacific), and LAMEA (Latin America, Middle East, Africa) as represented in Fig. 1.5. Various countries covered under each region are studied and

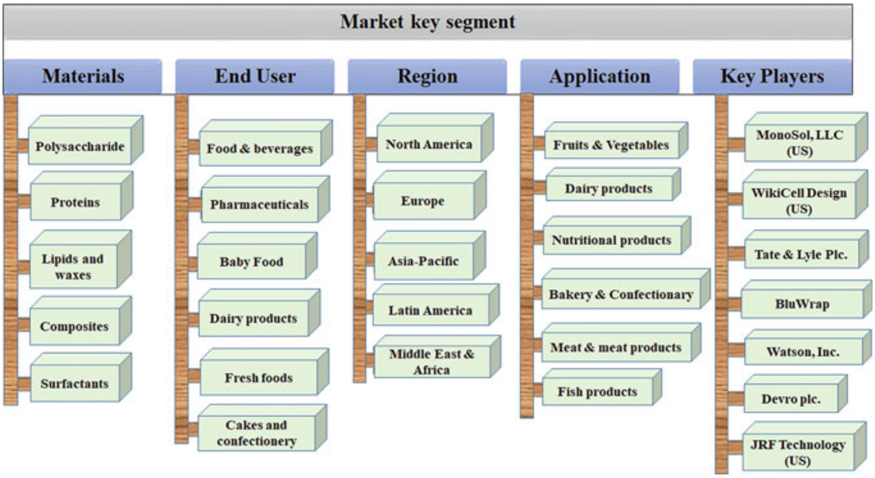

Fig. 1.5 Market key segment of edible food packaging

analyzed to identify the major trends demonstrated by these respective regions. Europe dominated the global edible packaging market in 2016, followed by North America.

The global market for edible food packaging products is available with many products such as seaweed, edible cutlery, Ooho edible water, LOLIWARE edible cups, casein, etc.

The company for edible food packaging materials are Bakeys, India (sorghum, rice, wheat flours), Coolhaus, Los Angeles (potato wafer paper wrapping), Do Eat, Belgium (water and potato starch), Ecovative, New York (mycelium packaging and Founder: Gavin Mcintyre), Eco Six Pack Ring (E6PR), Mexican (compostable matter and by-product waste), Evoware, Indonesia (seaweed), LOLIWARE (seaweed, organic sweeteners, fruit and vegetable coloring), MonoSol, Indiana (edible pods for instant beverages), NVYRO, UK (Cassava plants), Poppits, Florida (food-grade edible films), Scoby, Poland (edible, recyclable package), TIPA, Israeli (biomaterials and technology), etc. In this regard, the available edible food packaging in the worldwide has been represented in Table 1.1. Further, the commercial

Table 1.1 Global market of edible packaging

Sl. No.	Company	Components of packaging	Product and properties	References
1	Bakeys, Hyderabad, Telangana, India Trade Name: Bakeys Foods Private Limited	**Different types of flour** such as Sorghum, wheat, rice, millet **Several flavors in spoon:** cumin, mint-ginger, sugar, carrot-beetroot	Edible Cutlery, Edible spoons, Edible forks, Edible chopsticks	[70–72]
2	Coolhaus, Los Angeles, California	Potato starch	Edible wrappers for ice-cream sandwich	[73]
3	Do Eat, Belgium	Water and Potato starch	Edible Tableware, Verrines, Sandwich rings, Cupcake Holders, Food bags	[74]
4	Ecovative Design, New York	Mushroom materials from Fungal mycelium, Non-food agricultural materials	Mycelium packaging, MycoComposite, MycoFlex, Atlast Protective packaging, structural biocomposites, thermal insulation, etc.	[75]
5	Eco Six Pack Ring (E6PR), Mexico	Compostable matter and by-product waste	Holder for beer cans	[76]

(continued)

Table 1.1 (continued)

Sl. No.	Company	Components of packaging	Product and properties	References
6	Evoware, Indonesia	Burger wrapper, Instant noodle seasoning sachets, Coffee pouches	Seaweed	[77]
7	MonoSol, Indiana, US	–	Food-grade and water-soluble films, edible pod for instant beverages, wrapper and sachets, soluble in hot and cold water, water-soluble films	[3, 68, 69]
8	NVYRO, United Kingdom	Cassava starch Tapioca starch	**Nvyro disposable food packaging products:** Plates, Cups, Lunch boxes, Trays, Lunch plates, Ready to eat foods, Eatable plates **Suitable food products**: Liquid, cold, hot, dry, semi-liquid foods	[78, 79]
9	TIPA, Israel	Fully compostable plastic packaging, end life is like orange peel, decomposting in 180 days Can be used for dry, baked and frozen products	Biomaterials and technology	[80, 81]
10	Scoby, Poland	Kombucha	Edible, recyclable package **Properties**: Fully edible and recyclable, Sachet, bag, and bowl Zero waste production	[82]
11	Loliware, New York	Alginate (Seaweed), agar (Red algae)	Flavored straws **Properties**: Behave like plastic for 24 h	[77]

edible coatings and films available in market are BioEnvelop®, Chris-Kraft Polymer Inc. COGIN®, ENAK®, Freshseel™, Fry Shield™, GREENSOL®, Nature Seal™, Nutrasave™, Opta Glaze™, Seal gum, Spray gum™, Semperfresh™, SHELLAC(E904), Z-Coat™, etc. [6]. The market trend in edible food packaging is growing day by day due to the increased socio-scientific demand in the current situation.

An overview of the available global market of edible food packaging has been made as listed below:

I. The edible packaging from seaweed is created by Evoware, an Indonesian company. The seaweed-based edible packaging is eatable and developed using sustainable materials, heat sealable, printable, dissolve in warm water and provides complete biodegradability, which further acts as fertilizer for plant materials. The Indonesian company used this seaweed for packaging dissolvable coffee sachets, burger wraps, sandwich wraps, etc.

II. The edible cutlery such as Bakey's edible cutlery can be developed from sorghum, wheat, and rice having a very delicious taste, which acts as a great replacement for disposable cutlery and replaces the plastic-based waste decreasing global warming. The Bakey's company was founded in India by Narayana Peesapaty due to groundwater depletion and the creation of plastic-based toxins on human systems.

III. The edible potato wafer paper wrapping was developed by Coolhaus, a Los Angeles-based ice-cream company. The edible potato wafer is a sustainable and environmental friendly replacement to the other available plastic-based wrappers.

IV. Another Belgium-based company, viz. Do Eat offers edible packaging materials using potato starch and water which provide gluten free packaging material.

V. MonoSol is another packaging company in India, which developed edible pods for instant beverages, where the pod completely dissolves in water and are safe for eating; thus, the company is trying to replace the wrapping of food products [3, 68, 69].

VI. The edible water ball has been developed by Skipping Rocks Lab, a startup in London using naturally available plants and seaweed. The developed edible water ball is developed from natural agents, and further, it can flavored, colored, and biodegrade within 4–6 weeks, if not consumed.

VII. The edible and biodegradable cups by Loliware's are developed from seaweed, organic sweeteners, color materials (available from fruits and vegetables) and exists in various flavors like cherry, grapefruit, and yogurt.

VIII. The casein-based films are edible, degradable, and are very effective in the prevention of spoilage of food products. The development of WikiCells similar to eggshells is a kind of edible packaging materials which are launched by David Edwards in 2012. Further, the WikiCells are developed using charged polymers and food particles, where wine, chocolates, and juices can be filled.

1.7 Significance of Edible Food Packaging

Several traits of edible food packaging have been displayed in Fig. 1.6. The addition of health beneficial agents such as antioxidant agents, antimicrobial agents, to food products by means of edible food packaging is considered as another effective way for an enhanced product life of perishable food products. The edible food packaging acts as a safeguard against various kinds of injuries such as mechanical, thermal, chemical, physical, microbiological, and others [54]. The

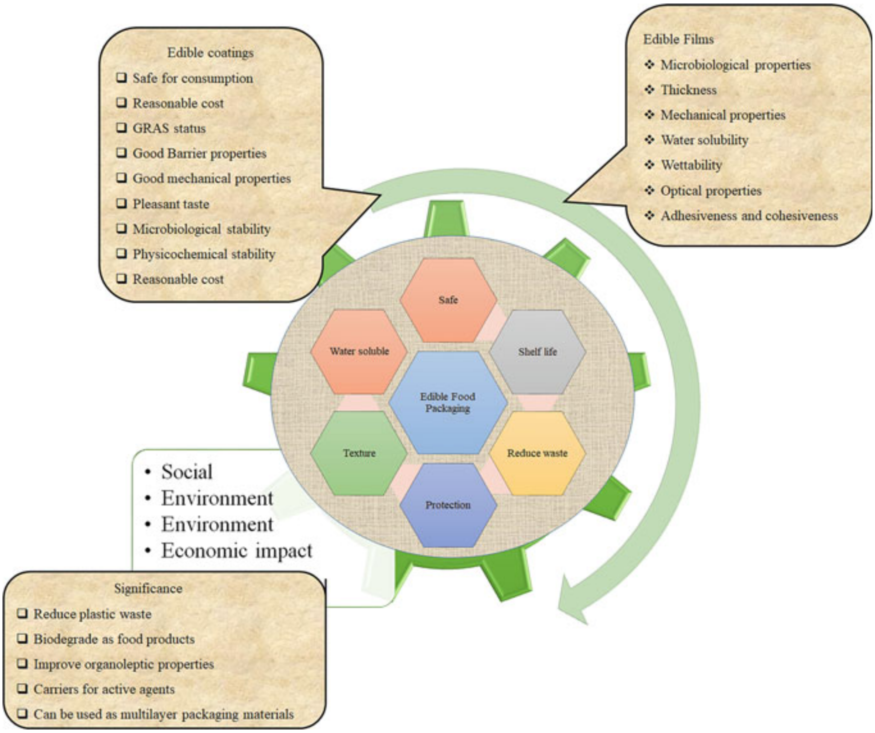

Fig. 1.6 Significance of edible food packaging

single-layer edible coating cannot provide effective barrier agents against the environment. Thus, a multiple layer of edible coating can serve the purpose of improved shelf life of food products efficiently. From very early days, cellulose-based biopolymers are widely utilized for food packaging applications such as paper, polymer composites, edible packaging. The biopolymer extraction and their usability are dependent on several factors such as temperature, relative humidity, microbial spoilage, etc. In recent past years, the use of biopolymers has attained a remarkable attention in food packaging technology for its wide applicability. Based on this, the use of biopolymers as an edible food packaging materials are gaining significant attention.

There are several advantages of edible packaging, such as:

- Reduce plastic-based waste and solid waste
- Reduce carbon footprint and global warming
- Increase the nutritional value of food products
- The edible packaging can act as a carrier for antimicrobial, antioxidant, or other active agents

- The edible packaging can capture various active agents as encapsulation
- Improves aesthetic property of food products.

The limitations of edible packaging include:

- Increased cost
- Secondary packaging is required to carry edible-coated food products such as blown film materials [83]
- Secondary packaging materials are costly
- Environmental sensitive packaging materials.

1.8 Edible Food Packaging with Medicinal Effects

In the modern world of fast moving people, health-preserving diet is an essential need of a healthy lifestyle. However, we also tempted to eat unhealthy food which may have harmful effects, especially to the diseased person. Therefore, it is proposed herewith to develop an approach to administer the required amount of nanomedicinal doses along with food without compromising the essence of food products. This will avoid the consumption of high doses of drugs at once when administer using tablets which lead to the possible toxicity. Further, the nanodispersion of natural medicinal agents in the form of nanocoating on the restricted diet may tune it as a balance diet with no adverse effects on the body. Thus, targeting various diseased conditions, preparation of coating material from various herbals plant materials, and others such as extract of karela, *Terminalia chebula*, punarnava, ginger, seeds of bitter apricot, green tea extract, fig, and chirayta medicinal plant on the restricted diet of a diseased person can be obtained. The edible medicinal food packaging can be developed through incorporating medicinal filler materials which can be extracted from available medicinal plants. Further, the nanofiller materials can be used to deliver medicinal agents to food products. The medicinal agents added edible packaging can be applied on food products as a human disease suppressing agents.

1.9 Nanotechnology in Edible Food Packaging

The nanotechnology in food processing has a great deal of interest to provide potential benefits such as producing functional food products, extended shelf life of food products, intelligent packaging using nanosensor, increase food production, etc., as shown in Fig. 1.7. In the present book, the inclusion of nanotechnology in edible food packaging developed from available biocompatible and non-toxic biopolymeric materials obtained from available sources will be summarized to highlight the necessity and applicability of edible packaging in today's world.

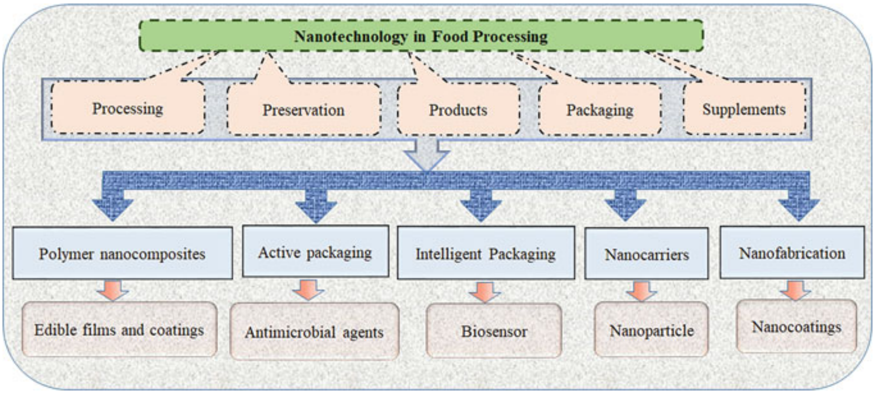

Fig. 1.7 Nanotechnology in food processing

Further, the present book will also discuss the synthesis strategies of edible packaging utilizing different methods, targeted food products, characterization techniques, and related other multifaceted applications. Based on this, the book has been classified into four different distinct areas such as recent advances in edible food packaging, edible films and coating application, packaging technology for the prolonged shelf life of edible packaged food products, and targeted food products and characterization tools. However, the current trend of the food packaging market is widely utilizing the sustainable nanostructured materials for delivering tailored-made properties of eco-friendly food packaging.

The present book provides a complete overview of the current trend and need for nanostructured materials in the field of edible food packaging as shown in Fig. 1.8. A brief overview of the various types of edible food packaging in terms of edible coating, films, and others including the global market of edible packaging with related pros and cons will be discussed in this chapter. The future prospects of market growth and the customer prospects in edible food packaging will also be discussed in this chapter. There are various naturally available materials which are utilized to develop edible packaging, which will be detailed in Chap. 2. Further, a detailed about the available materials and their derived forms for the synthesis of edible packaging of food products will be deliberated in Chap. 2. In Chap. 3, the extraction of cellulose and its derived forms including nanostructured materials for edible packaging will be mentioned. Similarly, a detailed discussion about other nanoforms of some of the polysaccharides such as CS, starch, and others are elaborated in Chaps. 4 and 5. The another kind of biopolymeric materials such as protein which is one of the crucial materials for the development of edible packaging in terms of edible films and coatings will be discussed in Chap. 6. Further, the nanostructured form of protein-based materials for edible packaging is another remarkable area for edible food packaging. In Chap. 7, the use of lipid-based nanostructured materials in the field of edible food packaging will be discussed. In Chap. 8, the utilization of various available inorganic nanofiller materials including

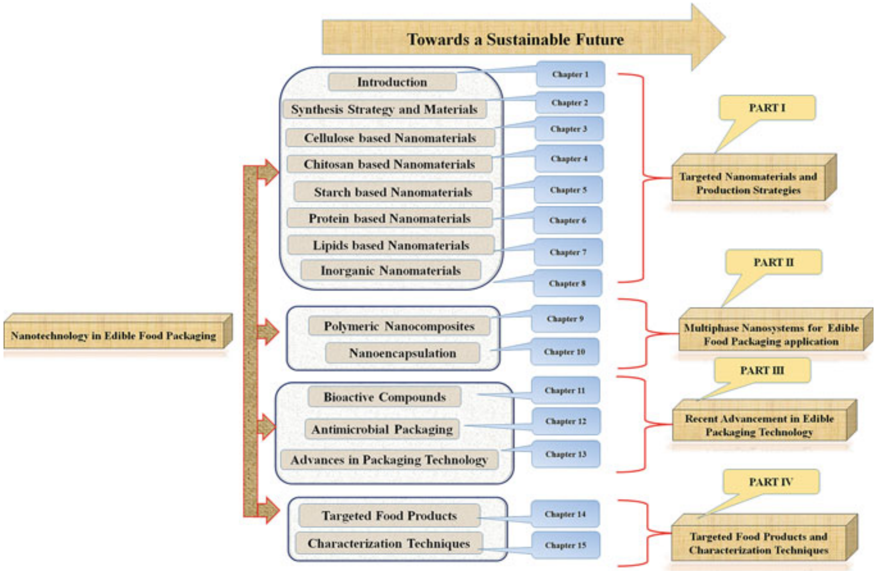

Fig. 1.8 Overall representation of the book "Nanotechnology in Edible Food Packaging"

titanium dioxide, silicon dioxide, zinc oxide, iron oxides, and others in the development of edible food packaging will be discussed. Additionally, an elaborate discussion on using biopolymeric composites and their modified forms in edible food packaging has been made in Chap. 9. Nanoencapsulation is a category of edible packaging, where active compounds are captured using nanomaterials for improved properties, which is a matter of discussion in Chap. 10. In Chap. 11, a discussion on bioactive compounds for edible films and coating with the aid of nanostructured materials of biopolymers such as CS, pectin and dextran with enormous potential will be made. In Chap. 12, the application of various antimicrobial property assisted materials such as CS, essential oils for edible food packaging and others will be done. Further, the application of several advanced packaging technologies such as modified atmospheric packaging, controlled atmospheric packaging, active packaging and smart packaging can help to safe transfer of food products to distant places (Chap. 13). The edible packaging in terms of films, coatings, and others are extensively utilized to improved product life of various perishable and semiperishable food products. Further, the targeted food products such as fruits and vegetables, meat and meat products, dairy products, bakery products which are packed using edible packaging (Chap. 14). Finally, the various characterization techniques needed to analyze edible materials, their products and shelf life analysis of edible food packaged products will be discussed in Chap. 15.

1.10 Conclusion

The production of edible food packaging is continuously being researched to aid functionality and to develop different types of packaging materials. The use of nanotechnology in edible food packaging has brought a real sense in making a large contribution to the food industry. However, nanotechnology has brought a revolution in the field of food packaging industry with the use of various nanostructured materials such as nanocellulose, nanochitosan, nanostarch, protein nanoparticles, lipid-based nanoparticles, inorganic nanoparticles, and others. The addition of nanostructured materials fortifies the food products and further can act as a delivery agent for bioactive components to improve shelf life.

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Chapter 2
Edible Food Packaging: Targeted Biomaterials and Synthesis Strategies

Tabli Ghosh, Deepshikha Das, and Vimal Katiyar

2.1 Introduction

From very past years, edible food packaging being a kind of green packaging materials with eatable nature has attained great interest in food packaging industries with additional benefits of using natural resources. As discussed in the previous chapter, the "edible food packaging" is generally availed in the form of "edible films" or "edible coatings" to provide value-added food products with improved quality and shelf life. Among available packaging, the edible food packaging helps in keeping the food product safe and fresh from growing bacteria and spoilage. Additionally, the edible packaging incorporating active agents is one of the well-known packaging concepts in order to control the growth of unwanted microorganisms on the surfaces of the food. However, most of the food packaging products are wrapped in packaging, which are non-biodegradable in nature and create several environmental problems. Further, the use of biodegradable-based edible films for packaging food has been designed in a way to minimize health hazards and global transformation. The main objective of edible packaging films is to protect the food from microorganisms and preserve the foodstuffs. The edible films have been developed using many biodegradable materials targeting several applied areas such as oral-disintegrating films, edible oven bags, active packaging, wraps, fruit and vegetable leathers. The targeted packaging based on specific application is developed based on the selected materials and processing techniques. The successful development of edible coating depends on several factors such as employed method and properties of coating materials such as category, quantity, surface property, and chemical compositions. In this regard, the chapter discusses

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several biomaterials and synthesis strategies for developing edible food packaging for different applications.

The various renewable resources used in edible food packaging include polysaccharide, protein, lipid, and others, which are availed from several renewable resources [1]. The extensively available commercial edible packaged foods in terms of edible films or coatings developed from renewable resources are ice-cream cone, commercial waxing on fruits, collagen casing, chocolate coatings, cellulose ether-based water-soluble pouches, gelatin-based coatings, and others. The mentioned biomaterials are obtained from several available renewable sources. Interestingly, the used renewable resources for edible food packaging are also extracted from waste materials such as fruit peel, banana plant waste, sugarcane bagasse, corn stover, and sea-based waste. In this way, the utilization of natural resources in edible food packaging can be a strategic way to convert “waste into wealth” and further will help to reduce waste materials. However, the success of edible food packaging for obtaining in prolonging the shelf life of food products also depends on the structure, chemical composition, and film-forming properties of selected polymers, storage conditions, and the surface interaction (wettability and spreading coefficient) between fruit and coating materials. Additionally, the edible-coating process is also dependent on several factors including materials composition, surface morphology, geometry of product, etc. In this regard, the inclusion of nanosystem-assisted technique in developing edible coating has several advantages such as transportation of bioactive compounds to food products [2]. The application of nanotechnology in developing edible food packaging involves polymeric nanoparticle, nanocomposite, nanoencapsulation, nanoemulsion, inorganic nanocomposite, etc. The nanosystem can also transfer active components to food components in a controlled manner. The biopolymeric nanostructured materials are available in different morphologies such as nanocrystal form, nanofiber form, nanoparticles, and others, which deliver tailored-made properties of edible-coating materials. In this regard, an overview of the available nanostructured form that is utilized in developing edible food packaging materials has been discussed in the current chapter.

There are several strategies which are employed to develop edible films and coatings, where the selection of processing techniques is based on several factors such as targeted food products, quantity, and food property. Additionally, the processing strategies are applied to develop edible food packaging for food products as the technique is gaining interest as the packaging material can be eaten with the food products. Moreover, the consumer demands for ready-to-eat, high quality, safe and convenient food, which provide a great need in developing the large-scale production or commercialization of edible food packaging. However, the, challenging factors in edible food packaging are process equipment, food product to be packaged, and processing conditions, where several processings are suitable for different product types.

Based on the above discussion, the current chapter discusses the available biopolymeric materials and the modified forms to be utilized in developing edible food packaging. The edible biopolymeric materials such as polysaccharides (cellulose, chitosan, starch, glycogen, pectin, etc.), proteins, and lipids are used for developing edible food packaging. A detailed overview relating these biopolymeric materials, their modification strategies to overcome the existing shortcomings of materials such as hydrophilic property of some biopolymers, poor mechanical property, and low water solubility. In this regard, the chemical modifications and development of biocomposites and blends can modify the properties of biopolymers to obtain enhanced shelf life of food commodities. Further, a brief overview of the nanostructured materials used for the fabrication of edible food packaging has been done. The chapter also details several synthesis approaches for developing edible coating and films on food products. The processing steps that are adopted to develop edible-coated food products and edible films will be summarized in the subsequent section with their advantageous properties and associated shortcomings.

2.2 Biomaterials in Edible Food Packaging

The principal components of edible packaging commonly include polysaccharides, proteins, and lipids-based materials. Further, the categories of biomaterials for developing edible food packaging are represented in Fig. 2.1. Besides, the other components of edible food packaging including the biopolymeric materials are (i) puree, juices obtained from fruits and vegetables; (ii) inorganic materials; (iii) natural fibers, nanomaterials as filler materials; (iv) alcohol and sugars as plasticizers; (v) antimicrobials, antioxidants, nutraceuticals, probiotics as functional additives; and (vi) browning inhibitors and cross-linking agents as additives. The used materials in edible packaging are biodegradable, reduce the solid waste management problems, and do not require any mode of recycling. The main characteristic features of edible food packaging (Fig. 2.2) include several attributes such as (i) packaging properties (barrier, mechanical, and thermal properties), (ii) sustainability developed from renewable and biodegradable materials, (iii) provide improved shelf life for offering low moisture content, low water activity, good stability, (iv) active functions such as antibacterial, antifungal, antioxidant, (v) sensory attributes such as texture, color, and flavor, and (vi) health benefits such as food fortification with nutraceuticals and others.

2.3 Polysaccharides in Edible Food Packaging

Polysaccharides are a class of carbohydrates consisting of chains of monosaccharide units linked together by glycosidic bonds. The other classes of carbohydrates are termed as monosaccharides and oligosaccharides consisting of single and 2–10 monosaccharide units, respectively. The polysaccharides are classified based on various factors such as repeating units, sources, and origin. The polysaccharides based on origin (as shown in Fig. 2.1) are categorized as (i) animal-based origins:

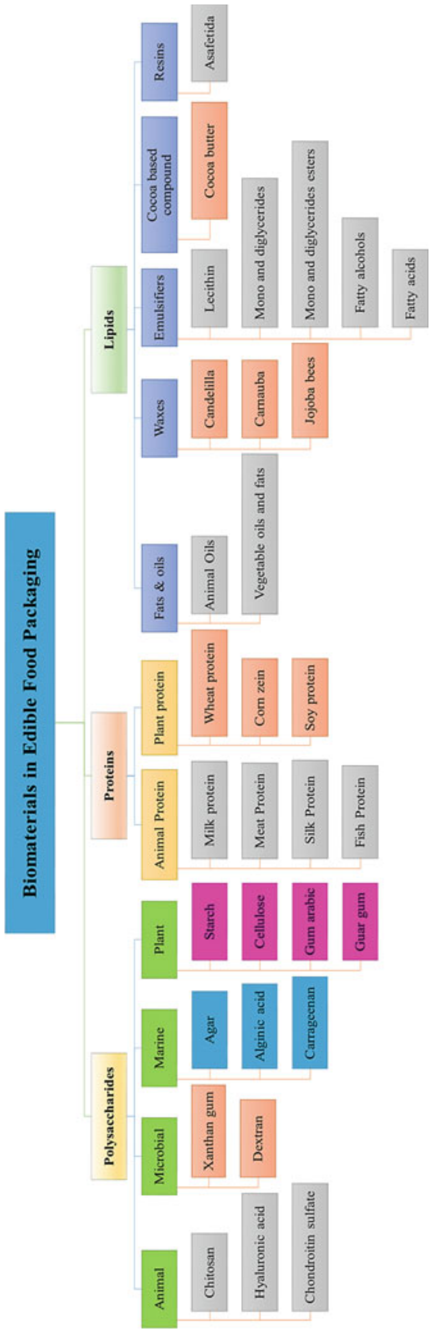


Fig. 2.1 Targeted biomaterials for developing edible food packaging

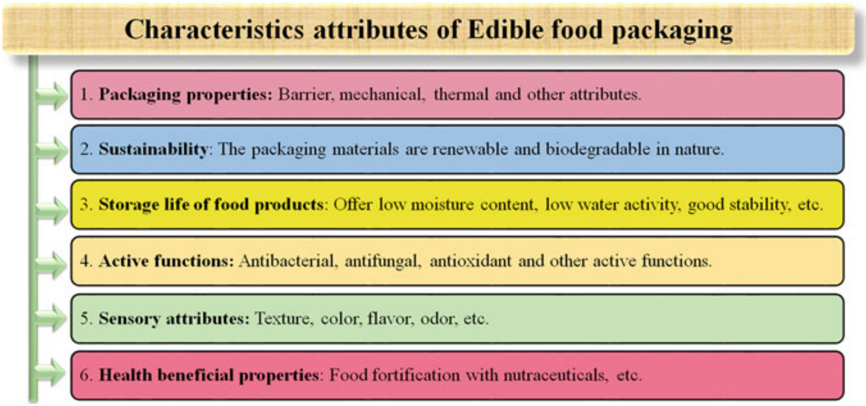
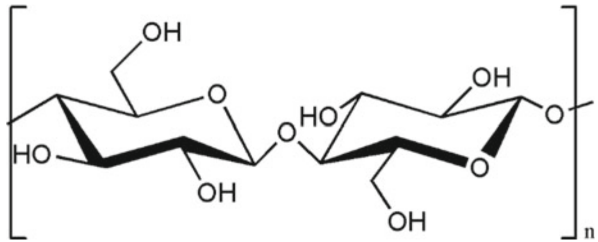
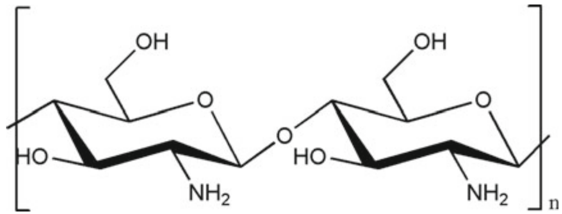
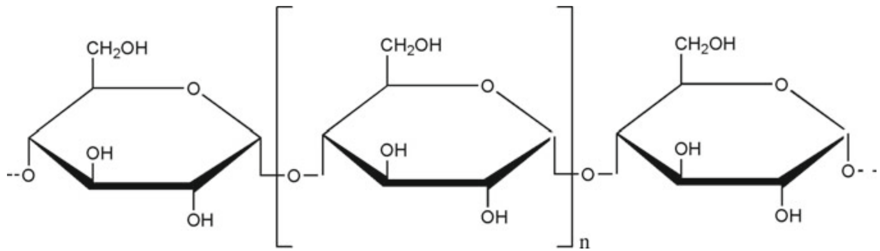


Fig. 2.2 Characteristics attributes of edible food packaging

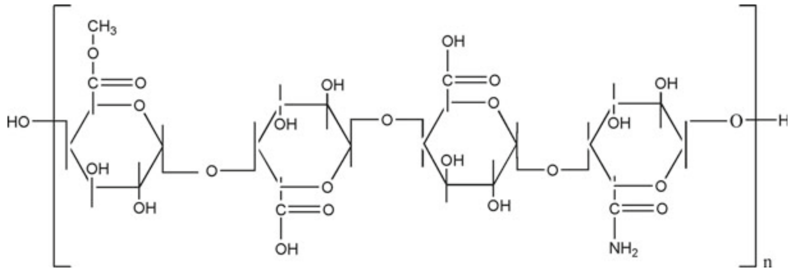
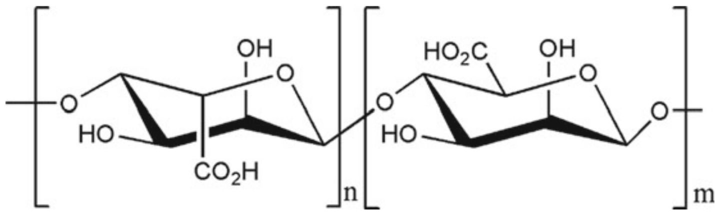
chitosan (CS)/chitin, hyaluronic acid, and chondroitin sulfate obtained from marine wastes such as cartilage, fish eyeballs, and crustacean shells); (ii) microbial origin: xanthan gum, dextran; (iii) marine origin: agar, alginic acid, carrageenan; (iv) plant origin: starch (ST), cellulose, gum arabic, gum karaya, and guar gum. Additionally, the polysaccharides are also classified into main three types such as storage polysaccharides (ST, glycogen, and inulin), structural polysaccharides (cellulose, chitin, pectin), and mucosubstances (mucopolysaccharides). The classes of polysaccharides based on structure include linear and branched polysaccharides. The linear polysaccharides include cellulose, amylose, algin, pectin, and the branched polysaccharides include amylopectin, gum arabic, galactomannans, xanthan, and xylan. The several kinds of polysaccharides are considered as potential candidates to be used in edible food packaging (edible films and coatings) as biocomposites and blends. The presence of hydroxyl groups and other hydrophilic moieties in the chemical structure of polysaccharides serves a significant role in the film formation. In this regard, the chemical structure of several polysaccharides and their derivatives are represented in Table 2.1. The polysaccharides are also used in both edible and non-edible food packaging preparation as matrix and filler materials. Additionally, the polysaccharide-based films exhibit better adherence property to cut fruit surfaces and also have good gas barrier properties [3]. However, the low water resistance property, mechanical property, and cost effectiveness are the main drawbacks of polysaccharide-based materials to be used in food packaging. The structure of polysaccharides can be tuned to improve their physicochemical properties by salt addition, solvent change, pH change, and chemical modification of hydroxyl groups. In this regard, the composite preparation of hydroxypropyl methylcellulose (HPMC) reinforcing CS/sodium tripolyphosphate (STPP) nanoparticles can provide an improved barrier, thermal, and mechanical properties [4]. The STPP acts as a cross-linking agent in developing CS/STPP-based nanoparticles via ionic cross-linking process (at lower pH). Besides, there are

Table 2.1 Targeted biopolymers for edible food packaging

Biopolymer	Chemical structure
Cellulose	
CS	
ST	

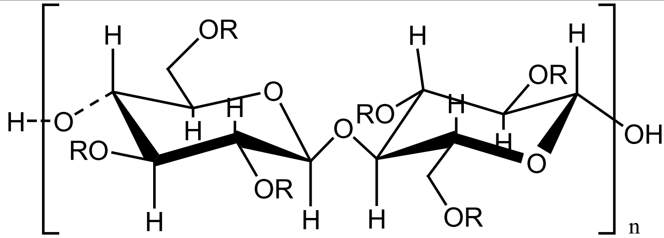
(continued)

Table 2.1 (continued)

Biopolymer	Chemical structure
Pectin	
Alginate	

CS: Chitosan; *ST*: Starch

Table 2.2 Chemical modification of cellulose for edible food packaging application

Chemical structure	Cellulose derivatives	R groups
	MC	H, CH_3
	EC	H, CH_2CH_3
	HPC	H, $[CH_2CH(CH_3)O]_nH$
	HPMC	H, CH_3, $CH_2CH(OH)CH_3$
	CMC	H, CH_2COONa

MC: Methylcellulose; *EC*: Ethylcellulose; *HPC*: Hydroxypropyl Cellulose; *HPMC*: Hydropropyl methylcellulose; *CMC*: Carboxymethyl Cellulose

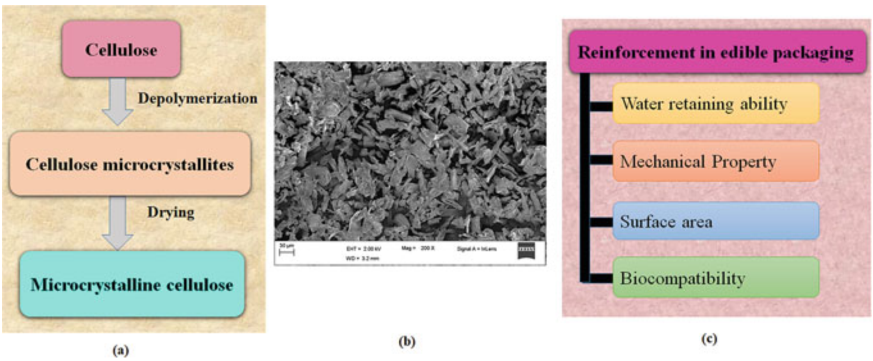


Fig. 2.3 FESEM micrograph of MCC

several strategies are applied to modify the properties of polysaccharides to act as a better reinforcing agent to develop composite materials.

2.3.1 Cellulose and Its Derivatives

Cellulose is a class of structural polysaccharide, consisting of β (1 → 4) linked D-glucopyranose units which are extracted abundantly from plant -based sources and algae-based sources. Cellulose is the most abundantly available biopolymeric material in earth and is considered a potential candidate in developing edible films and coatings [5]. In 1838, cellulose was isolated for the first time from plant matter by Anselme Payen, a French chemist [6]. In 1870, the first successful development of celluloid, a cellulose-based thermoplastic polymer was obtained by Hyatt Manufacturing Company. These are insoluble in various aqueous solutions due to their rigid crystalline structure imparted due to the presence of hydroxyl groups (OH) between and within the strands. When the hydroxyl groups on the glycosyl units are substituted with the bulkier groups, it helps in separating the polymer chains of the crystalline structure by breaking the H-bonding. Cellulose, when untreated is insoluble in water but when chemically treated with alkali and further reacts with propylene oxide, methyl chloride, etc., become water soluble. As represented in Table 2.2, the forms of cellulose derivatives are carboxymethyl cellulose (CMC), methylcellulose (MC), ethylcellulose (EC), hydroxypropyl cellulose (HPC), HPMC, etc. These forms of cellulose ethers are the polymer substances that are formed by partially substituting OH groups with ether functionalities. These forms are water soluble and are used to develop edible polymer films having properties of transparency, odorless, tough, and flexible. These are usually poor water resistant, however provide resistant to fats and oils. Further, their hydrophilicities are arranged in the order of HPC < MC < HPMC < CMC. Interestingly, cellulose derivatives in combination with nanostructured cellulose materials are also used to develop food packaging materials with better properties [7]. Besides, MCC is also extensively utilized for developing edible films and coatings in combination with other biopolymeric materials for improved attributes. The cellulose microcrytsallites from cellulose can be developed via depolymerization technique which on drying provide MCC (Fig. 2.3a). The FESEM micrograph of MCC has been represented in Fig. 2.3b. The MCC as a reinforcement has several attributes to be used in edible food packaging applications such as mechanical property, water retaining property, surface area, and biological property.

Methylcellulose (MC). It is obtained from cellulose derived plant-based sources such as cotton, jute, bamboo, and pulps of plants. These are more biodegradable in nature which is produced by chemical treatments using alkali concentrated NaOH solution and further treating with methyl halide. The produced films from MC have application in the food packaging industry due to their low cost, transparency, high strength, easy processability, excellent film-forming properties, etc. [8, 9]. These polysaccharides are composed of linear chains of β (1 → 4) glycosidic units consisting of methyl substituent replacing the all or part of the three hydroxyl groups on cellulose, which imparts the films with sufficient oxygen and barrier property [9]. For many years, MC has been used to produce fine chemicals for the pharmaceuticals, packaging, civil construction, petrochemical industry, paints, adhesives, cosmetic industries, and others [10]. Also, it has been observed that MC has many application as emulsifiers, viscosity, and flow controllers. Due to the hydrophilic nature of MC, it has been used as an active material to protect the food from oxidation or food spoilage and further, which improves the shelf life. Further, nanoemulsions (food-grade) based on cellulose, its derivative, and essential oils can be used to prepare edible packaging and others to improve the quality and shelf-life of foods, which further helps in the delivery of functional compounds and antimicrobial agents into the food products [11]. Otoni et al. reported the fabrication of clove bud or oregano oil emulsified MC-based films, which provide antimicrobial property against spoilage fungi and further help in maintaining quality and shelf life of sliced bread [12]. Interestingly, MC-based films are more effective to be used as a coating for deep fat fried potato strips in order to minimize the oil uptake in comparison with HPMC films [13]. The addition of plasticizers such as sorbitol with cellulose derivatives may provide additional benefits in comparison with individual cellulose derivative films. The combination of modifiers such as propylene oxide and methyl chloride can be useful to generate polysaccharides with multiple modifications, viz. HPMC. In this regard, edible films based on HPMC composites using microcrystalline cellulose (MCC) nanoparticles are also developed to provide improved moisture barrier properties in comparison with neat HPMC films [14].

Ethylcellulose. In the food packaging applications, researchers are now focused mainly on biodegradable films. Ethylcellulose (EC) is one of the cellulose derivatives consisting of repeating structure of anhydrous glucose units with ethyl ether groups replacing some of the hydroxyl sites. It has attracted attention in the food and pharmaceutical sectors. Kaur and Yadav et al. prepared polymeric films for food packaging by using various ratio of EC and polyethylene glycol (PEG) as a plasticizer. The addition of PEG at a different ratio to EC film provides tailored property, where the fabrication of films with EC: PEG in the ratio of 2:1 has good flexibility and collapsibility providing the suitability for food packaging application. Further, the EC and PEG-based films can also withstand a maximum load of 2.65 kN for a long time showing good mechanical stability [15]. The EC-based edible coating is helpful in reducing the moisture loss from packaged meat in comparison with the agar-based coatings (considerable thickness: 0.8–0.9 mm) [16]. Moreover, acetylated glycerol monostearate coatings are slightly more water vapor permeable in comparison with EC, whereas the specific coatings based on acetylated glycerol monostearate provide less oxygen permeability in comparison with EC films [17]. Furthermore, the association of several other biopolymeric materials such as proteins and lipids can be added to cellulose derivatives to develop composite-based films with improved properties [18]. Romero-Bastida et al. prepared zein–EC films with the aid of plasticizer stearic acid (SA), which showed the lowest water sorption property in comparison with pure EC and zein films. Further, the biodegradability behavior of zein and EC film shows significant changes, where 100% zein-based films provide highest biodegradability [19]. Moreover, EC, having the properties of water insolubility, excellent membrane forming ability, drug release property, is used as films and microspheres in pharmaceutical purposes [20]. Li et al. prepared blends of EC/konjac glucomannan blend films provide increased thermal stability, moisture resistance, tensile strength, and elongation at break in comparison with the konjac glucomannan films [21]. In the year 2010, Guiga et al. designed multilayered nisin-loaded EC/HPMC/EC films using lecithin, a surface-active plasticizers, which impart antimicrobial activity to improve product packaging property [22]. Moreover, EC-based delivery systems have been studied which could serve as a potential for producing healthy and low calorie formulations at the industrial level.

Hydroxypropyl Cellulose (HPC). HPC is another cellulose derivative used for developing edible films or coating besides MC, CMC, and HPMC [23]. It is particularly used as the main component for multicomponent dispersion-coating applications. It is also used for dispersion-coating formulations for sustainable packaging applications with excellent convertibility with a grease barrier. It acts as a barrier for oil resistance and also the presence of additives like talc reduces the stickiness of the coated layer. The casted films made from MC, HPMC, HPC, and CMC generally have moderate strength, flexible, resistant to oil and fats, transparent, tasteless, and water soluble, and are moderate barriers to oxygen. The edible coatings made out of CMC, MC, HPC, and HPMC for various fruits serve as a barrier to oil and moisture transfer.

Hydroxypropyl Methylcellulose (HPMC). HPMC, also known as hypromellose, refers to the group of cellulosic ethers in which the substitution of the hydroxyl groups with methyl and hydroxyl propyl groups is done with hydroxyl groups that are present in the cellulose ring. The hydrophilic, biodegradable, and biocompatible nature of the HPMC makes it applicable in various fields such as in drug delivery, dyes, cosmetics, adhesives, coatings, agriculture, and textiles. It is a potential replacement for various synthetic plastics because of its excellent film-forming properties, flexibility, biodegradability, transparency, and its gas barrier properties. It is soluble in polar organic solvents both aqueous and non-aqueous solvents. It has a distinctive solubility property in both hot and cold organic solvents. As compared to other cellulosic derivatives, HPMC possesses increased thermoplasticity and organo-solubility with a gelation temperature of 75–90 °C. Being biodegradable in nature, this type of polymer creates composites that are easily processable, eco-friendly in nature, and deliver desirable properties. Also, numerous studies have also been carried out to study the impact of various additives on the physicochemical properties of HPMC. The incorporation of antimicrobial agents such as nisin and potassium sorbate to tapioca ST and HPMC-based edible films provides an active packaging, where the combined use of ST and HPMC delivers increased elastic modulus and stress [24]. Additionally, the combined use of nisin and potassium sorbate is more advantageous in providing inhibitory spectrum. Further, studies based on edible films from HPMC with saturated and unsaturated fatty acids such as lauric acids (LA), myristic acid (MA), palmitic acid (PA), SA, and oleic acid (OA) provide tailored-made film properties in terms of microstructural and physical properties [25]. The addition of the film microstructure affects the several film properties including water barrier, mechanical, and optical property. The bigger lipid micellar structures are obtained in HPMC (aqueous) system when LA, MA, and PA are used in comparison with SA and OA. Besides, the HPMC films due to their hydrophilic nature have a shortcoming of providing lower moisture barrier properties. Thus, incorporation of lipids such as beeswax in the HPMC film increases the effectiveness toward water barrier properties, and it further helps to reduce the water vapor permeability (WVP), delivering an application of HPMC-lipid composite-coated papers in food packaging areas [26]. Polysaccharides and lipids-based coated papers are extensively used to develop

packaging in food industry. The addition of carrageenan, plasticizers, and carnauba wax emulsions can improve the properties of HPMC-based edible films such as rheology and water vapor properties [27]. Also, the moisture barrier of any HPMC film can be improved by incorporation of SA in the film-forming solution. For the preparation of a composite film with both hydrophobic and hydrophilic compounds, chemical modification improves the hydrophobicity of the film. In this manner, a homogeneous packaging film can be prepared. Again, the cross-linking of HPMC can be another approach to produce biodegradable packaging materials with improved water vapor barrier properties [28]. The addition of SA to HPMC films helps in providing improved quality of edible films with improved film moisture barrier properties [29]. The studies also revealed that the plasticizer type and proportions can influence the properties of HPMC/beeswax-based edible films [30]. Interestingly, the effectiveness of these coatings depends on the type of the fruit and cultivar. In pharmaceutical industry, it is mainly used as a hard outer coating for tablets. It protects the medicine against light, moisture, and shape distortion. Further, HPMC, PEG, and calcium lactate pentahydrate-based coating are also used to develop white film for pharmaceutical products [31]. The use of pharmaceutical coatings on tablets helps to protect the tablets against photolytic degradation and other environmental agents. HPMC is further used in pharmaceuticals as binder, film coating, film-forming material, controlled release agent, biological adhesive, etc. [32]. This specific biopolymer, being a hydrophilic biopolymer, is used for the development of oral-controlled drug delivery system, where the mathematical modeling of HPMC-based delivery system can also be developed [33].
On the other hand, HPMC in combination with other biopolymeric materials is used to develop water-soluble edible pouches, to deliver dry food products. Sebti et al. (2002), developed a bioactive edible food packaging material by associating nisin and SA to HPMC, where the molecular interactions are pH-dependent and by adjusting the pH to 3, there induced a high film inhibitory activity [34]. Further, HPMC bears certain limitations and drawbacks in the food packaging applications for having limited moisture resistance. Recently, researchers have investigated on enhancing the barrier properties of HPMC by incorporating cellulose fibers [35], fatty acids [36], and essential oils [37]. In the year 2010, Pastor and others developed HPMC-based composite films incorporating extracts of propolis which provide improved WVP in comparison with neat HPMC films and also provide colored film with antifungal activity [38]. However, in case of food, pharmaceutical, and paint industries, lacquers and varnishes are used as a barrier against moisture. Shellac is comprised of polyesters and single ester, which can provide protection against moisture and has great applications in the area of food industry and pharmaceutical industry [39]. In this regard, the HPMC/shellac-based composite films with emulsifiers SA and LA provide tailored-made mechanical and moisture barrier properties, where LA is a more effective emulsifier than SA [40].

Carboxymethyl Cellulose (CMC). CMC is a typical anionic polysaccharide, which is one of the most important cellulose derivatives. It is water soluble in nature and is used in the food, pharmaceutical, and cosmetic industry as an effective additive. CMC consists of chains of linear β(1 → 4) linked glucopyranose units with carboxymethyl group bound to hydroxyl groups. In addition, CMC contains a hydrophobic polysaccharide backbone and many hydrophilic carboxyl groups and hence shows amphiphilic characteristics. It is non-toxic, biocompatible, biodegradable, hydrophilic, and possess good film-forming ability. CMC has been used in numerous edible film formulations. It is generally used as a hydrophilic polymer coating to food products and has many areas of application in food industry [41]. The formation of covalently bonded CMC-casein complex can be achieved by electrosynthesis and are very pH stable. These complexes also exhibit good emulsifying properties and thermal stability. Also, the Maillard reaction may occur when proteins are mixed with carbohydrates at higher temperatures. Additionally, the development of edible films is based on CMC and soy protein isolate (SPI) blends and glycerol (as a compatibilizer). The films showed improved mechanical properties with increased CMC content and further provide reduced water sensitivity and tailored morphological behavior of the SPI films [42]. Also, the use of citric acid (CA) as a cross-linker for the development of CMC/polyvinyl alcohol (PVA)/aloe vera (AV) films has been done. The films have improved moisture barrier and mechanical property, and the use of AV acts against UV radiation. The developed films are eco-friendly and water-resistant active packaging film for meat products [43].

2.3.2 Chitosan

The another polysaccharide material, CS, is the second most abundantly available material after cellulose. It is developed by deacetylation (using sodium hydroxide) of chitin, which is availed from sea-based waste [44]. It is a flexible biopolymer derived from various renewable resources and is considered a sustainable material widely used for edible food packaging. Additionally, it is a non-toxic, functional, and biodegradable material used for varied applications. It is a linear polysaccharide containing (1 → 4)-linked 2-amino-2-deoxy-β-D-glucan. It also serves as a natural antimicrobial agent from animal origin and is insoluble in water and any other organic solvent. It is soluble in slightly acidic water below pH 6.3 producing viscous solution, which is preferable for some edible-coating approaches. The solubility of CS depends on the molecular weight of the polymer. Although a precise mechanism has not been yet generated for CS's antimicrobial mechanism. The several properties of CS may be imparted due to several reasons such as (a) electrostatic interaction between amine groups of the polymer and microbial cell membrane provides antimicrobial property; (b) it acts as a chelating agent; (c) it acts a penetrating agent through the microbial cell membrane and (d) modifies the cell surface and nutrient transport in the bacterial cells. In this regard, CS has antimicrobial properties offered by the polycationic nature of the molecule [45]. Vásconez et al. developed CS and ST-based edible coating and films to improve the quality of salmon muscle, where the addition of CS reduces the WVP and solubility of the films [46]. Additionally, CS-based blends with thermoplastic polymers are developed to attain humidity-resistant property [47]. CS having antimicrobial properties has attained great attraction in food packaging application, where the antimicrobial properties are affected by pH, intrinsic factors, CS-metal complex, etc. [48]. CS-based edible films or edible coatings are developed using CS blends or composites, which offer modified packaging properties to extend shelf life of food products [49].

2.3.3 Starch

ST, a kind of storage polysaccharide, is obtained from several renewable resources and consists of amylose (a linear polysaccharide) and amylopectin (a branched polysaccharide). In cereal kernels, ST is a constituent consisting of more than 60% of cereal kernels, which can be easily separated from other chemical components of the kernels [50]. Additionally, ST can be obtained from various other sources such as rice, oat, corn, wheat, maize, sago, potato, and cassava. [50, 51]. Based on the origin of ST, it may have varied shape, size, structure, and composition of amylose and amylopectin. This polysaccharide is naturally available, inexpensive, biodegradable, edible in nature, and a potential candidate for developing edible food packaging materials. ST is available as granules, which make them insoluble in water, where the constituent's amylose is water soluble and amylopectin is water insoluble. However, properties of ST are modified through formulating blends, composites, and plasticizers to obtain improved properties.

The ST-based packaging materials are developed employing two types of techniques such as wet and dry process. The wet process for developing ST-based edible films is generally preferred over dry process. The dry process involves extrusion technique, where the thermoplastic materials are preferred. The ST is mixed with plasticizer at high temperature to deliver the thermoplastic behavior. The addition of plasticizers and other biopolymers can offer tailored flexibility of ST-based films. As already mentioned, ST is insoluble in water, however, get solubilized in hot water, thus ST is dissolved at high temperature to prepare edible films or coatings, where the ST granules lost its semicrystalline nature at high temperature. In wet process of developing edible films, the biopolymer is applied to food products via dipping, spraying, or brushing [52]. The ST being hydrophilic in nature has a poor WVP. However, the WVP of ST films are dependent on temperature, thickness of film, plasticizers content, etc., which further varied in terms of intermolecular interactions [53]. ST can provide excellent oxygen barrier properties by tailoring the crystalline or amylopectin part of the polymer. On the other hand, the brittle nature of ST provides poor mechanical property, which can further be modified by developing composites or blends of polymers. In this way, ST-based blends and composites with added plasticizers are extensively utilized to develop edible food packaging materials for storing food products.

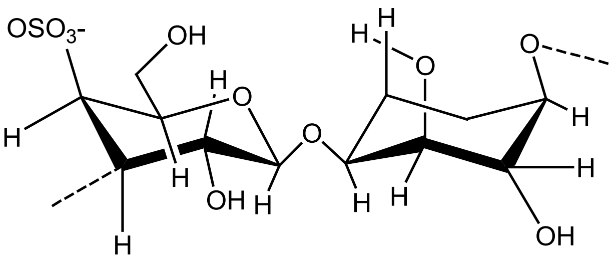
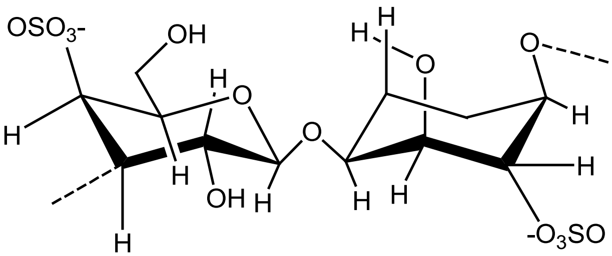
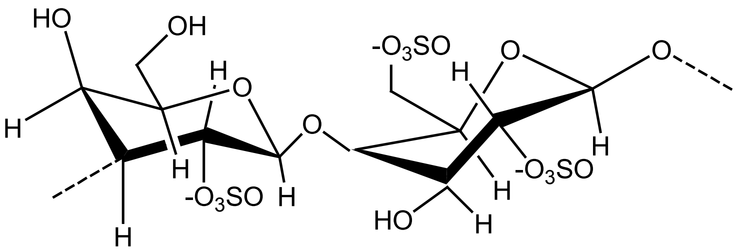
2.3.4 Pectin

It is one of the main components of plant cells and a citrus by-product. It is basically a by-product of agricultural crops such as apple (1–1.5%), citrus peels (30%), oranges (0.5–3.5%), and carrots. However, pectin is widely extracted from apple pomace and dried citrus peels. It is commonly classified into two categories based on the degree of methylation (DM), low-methoxyl pectins (DM < 50%) ,and high-methoxyl pectins (DM > 50%) [54]. It consists of repeating units of α-1,4-linked galacturonic acid and a major plant cell wall component. The pectin-based edible films have poor thermomechanical, barrier, and low water resistance properties, which decrease its use in food packaging applications. However, pectin with other components is widely used in combination with other materials. In this regard, the pectin and green tea powder-based edible coating on irradiated pork patty have a positive impact on improving the product quality [55]. Further, films developed from pure pectin promoted microbial growth as pectin is generally used as a carbon source by the microorganisms. As, pectin serves poor antimicrobial properties, so addition of antimicrobial agents such as oolong tea, green tea, and others can improve edible food packaging quality. In this regard, pectin and oregano essential oils based edible coating provide antifungal activity in maintaining the quality of tomatoes [56]. Further, pectin is also used as a gelling agent in developing jams and jellies and also stabilizing agents in dairy products. Edible active packaging made from pectin with essential oils has been reported as potential antimicrobial coatings for strawberries for enhanced shelf life [57]. Recently, pectin incorporated with nanochitosan in the ratio of 50:50 provides improved edible film properties in terms of tensile strength, water solubility, WVP, and others [58]. Nano-CS being the most natural antifungal agent that has been used along with the pectin. Manrich et al. produced edible films (casting technique) made from tomato cutin and pectin in various proportions which provide low water uptake and solubility and thermal and mechanical stability similar to tomato peels [59]. Thus, it is blended with natural polymers and hydrophobic compounds to get the desired property.

2.3.5 Carrageenan

Carrageenan is an anionic and linear sulfated polysaccharide obtained from red edible seaweed. According to the Food and Drug Administration (FDA), carrageenan and alginates are generally recognized as safe (GRAS) materials for edible films and coatings. It is a combination of several polysaccharides. Carrageenan is composed of repeating β-d-galactose units and 3,6-anhydro-α-d-galactose (both sulfated and non-sulfated) linked by alternating α-(1,3) and β-(1,4) glycosidic linkages. The three major forms of carrageenan are kappa (κ), iota (ι), and lambda (λ) with varied degree of sulfation [60]. The chemical structures of different types

Table 2.3 Chemical structure of different types of carrageenan

Type of carrageenan	Chemical structure
κ-carrageenan	
ι-carrageenan	
λ-carrageenan	

of carrageenan have been represented in Table 2.3. The carrageenan-based edible films and coatings are widely applied to various food products such as meat products, oily foods, dry solid foods, and food products [61]. In this regard, thermo-reversible carrageenan gels can be used as food coatings to retard moisture loss from enrobed food by acting as a sacrificing agent. These are natural hydrophilic polymers, and the film formation follows a gelation mechanism to form a three-dimensional network and a solid film after evaporation of solvent. It is applicable in desserts, milkshakes, and condensed milks, where carrageenan helps to form gels with increased viscosity. The edible films based on semi-refined κ-carrageenan with plasticizers (glycerol and sorbitol) provide improved mechanical (tensile strength and elongation at break) and oil barrier properties in comparison with neat semi-refined κ-carrageenan [62]. Thus, modified carrageenan is used to obtain good film properties, where the carrageenan is found mostly in the seaweeds of *Kappaphycus alvarezii* and *Eucheuma denticulatum* [63]. Additionally, the alginate and carrageenan are best used as a film coating for meat and meat products. The coatings generally prevent shrinkage, microbial attack, and surface discoloration by delaying the moisture transport. The fabrication of edible films based on κ-carrageenan and locust bean gum provide improved functionality, physical properties, and further deliver improved barrier and mechanical properties [64]. In this way, several polysaccharides including CS, carrageenan, and others are utilized to develop edible coating to extend shelf life of food products [65].

2.3.6 Alginate

Alginates are derived from seaweeds and deliver good film-forming property. This biopolymer exhibits unique colloidal properties and is used as a deliverable in pharmaceuticals. In this regard, alginate beads containing 5-fluorouracil can be used for the treatment of breast cancer [66]. It has been used extensively as an impression-making material in dentistry and prosthetics. Moreover, alginates having the properties of film forming, gelling, and thickening has attained a great interest to be used as a food ingredient [67]. It is also used in the food industry, for thickening soups and jellies and forms strong films which exhibit poor water resistance because of their hydrophilic nature. The alginate and apple puree-based edible films attributes can be modified using plant essential oils such as oregano oil, lemongrass oil, and citral oils., where the essential oils provide antimicrobial property to the films [68].

2.4 Lipids in Edible Food Packaging

These are edible polymers that consist of fats, glycerides, and acetylated glycerides (monoglycerides, diglycerides, triglycerides), natural wax, and surfactants. The fats and oils in edible films and coatings include animals and vegetable oils and fats. The vegetable fats and oils are coconut oil, peanut oil, palm oil, cocoa, milk butter, etc. These are meant mainly for blocking moisture with their low polarity. The films with lipid-based materials are more hydrophobic and brittle in nature. So, these films are associated with the polymer matrix and other cellular derivatives to improve the mechanical strength. The biomaterials polysaccharides, proteins, and lipids with their combined form are used to develop edible-coating materials [69]. In this way, edible films based on ST-MC-lipid blends are used to improve the shelf life of dry bakery products, where the films properties can be modified according to the blending levels, concentration, plasticizers, etc. [69]. Additionally, the lipid-based materials can also develop a continuous layer over hydrophilic materials such as polysaccharides or proteins [70]. On the other hand, lipid can be used as a dispersed phase in the matrix materials to develop edible films and coatings [70].

The properties of lipid-based films are based on several factors such as type of lipid, volume fraction, drying condition, and processing type. Moreover, lipid is extensively used materials with polysaccharides and proteins for attaining improved moisture barrier properties [71].

2.5 Proteins in Edible Food Packaging

Proteins are linear randomly arranged copolymers made from different amino acid monomers. In the early twentieth century, these materials attracted many textiles and plastic industries. There are different classifications of protein based on sequence of amino acid, shape, and composition. Further, the different proteins are random coil (milk casein), fibrous (collagen), or globular (corn zein, soy protein, wheat gluten, whey protein), etc. [72]. The fibrous proteins are completely extended and are linked through H-bonding in different parallel structures to form fiber like structure. Similar to polysaccharides, protein films are also susceptible to more WVP. The limitation is due to the presence of hydrophilic plasticizers, viz. glycerin and sorbitol to impart flexibility to the films. The proteins and polysaccharides along with the lipids can form a homogeneous hydrocolloid matrix that can overcome the mechanical strength and WVP of the film. Researchers have found, fatty acids such as saturated fatty acids (LA, SA, and PA), and unsaturated fatty acids (oleic acid) provide tunable film properties of sodium caseinate in terms of water vapor barrier properties, mechanical properties, optical properties, etc. [73]. The intrinsic properties of proteins such as amino acid content, hydrophobicity and hydrophilicity, crystallinity, and molecular size make them exceptional starting materials for developing films and coatings. However, the extrinsic factors include processing temperature and its relative humidity, pH, ionic strength, salt type, shear, and pressure. The different protein sources used for developing edible films and coatings include whey, soy, wheat gluten, keratin, casein, etc. [74]. The protein-based films and coatings are biodegradable and compostable. These kinds of films serve as a source of nitrogen as they degrade on soil. These can be of plant and animal origin, and the films are generally prepared by combining protein, plasticizer, and solvent. The following are some of the proteins, which are widely used for developing edible films.

2.5.1 Animal Proteins

Milk Protein. This comprises whey protein and casein protein, and the casein contains 80% of the milk content, which comprises α, β, and κ components [75]. The α and β casein are calcium ion sensitive and are unable to participate in disulfide bond formation and cross-linking. These provide better barrier property but are rigid in nature. The obtained casein solution is generally treated with alkali to attain pH 7 and dried for further treatment. The whey protein is a by-product obtained from cheese-making process. It is prepared by precipitation of casein protein in milk at 4.6 pH and temperature of 20 °C.

Meat Protein. There are three types of meat proteins, viz. sarcoplasmic, stromal, and myofibrillar. The stromal proteins include collagen and elastin. The collagen is a by-product of meat processing. These are fibrous protein obtained from connective tissue, tendons, skin, bones, and vascular system. Gelatin is produced by treating collagen with acid or alkali solution. The pure gelatin formed is brittle in nature, and it forms a film once it is dissolved in hot water. Additionally, gelatin is extensively used in several areas including edible films, food, cosmetic coating, pharmaceuticals, etc. [76]. It is served in the food industry because of its good gelling property. It also offers excellent foaming property that helps in making a good edible film. It has been used as a coating for food and pharmaceutical industries for years, where the properties of gelatin can be modified using other biopolymeric materials such as CS and cellulose derivative.

Silk Protein. There are several silk-based proteins, among which the sericin is a glue-based protein produced by silkworm *Bombyx mori, Antheraea assamensis,* and many other sources. This type of protein is edible in nature, antibacterial, and it is oxygen and UV resistant. Studies have revealed that the incorporation of these proteins into some other macromolecular materials helps in enhancing the mechanical property of the blended system [77]. Wang et al. developed whey protein and sericin blended films, where sericin protein provides tunable properties of whey protein films in terms of WVP, solubility, and mechanical property [78]. The WVP of sericin and whey protein blended films has found to be reduced due to the sericin content. Also, studies have reported the preparation of silk fibroin-based thin films. By using a postprocessing water-based method to control the protein polymorphism which enable the modulation of diffusion of gases such as oxygen, carbon dioxide, and WVP, can be used as a parameter to confirm the freshness of the food and by using the dip-coating technique on fruits like strawberries and bananas, it is found, that the silk fibroin enhanced the shelf-life of the food at room temperature [79].

2.5.2 Plant Proteins

Wheat Protein. These constitute a major part of the bread-making industry. Wheat gluten, which contains a small proportion of charged amino acids (lysine, histidine, arginine) and a high proportion of non-polar amino acids, aggregates easily due to hydrophobic interactions. There are four main wheat protein fractions, based on their solubility: albumins (water soluble), globulins (dilute salt soluble), gliadins (soluble in 70–90% ethanol), and glutenin (insoluble under any conditions). The

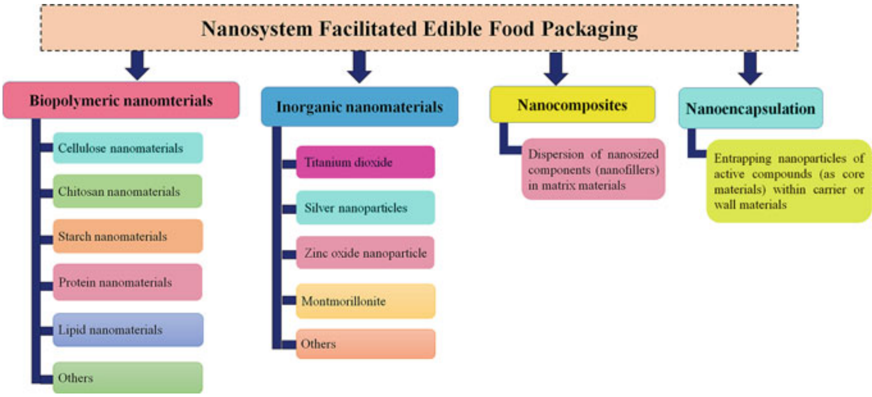


Fig. 2.4 Several nanosystem in edible food packaging

properties of wheat gluten-based edible films can be tailored using plasticizers such as water and glycerol, where the glycerol helps to improve the film extensibility [80]. Glutenin and gliadin are the major proteins making up 47 and 34% of the total wheat protein content, respectively [81]. Gliadin contains intramolecular disulfide bonds which are responsible for film formation.

Soy Protein. It is basically extracted from soya beans (38–44%). It comprises a mixture of globular proteins. It contains glycinin which is used as a gelling agent and provides film-forming property. These types of films are superior in film making as compared to lipids and polysaccharides based on their gas barrier property. Additionally, soy protein-based films can be modified using acids and oils [82]. In this regard, the use of lactic acid in developing soy protein-based films can improve the hydrophobicity and maintain the UV barrier properties.

Corn Zein. It is a polyamine and a main component extracted from corn. These are used for edible films and coatings. These are insoluble in water. The two major parts of zein are α and β zein. These are glossy, tough, and grease proof. They show low WVP as compared to most other agriculturally based protein films. It has been commercially used as a coating agent for pharmaceuticals [83].

2.6 Other Materials in Edible Food Packaging

The various materials such as fruit purees, pomace, and peel are also used to develop edible food packaging materials. Fruit purees are extracted from apple, oranges, banana, pear, etc. These are mainly composed of cellulose and pectin, which provide less flexible films. The flexible films using the specified materials can be fabricated by adding plasticizers, which makes it mechanically stable and improves the water barrier property. Also, fruit pomace is a cellulose extract from fruit juice industries used for making edible films. It also imparts flavor and color to the food. The development of edible films based on mango puree reinforced with cellulose nanofibers forms a fibrillary network within mango puree, which further delivers improved mechanical properties [84]. Another example, the carrot puree films are brittle and rigid in nature, so the addition of plasticizer makes the film more flexible and resistant to oxidation. However, several fruit purees such as peach, apricot, pear, apricot-based edible films can provide tailored permeability properties at different storage conditions [85].

2.7 Overview of Nanosystems Facilitated Materials in Edible Food Packaging

The nanosystem facilitated edible food packaging has attained great interest in developing edible food packaging to combat the available packaging materials with additional benefits. As shown in Fig. 2.4, the nanosystems in edible food packaging commonly include the use of biopolymeric nanostructured materials, inorganic

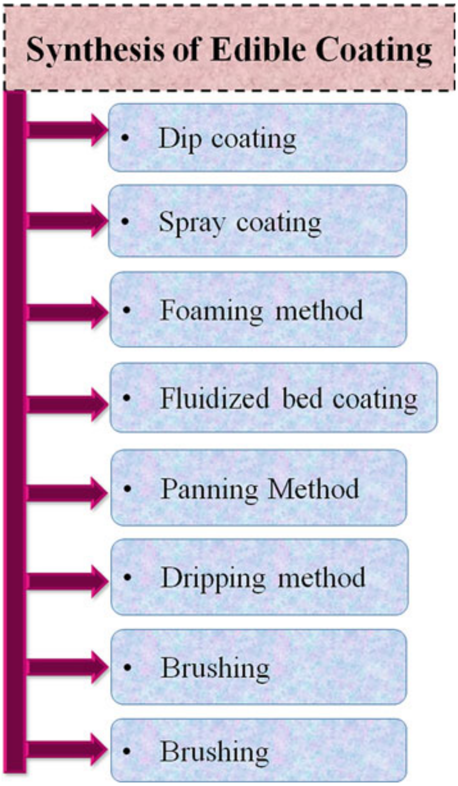


Fig. 2.5 Approaches for developing edible coating on food products

nanomaterials, nanocomposites, nanoencapsulation, nanosystems, etc. The extensively used biopolymeric nanostructured materials in food packaging include cellulose, CS, ST, protein, and lipid-based nanostructured materials [86]. The nanostructured materials are available in different sizes and shapes such as nanocrystals, nanofibers, nanoparticles, and nanotubes. Additionally, several inorganic nanomaterials also used to develop edible food packaging, where some of the inorganic materials deliver antimicrobial, antibacterial activity to improve the shelf life of food products. The nanocomposites are defined as the dispersion of nanoscale materials (dispersing materials) in matrix materials (continuous phase), where developing nanocomposites provide improved properties in terms of barrier, mechanical, thermal, and optical properties. Additionally, nanoencapsulation is another technique to entrap active compounds using carrier materials such as gum arabic, ST, and maltodextrin.

2.8 Synthesis Strategies to Develop Edible-Coated Food Products

As mentioned in Fig. 2.5, the several methods used for developing edible-coated food products include dipping, spraying, fluidized-bed coating, dripping, foaming, panning, brushing, etc. In wet processes of developing edible coating, liquid forms of coat materials are used to coat regular and irregular products via dipping, spraying, electrospraying, and others followed by drying. Additionally, the selection of a particular coating method depends on several attributes such as surface

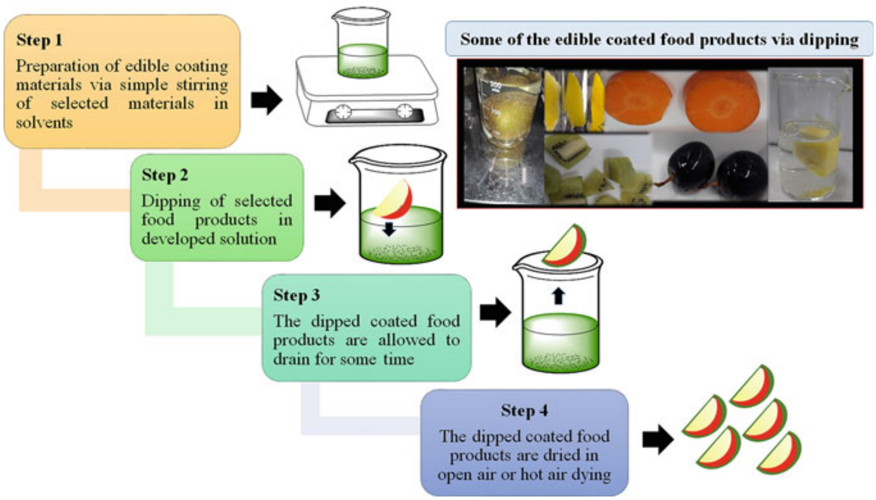


Fig. 2.6 Processing steps in developing dipping-based edible coating

Table 2.4 Development of edible coating on food products using dipping method

Sl no.	Materials	Method of edible-coating development	Targeted food product	References
1	1.5% w/v CS solution	**Dip coating** Dipped in prepared solution for 10, 20, 30, 40, 50, or 60 s Draining of solution: 180 s	Frozen fish samples	[88]
2	**Formulation 1 (F1):** CS, glycerol, acetic acid **Formulation 2 (F2):** Sodium alginate, glycerol **Formulation 3 (F3):** Soy protein isolate, glycerol	**Dip coating** Coating time for several formulations: F1: 20 s F2: 20 s F3: 15 s	Mozzarella cheese	[93]
3	Polysaccharide-based edible coating Sodium alginate and pectin	Dipping time: 2 and 4 min Removal of excess coating solution: 1 min	Sapota fruit	[101]
4	Pomegranate juice CS Acetic acid ZEO	**Dip coating** • Dipped in the coating solution for 2 min and draining • Dipping for second time (2 min) and drained for 5 h at 10 °C	Chicken breast meat	[91]
5	CS Oregano oil under MAP	**Dip coating** • Dipped in CS solution for 1.5 min • Excess solution was drained immediately after dipping • Then oregano oil is applied to the samples	Chicken breast meat	[92]
6	Alginate, pectin, gellan, calcium chloride, antibrowning agents (N-acetylcysteine and glutathione)	• Dipping of cut pears in the polysaccharide solution for 2 min • Draining of excess solution for 1 min • Dipping in calcium chloride solution with antibrowning agents for 2 min	Cut pears	[99]
7	Soy protein isolate HPMC Olive oil Potassium sorbate	**Dip coating** • Washing of fruits in distilled water • Washing in sodium hypochlorite solution (disinfection) • Dipping of fruits in the solution for 2 min	Fresh pear fruits	[95]

(continued)

Table 2.4 (continued)

Sl no.	Materials	Method of edible-coating development	Targeted food product	References
8	Silk fibroin	**Dip coating** Dipping time is 10 s (for 1, 2 and 4 times) Drying conditions: 22 °C, 38% RH, 4 h	Strawberry and banana	[79]
9	Sodium alginate Trans-cinnamaldehyde encapsulate powder (antimicrobial agent) Calcium lactate Pectin	Layer-by-layer dip-coating method (Each dipping for 2 min and allowed for draining 2 min) • Dipping in alginate and antimicrobial solution for 2 min • Dipping in calcium lactate solution • Dipping in pectin solution • Dipping in calcium lactate solution	Fresh-cut watermelon	[89]

CS: Chitosan; *ZEO*: *Zataria multiflora* essential oil; *MAP*: Modified atmospheric packaging; *HPMC*: Hydroxypropyl methylcellulose

property, selected coating materials (rheology and other property), and adhesion property. However, the stability between targeted food products and coating materials is essential to develop a good coat on food products. The layer by layer coating is another approach to improve the shelf life of food products, where the coating materials can be applied simultaneously or sequentially on food products. Further, the selection of processes also depends on large-scale coatings development and required time, where a discussion related to available techniques for developing edible coating has been made in the below sections.

2.8.1 Dip-Coating Method

The dip-coating method is extensively used laboratory scale approach for developing edible coating on food products, which provide several beneficial approaches in comparison with other available techniques such as simple, cost effective, and develop uniform coverage on both regular and irregular food products. The several processing steps for developing edible coating using dipping are represented in Fig. 2.6, which involves dipping of the selected food products in the developed edible-coating solution. However, to develop a dipping-based edible coating on fruit and vegetables, viscous solutions are preferred [87]. The dipped food products are further allowed to drain the excess materials from the food products and then dried in the open air or hot air dried to solidify the coating on food products followed by storage in specific conditions. This kind of coating process is commonly used to develop a uniform coating on food products. The coating thickness on food products is a critical parameter to consider in edible-coated food products, where the coating thickness on food products via dip-coating treatment depends on various factors such as dipping time, product temperature, and temperature of prepared coating materials. [88]. However, the controlling of processing conditions and continuous production of dipped coated food products are generally the shortcomings in developing dipped coated food products. Moreover, the dipping method involves the direct contact of the selected food products and coating solution, which may lead to microbial contamination in the coating solutions. In dip coating, the fruit products are washed using distilled water, disinfecting agents to avoid contamination before dipping into the coating solution. Further, the layer by layer coating can also be developed using dip-coating method, where the selected food products are dipped into different coating solutions followed by draining the excess solution after successive dipping [89].

The use of dip-coating method has been applied for developing coating on several categories of food products such as fruits and vegetables, meat and meat products, fish and fish products, and milk products. [88–93]. In this regard, the development of edible coating on several fruit products using dip-coating method has been represented in Table 2.4. The edible coating using dip coating can be given on whole (apple, pear fruit, strawberry) [94–96] and cut fruits such as apple slices [97], cut pineapple [98], fresh-cut pears [99], fresh-cut melon [100], fresh-cut kiwi fruits [100], and others, which are widely edible coated using dipped coating methods. Further, edible coating using dip-coating method is also developed by dipping in different solutions such as edible coating of cut pears via dipping in polysaccharide solution (first dipping) and calcium chloride solution with antibrowning agents (second dipping) [99]. The use of antibrowning agents can control the enzymatic browning and retard microbiological degradation of fruit products. The dipped coating of chicken breast meat can be obtained by first applying CS solution and then oregano essential oils under modified atmospheric packaging (MAP) conditions can retard lipid oxidation and significantly affected mesophilic count, lactic acid bacteria, yeast-molds, etc. [92]. The dip-coating of meat samples using CS, glycerol, tween 80 and *Zataria multiflora* essential oil (ZEO) is reported to perform, where development of edible coating on meat samples followed several steps such as (i) dipping in coating solution for 2 min, (ii) draining of excess coating solution from the samples, (iii) dipping again for 2 min, and (iv) drained for 5 h at 10 °C [91].

2.8.2 Spraying-Based Edible Coating

The spraying approach for developing edible coating delivers uniform coating on food products, with well-controlled processing as the product and the coating solution are in direct contact due to the spraying action. The use of CS-based preharvest spraying and postharvest coating on fruit products are beneficial to provide improved shelf life [102]. Among the available approaches utilized for developing edible coating, spray-based edible-coated food materials are available commercially. Additionally, a uniform thickness can also be developed on food products, where the spraying-based coating is suitable for low viscous solution. The spray-based edible coating is more acceptable when the coating is applied to a particular side of food products. The coating solution is sprayed on the food products by controlling the final drop size (drop size distribution up to 20 μm), which further depends on spray gun type, temperature of the nozzle, flow rates of air and liquids, drying conditions, etc. The spray-based edible coating is one of the most popular edible-coating methods applied for fruits and vegetables using atomizers and high-pressure spraying systems [103]. The processing steps in spray coating includes several steps such as (1) formation of droplets through atomization: This process begins by pumping coating materials through a nozzle; (2) drop impact spreading: This process involves spraying the drops of coating on the food surface, where based on the interaction between materials and food surface the drop may splash, rebound, or deposit on the food; (3) secondary leveling of droplets; (4) consolidation; and (5) drying of coating layer. The several wetting behaviors of drops onto food products or impacts of drops on food surface (such as banana, eggplant, purple cabbage) follow several stages such as kinematic phase (depends on the kinetic energy of the drops), spreading phase (the drop has a thin film shape), retraction, and relaxation. However, the coating solutions are optimized based on wettability (represents ability of coating materials to spread on a solid surface), adhesion forces (related to spreading the liquid on food surface), and cohesion forces (causes liquid to contract on food surfaces) [65]. Additionally, the control of drop impact on food product depends on several properties such as physico-chemical properties of liquid (surface tension, temperature, viscosity, impact direction, kinetic energy of drop; surface roughness of food, surface energy of food, surface temperature of food, air temperature, and pressure) [104, 105]. The spraying-based edible coating has several beneficial attributes such as provide uniform coating, thickness control, multilayer of coating, continuous coating, and temperature control. However, operating cost is also a crucial parameter in selecting the nozzle type. The limitations of spray coating are further related to surface distribution and transfer efficiency. The development of spray-based edible coating depends on spray conditions such as product surface, spray surface, spray time, and distance between pneumatic nozzle and product. In this regard, to obtain a specific thickness, the several spray conditions need to be standardized. The spray-based edible coating on food products is a widely used technique to develop coating at large and continuous scale.

Spraying is used to develop an edible coating on different food products such as cheese, meat, and meat products, dry bakery products, fruits (strawberry, mandarin oranges, grapes, etc.). [69, 93, 103, 106]. The development of spray-based edible coating on a commercial cracker is obtained via developing coating on upper and lower surface separately. The emulsion-based coating is applied on the upper surface followed by drying at 60 °C for 2 h. The spraying of emulsion-based coating consisting of ST, MC, and soybean oil has been obtained at a pressure of 2 bar at 70 °C [69]. The development of antimicrobial edible nanocoating using spraying techniques on fruit products can enhance the product life, where the antimicrobial nanocoating is developed using supramolecular metal-organic coordination complex of ferric ions and tannic acid [106]. In this process, the spraying of tannic acid and ferric ions is applied sequentially and simultaneously on fruit products. In sequential spraying, the mentioned coating materials are applied to food products sequentially, whereas, in simultaneous spraying systems, the coating materials are applied to food products simultaneously, which is generally employed for bulk scale coating. Spray-based methods can develop a uniform coating on food products, further, bakery products require the formation of coating directly on food product, which can be developed using spray-based coating. The liquid droplet size depends on atomization pressure, where smaller sizes of liquid droplets can be obtained at higher pressure. The formulation of edible coating (via spraying technique) on grapefruits are obtained with the hydrocolloid materials such as HPMC, κ-carrageenan, glycerol, and cellulose nanofibers [103]. The spraying application of grapefruits is also optimized in terms of thickness of coating and percentage of coating and independent variables: liquid suspension flow, air pressure, and height of impact as 1–5 L/h, 50–200 kPa, and 0.3–0.5 m, respectively [103]. In spray-based technique, the optimized conditions for suspension flow rate, air pressure, and spray nozzle height are 1 L/h, 200 kPa, and 0.5 m, respectively, where coating thickness between 24.2 ± 0.9 and 38.5 ± 1.4 μm can be obtained. Further, the physical properties of liquids such as viscosity, density, surface tension, and others are different, thus the droplet formation and generation are different depending on the specific liquids.

2.8.3 Electrospraying-Based Edible Coating

Electrospraying technique is an efficient approach to develop an edible coating on complex food products and further help to reduce the processing cost in industries [107]. In the electrospraying technique, fine droplets of edible materials are obtained on account of electrostatic forces, which produce charged liquid surfaces. The electrospray process has an ability to develop fine droplets with a narrow size distribution of 0.1–1000 μm. The fine droplets provide thin coatings on food products. The electrospray technique is used to increase the shelf life of minimally processed food while maintaining the sensory properties. The electrostatic spraying method has many benefits over other common spraying techniques such as controlled droplet size, reduced waste of coating materials, increased droplet coverage, and develop homogeneous distribution. However, the electrostatic spraying approach is more preferred in comparison with non-electrostatic or conventional spray-based systems in delivering higher transfer efficiency and coating uniformity [107, 108]. The use of electrostatic spray-based approach for developing coating

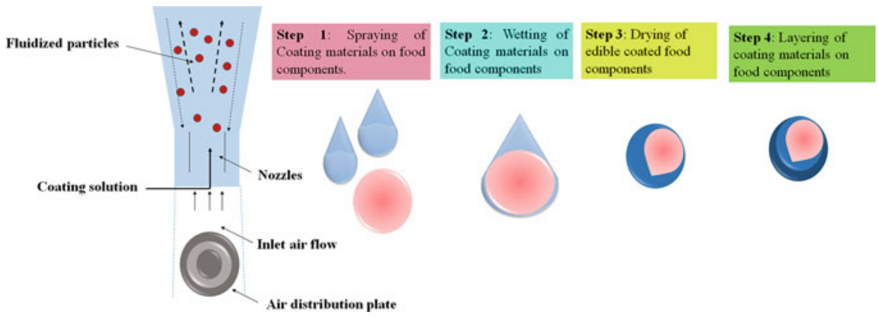


Fig. 2.7 Processing steps in developing edible coating using fluidized bed drier

using materials alginate with carvacrol, and methyl cinnamate on strawberry fruits, helps in maintaining fruit firmness, weight loss, retain color, and others [108]. The sunflower oil and dark chocolates can be used to develop moisture barriers-based edible coating on hygroscopic tablets via electrospraying technique, where electrospraying of sunflower oil offers smaller droplets having higher charge to mass ratio [107]. Moreover, a study reports the use of electrospray-based technique to develop an edible coating on different food models such as apple slices and candy tablets, where chocolate and water-in-oil emulsion-based coating have been developed. The emulsion and chocolate-based coating help in reducing browning and water loss in apple slices [109]. Lipids are extensively used materials for developing electrospraying-based edible coating, however, lipids are non-conductive and not suitable for electrospraying [110]. In this regard, lecithin is an ionic surfactant and can be used to increase the conductivity of targeted lipid materials to be used for electrospraying. Further, emulsion-based edible coating is developed using electrospraying technique for food products [110, 111].

2.8.4 Other Methods

The other methods applied for developing edible coating on food products include foaming, brushing, dripping, fluidized bed coating, panning, and others. The foaming is an approach to develop emulsion-based coating, where foam-based edible-coating materials are developed using a foaming agent. The foam is also developed by blowing compressed air in the applicator tank. To achieve uniform distribution of foam, tumbling action is employed to developed foam and the foams are applied to moving food products. Further, brushes are used to attain a uniform coat of emulsion over the surface of food products. Interestingly, the drying of the developed edible-coating materials on food products requires less time. The application of edible coating on food products has been developed using brushing technique on several food products such as fruits and vegetables and meat and meat products. However, the brushing method of edible coating is applied to food products, where the moisture loss is a problem. In this regard, the AV gel matrix with gelling agents is applied to develop an edible coating on fresh fruits and vegetables via brushing approach [112]. Further, carrageenan, CA, and cinnamon oil are used to develop edible coating on chicken filets employing brushing approach [113]. Edible coating is further developed on chopped papaya fruits using clove oil using brushing technique [114]. Dripping-based edible coating is suitable for agro-based products, where coating can be applied on the surface directly or brushes can be used to apply the same. However, good coating is obtained, when tumbling action is applied over several brushes. The dripping-based edible-coating approach is the cost effective. In enrobing-based edible coating, the developed sticky coating solutions are applied to flow vertically to the food products, where the products are edible coated by the action of viscous and gravitational forces. In enrobing method, viscosity of the coating solution is a crucial parameter in maintaining good food quality and weight of the food products. The enrobing is better suited for developing edible coating on flat food products. Enrobing based edible coating on chocolate, meat industry, and milk products such as cheese are developed. The enrobing-based coating processes are generally used in chocolate industries using enrobe-based processes. The fluidized bed drier (FBD) is considered as a potential edible-coating approach to develop thin layer of edible materials on dry particles of small sizes and very low density. The several processing steps to coat food components have been represented in Fig. 2.7, which include spraying of coating materials on food components (step 1), wetting of coating materials on food components (step 2), drying of coated food components (step 3), and layering of coating materials on food components (step 4). Moreover, the several attributes of functional food materials such as flavors, preservatives, and fortifiers can be

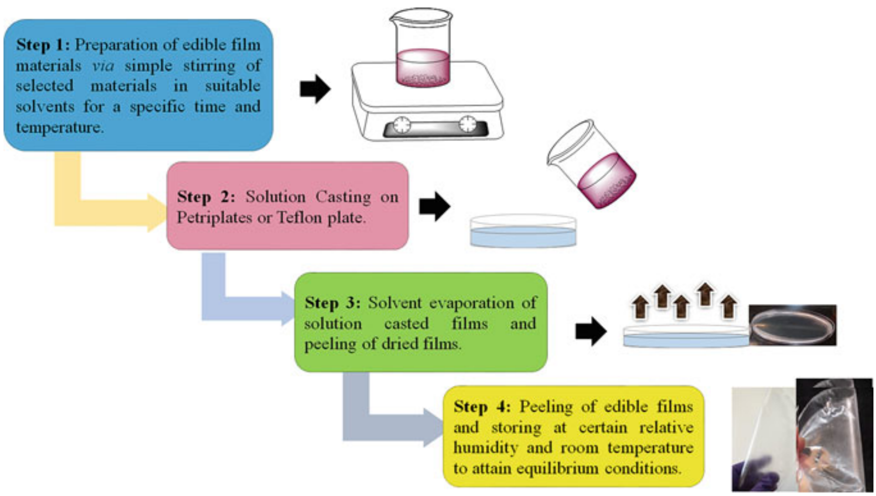


Fig. 2.8 Processing steps in fabrication of edible films

improved, which helps to improve texture and provide easy handling using FBD-based coating.

2.9 Synthesis Strategies to Develop Edible Film

The development of edible films is obtained via wet and dry processes. The wet processes of developing edible films include solution casting process, where the film-forming solutions are prepared by dispersing or solubilizing the biopolymers in a film-forming solution. On the other hand, the dry process for developing edible food packaging includes extrusion, where the biopolymer should possess thermoplastic properties. In this regard, some polymers can attain the properties of thermoplasticity by adding plasticizers and heating above the glass transition temperatures such as thermoplastic ST. However, the extruded films having the disadvantages of wrapping irregular food products, wet processes can be advantageous.

2.9.1 Solution Casting Methods

As shown in Fig. 2.8, the edible films using solution casting method are developed following several steps such as (1) dispersion or solubilization of selected biopolymers in solvents for a specific time; (2) casting of solution on pertiplates or Teflon plates, where before casting the solutions are kept for some duration to remove air bubbles; (3) solvent evaporation of solution casted films under hot air drying or open air; and (4) finally, peeling of the films followed by storage to obtain equilibrium conditions. The developed films are used to wrap food products or can be used as a sandwich material within food products. In fabricating solution casted edible films, the solvents for films materials are based on the properties of biopolymers (solubility), such as CS is soluble in acidic media (1% w/w acetic acid solution) [98], whereas the ST is soluble in hot water [115]. Further, the optimization of processing conditions for fabricating edible films can provide improved film properties. In this regard, the fabrication of edible films using cassava ST, glycerol, agar, and span 80 having concentrations of 1–3 g, 0.5–1.0 ml, 0.5–1.0 g, and 0.1–0.5 ml, respectively, are optimized using Box–Behnken experimental design to obtain second-order polynomial models for barrier and optical properties [116]. Further, the modeling of mechanical properties (tensile strength, Young's modulus, elongation, puncture force, puncture deformation) for edible films based on cassava ST, glycerol, agar, and span 80 is developed by solution casting technique [117]. The emulsion-based edible films are also developed via solution casting process and used to protect food products, where animal waxes and plant waxes are extensively used lipid materials for developing emulsified films [118]. Further, the attributes of glycerol plasticized gelatin films (developed by casting technique) can be improved using cross-linking agents such as dialdehyde CMC [119]. In this regard, the properties of edible films are improved preparing composites and blends of biopolymers, using plasticizers, cross-linking agents, etc.

2.9.2 Extrusion Technique

The edible films are also focused to develop by extrusion followed by compression molding and blown films. The application of extrusion-based edible coating mainly depends on thermoplastic properties of the utilized materials and is considered as the well-utilized method for developing edible coatings for industrial application. The development of edible films using extrusion technique depends on several processing conditions such as extrusion temperature, concentration of biopolymers, and plasticizer content. The edible films based on fish gelatin are prepared using extrusion (twin screw extrusion at 110 and 120 °C) followed by compression molding (80 °C) [120]. In this regard, the extrusion and compression molding processes are more feasible to develop commercialized edible films in comparison with solution casting process. The edible films based on glycerol plasticized sodium caseinate are developed using twin screw and blown film extrusion methods, where the films can be produced in a large scale with no surface defects [121]. Moreover, active edible packaging based on plasticized sodium caseinate containing lysozyme can also be prepared using blown film extrusion method [122]. However, the processing temperature and glycerol have a great influential effect on lysozyme stability. The natural fiber of pectin and food hydrocolloids-based antimicrobial edible films are developed using extrusion technique followed by blown film or compression molding, where mild processing conditions are beneficial to protect bioactivity of antimicrobial agents [123]. The extrusion of pectin and gelatin/sodium alginate for developing blends-based edible films or casings can be tailored using corn oil or olive oil [124].

2.10 Conclusion

From very early days, the available biopolymeric materials are a potential candidate to develop edible food packaging for its noteworthy characteristics such as edible, biodegradability, availability, easy processing, and others. The biopolymers used for developing edible food packaging include cellulose and its derivatives, ST and its derivatives, CS, and its derivatives, protein and their modified forms, lipid, etc. Interestingly, the nanosystem-assisted edible food packaging has attained great interest in terms of using biopolymeric nanostructured materials, inorganic nanofillers, polymeric composites, and others to provide improved health benefits and shelf life of food products. The edible food packaging is developed as edible coatings and edible films. The fabrication of edible-coated food products generally follows three processing steps such as the development of a coating solution, application of the coating solution on the food products, and drying to solidify the coating materials on food products. Similarly, the development of edible films can be obtained via solution casting (laboratory scale approach), and extrusion (large-scale approach for commercialization) based on the required packaging category. However, the selection of synthesis approach for edible food packaging development also depends on the targeted materials which will be utilized to develop the packaging materials. Moreover, the processing conditions for developing edible food packaging need to be optimized to achieve better packaging properties with an improved storage life of food products.

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Chapter 3
Cellulose-Based Nanostructured Materials in Edible Food Packaging

Tabli Ghosh, Doli Hazarika, and Vimal Katiyar

3.1 Introduction

The development of cellulose-based nanostructured materials has a great interest in the current trend of edible food packaging. The cellulose derivatives and its nanostructured materials are utilized in various food and beverage industries for obtaining tailor-made properties of food products. As shown in Fig. 3.1, the targeted food-based industries for using cellulose-based products include bakery industry, meat industry, dairy industry, cereal industry, veterinary foods, food packaging industry, and others due to its tunable physicochemical properties. In food industries, the cellulose and its derivatives are used in various formulations such as emulsifiers, bulking agents, anticaking agents, fat substitutes, and texture enhancers. Additionally, cellulose and various derivatives play a remarkable role in improving the food quality in terms of texture, color, and others with an enhanced shelf life of food products. The use of cellulose in food products and packaging (edible and non-edible) are largely applied due to increased food waste for careless handling, mechanical damages, and increase of plastic-based waste as packaging materials. Cellulose and its nanoforms are available extensively and have the characteristic attributes of biodegradability, biocompatibility, surface chemistry, improved packaging property, non-toxicity, which make them an ideal material to be used as a replacement for available conventional materials. Cellulose has a great market value for reducing the overall carbon footprint in the packaging industry and further provides improved food value. Interestingly, cellulose derivatives and nanoforms are also used in edible packaging materials to aid improved properties to other materials such as chitosan (CS), starch, and agar. Further, the use of nanocellulosic materials in edible and non-edible sectors has been increased for

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having tailored barrier, mechanical, thermal, color properties which further improved the shelf life of food products. As shown in Fig. 3.1, the properties of nanocellulosic materials can be modified via chemical, physical, mechanical methods, and developing biocomposites of nanocellulose.

Additionally, the various forms of nanocellulose are available as nanocrystals, nanofibers, nanoparticles (NPs) which can be availed by modifying the routes of synthesis. The various forms are comparable in terms of obtaining the tunable properties of packaging materials for both edible and non-edible packaging forms. The characteristic traits of nanocellulose include rheological behavior, high mechanical property, lightweight, barrier properties, nutritional properties, etc. The cellulose-based nanostructured materials are utilized in food packaging, developing starch-based foods, food stabilizers, delivery systems, etc. The nanocellulose is widely used as a stabilizing agent in food emulsions, in food packaging, and as functional food ingredients. Nanocellulose has several traits such as: (i) The rheological behavior of nanocellulose provides a route to be used as food additives; (ii) transparency, mechanical property, barrier property provide food packaging application; (iii) high surface area and surface functionality are helpful in developing food coating; and (iv) food products such as sweets, delicacies, and puddings; low-fat mayonnaise, milk ice-cream, fruit jelly, meat sauces. Further, bacterial cellulose (BC) as food packaging materials for sausage, meat casings, and others is also available.

Based on this discussion, the chapter will detail the extraction of cellulose-based materials from various available sources for the fabrication of nanoforms. Nanocellulose structures can be obtained from available sources utilizing a significant method and play a very significant role in nanotechnology-based research and development. However, the yield of nanocellulosic materials is dependent on several factors such as source, extraction process, and processing conditions.

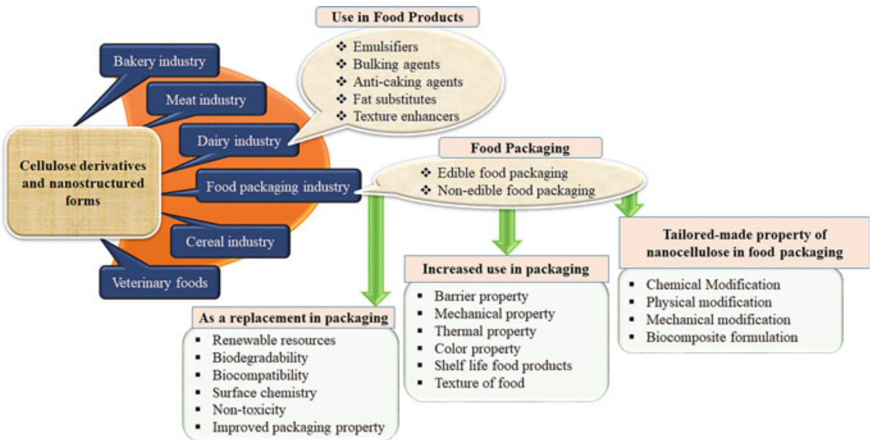

Fig. 3.1 Prospect of cellulose derivatives and nanostructured form in food sector

A brief discussion has been made on the status of using cellulose, its various forms in the field of food sectors such as bakery products, meat products, dairy products, edible food packaging, non-edible food packaging, and others. The global cellulose-based food packaging status has also been overviewed. However, the nanoforms of cellulose can also be fabricated utilizing several processes such as acid hydrolysis, mechanical methods, enzymatic hydrolysis, oxidation methods, and ionic liquid treatments. A detailed discussion about the several routes for the synthesis of nanocellulose materials has been made. Finally, the use of various forms of nanocellulose in the development of composite-based edible food packaging for enhanced shelf life of food products has also been detailed.

3.2 Outlook of Cellulose Resources to Be Used in Food Application

The wide usability of cellulose-based materials in developing packaging films is increasing such as bottles, boxes, and pouches. The plant fibers are the major sources of cellulose such as cotton, flax, hemp, and jute. Further, lignocellulosic biomass such as agricultural residues, forest residues, and energy crops is a source of cellulose. The agricultural residues include rice husk, wheat straw, corn stover, rice straw, sorghum straw, coconut husk, pineapple leaves, manure, seaweeds, barely straw, maize, barley husk, etc. The forest residues as a source for lignocellulosic biomass include woodchips, wood sawdust, wood branches, etc. Further, the energy crops as a source of lignocellulosic biomass include energy cane, Miscanthus, switch grass, etc. Cellulose is also available from plant foods such as cereals, fruits, nuts, legumes, potato with skins, seeds, and cabbage family of vegetables. In 1920, cellulose has been synthesized from delignified wood pulp, ramie, cotton, and other sources. The extraction processes for cellulose from available sources have been made in the below section. The various cellulose derivatives such as BC, carboxymethyl cellulose (CMC), enzymatically hydrolyzed CMC, cellulose acetate, ethylcellulose (EC), hydroxypropyl cellulose (HPC), hydroxypropyl methyl cellulose (HPMC), methylcellulose (MC), ethyl methyl cellulose, microcrystalline cellulose (MCC), and powdered cellulose are used as additives in food products. Further, cellulose acetate, EC, methyl cellulose, powdered cellulose are used as binders in food products. Nanocellulosic materials with versatile properties are also utilized in various food products and packaging materials for improved properties. Interestingly, the surface morphology and characteristic attributes of nanocellulosic materials are highly dependent on the pretreatment processes of lignocellulosic biomass which removes the non-cellulosic part.

3.3 Extraction of Cellulose from Available Sources

Cellulose is a homopolymeric unit consisting of β-1,4-linked anhydro-D-glucose having high molecular weight, and the repeating units are corkscrewed at 180° with their neighbor molecules. From very early days, the incorporation of cellulose fibers in composite materials has been increased tremendously in terms of effective cost, recyclability, biodegradability, and availability in comparison with the conventional reinforcing materials such as glass and aramid fibers. As discussed, the available sources of cellulose are plants such as ramie, sisal, flax, wheat straw, and potato tubers, whereas algae, bacterial source, and marine animals are also potential sources for producing cellulose [1, 2]. The cellulose and related nanoforms are a remarkable candidate to be used in edible food packaging materials for the noteworthy properties. The extraction process, sources, pretreatment process play a remarkable role in the fabrication of nanocellulose materials to be used in edible packaging materials. However, a brief discussion has been made related to the available sources of cellulose (Fig. 3.2), which are used globally to obtain cellulosic materials.

3.3.1 *Cellulose from Plant-Based Sources*

The wide availability of wood pulp and cotton fibers has made potential pathways to extract cellulose. As discussed, the plant materials such as bamboo, flax, hemp, jute, ramie, sisal, rice husk, and coconut husk are also a potential source of cellulose. The plant sources are generally considered as lignocellulosic biomass, having the main hierarchical architecture consisting of cellulose, hemicelluloses, and lignin. Additionally, the grasses, water plants, some plant parts (leaf, fruit, and stem), and agricultural wastes such as sugarcane bagasse, rice straw, and wheat

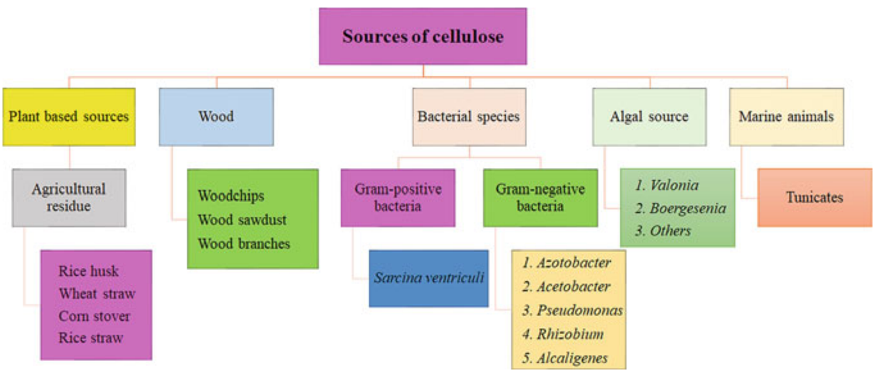

Fig. 3.2 Available sources of cellulose to be used in edible food packaging

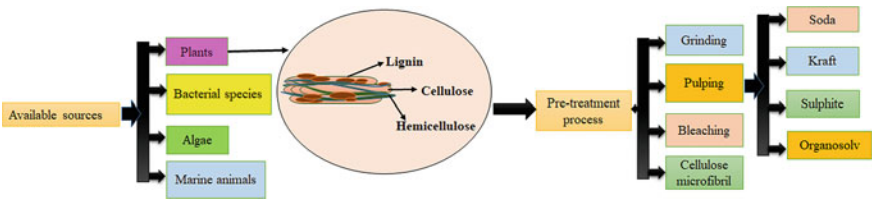

Fig. 3.3 Cellulose sources and pretreatment process for cellulose extraction

straw are available sources for cellulose. However, the percentage of cellulose from lignocellulosic biomass varies from sources to sources such as (i) cotton stalks: 58% cellulose, (ii) bagasse: 52% cellulose, (iii) sweet sorghum: 44% cellulose, (iv) corn ears: 38% cellulose, (v) pineapple leaf: 36% cellulose, (vi) corn cobs: 33.7% cellulose, and (vii) wheat straw: 32.9% cellulose. The extraction of cellulose from lignocellulosic biomass generally follows pretreatment methods such as chopping, pulping, and bleaching methods to obtain cellulose as represented in Fig. 3.3.

3.3.2 Cellulose from Bacterial Species

Gluconacetobacter xylinus (formerly known as *Acetobacter xylinum*) is the most used bacterial species for the fabrication of BC. However, BC can be obtained from both gram-positive (*Sarcina ventriculi*) and gram-negative bacteria (*Azotobacter, Acetobacter, Pseudomonas, Rhizobium, Alcaligenes, Salmonella*). The bacterial species produce cellulose microfibrils having the appearance of clear, flat, and thick gel with a large amount of water content (97–99%). The advantageous nature of BC is its high chemical purity with tunable microfibril formation and crystallization property, which can further be tailored by adjusting the culture conditions. The advantageous attributes of BC in comparison with plant cellulose include hydrophilicity, ultrafine network structure, mechanical property, water holding capacity, etc. Additionally, the BC has obtained an immense interest in edible food packaging for its characteristic attributes such as wettability property, purity, and mechanical property.

3.3.3 Cellulose from Algal Sources

The major component is the cell wall of algae with highly crystalline nature such as brown, green, yellow, gray, and brown. Various orders of algae are *Valonia*, *Boergesenia, Micrasterias denticulata, Micrasterias rotata*, *Dictyosphaeria, Siphonocladus*, *Cladophora*, *Boergesenia*, *Microdyction,*and *Rhizoclonium,* where

Valonia (I_{α} triclinic allomorphs type) or *Cladophora* are highly crystalline up to 95%. The algae-based materials for cellulose are recommended for their high crystallinity.

3.3.4 Cellulose from Marine Animals

Mostly focused marine invertebrate sea animal of Tunicates class is sea squirts (*Ascidiacea*), and their different species are *Metandroxarpa uedai, Halocynthiaroretzi,* and *Halocynthia papillosa*. The tunics (outer tissue) of tunicates are generally consisting of cellulose, lipids, mucopolysaccharides, sulfated glycans. The cellulose developed from tunics can be obtained through prehydrolysis, kraft cooking, bleaching process to remove the non-cellulosic parts. The fabricated cellulose can be obtained as 100% pure with highly crystalline in nature with CI_{β} lattice-type (monoclinic unit having two hydrogen bonding chains per unit cell) allomorph and further provide high microfibril aspect ratio, which is a useful approach to be used in packaging application. However, the cellulose from tunicates can be obtained as different morphological and chemical structures, and yields depending on the targeted sources of extraction.

Cellulose is assembled as an individual cellulose chain forming cell wall of the fiber (including primary cell wall and secondary cell wall). The secondary wall mostly consists of microfibrils comprising both the amorphous and crystalline regions. They are helically framed where the crystalline region is having inter- and intramolecular interaction networks. Even molecular orientations of crystalline regions also vary with different interchangeable cellulose polymorphs, namely I, II, IIII, IIIII, IVI, and IVII [3, 4]. The promising properties of cellulose have made researchers in increasing its usability for versatile application. Moreover, the isolation of cellulose in highly pure form is possible only by dissolution of hemicellulose, lignin, and remaining non-cellulosic components. Thereby, cellulose can be isolated from the various available sources with varied properties as mentioned [4, 5]. In the paper industry, pulping and bleaching methods are used to remove the components other than cellulose and further, the brightness of fabricated cellulose can be adjusted by mechanical processes. The alkali treatment is the most common method to remove the hemicellulose and lignin part because of their alkaline solubility (Fig. 3.3). Extraction of cellulose from agriculture waste (sugarcane bagasse) can be developed using steam explosion and xylanase as pretreatments followed by bleaching. The various resources have reported with their crystallinity percentage for cellulose such as sisal fibers (75%), wheat straw (77.8%), sugarcane bagasse (50%), sheath of coconut palm leaf (47.7%), and commercial microcrystalline cellulose (74%). Extraction of cellulose microfibrils from agricultural residue (coconut palm leaf sheath) has been done using chlorination and alkaline extraction process to obtain 10–15 μm diameter fibrils [6]. Another study has been made using agro-industrial biomasses (pomaces) to enhance its usefulness in the field of polymer. The obtained cellulose fraction for each pomace is having Segal crystallinity index (tomato: 48.97%, apple: 51.34%, cucumber: 53.61%, carrot: 68.73%) with low lignin content [7]. Cellulose fibers are extracted from rice husk with cellulose content of 96% after chemical treatment using alkali and bleaching to obtain a diameter of 7 μm with enhanced thermal stability [8]. Moreover, sugarcane bagasse has been used for isolation of cellulose by steam explosion and xylanase pretreatment [9]. The extracted cellulose materials have crystalline and amorphous regions, where the fabrication of nanocellulose mainly consists of crystalline regions with various percentages of crystallinity. However, the property of nanocellulose in terms of crystalline regions can be tailored for improved property.

3.4 Fabrication of Nanocellulosic Materials

Depending on the dimensions, functions, preparation methods, and sources, as shown in Fig. 3.4, nanocellulose has been subcategorized as cellulose nanocrystal (CNC), nanofibrillated cellulose (NFC), and bacterial nanocellulose (BNC). Among all, CNCs are mostly crystalline rod shaped having limited flexibility than NFC. Further, CNC may also be termed as nanowhiskers, rod-shaped cellulose crystals, or nanorods by various researchers. CNC is having low aspect ratio with diameter 2–20 nm and varying length between 100 nm and few several micrometers. The CNC or nanocrystalline cellulose (NCC) is highly crystalline nature with 100% pure cellulose content with crystalline percentage between 54 and 88% [10], whereas NFC (developed through chemo-mechanical method) is bundle of cellulose chains with long, flexible, and entangled cellulose nanofiber (CNF) with approximately 100 nm diameter having alternate crystalline and amorphous diameter and crystallinity index of 63.57% [11]. The bigger size of NFCs may be obtained due to aggregation of NFC molecules. Further, the NFC dimensions may be varied depending on the extraction method and sources. BNC is having superfine

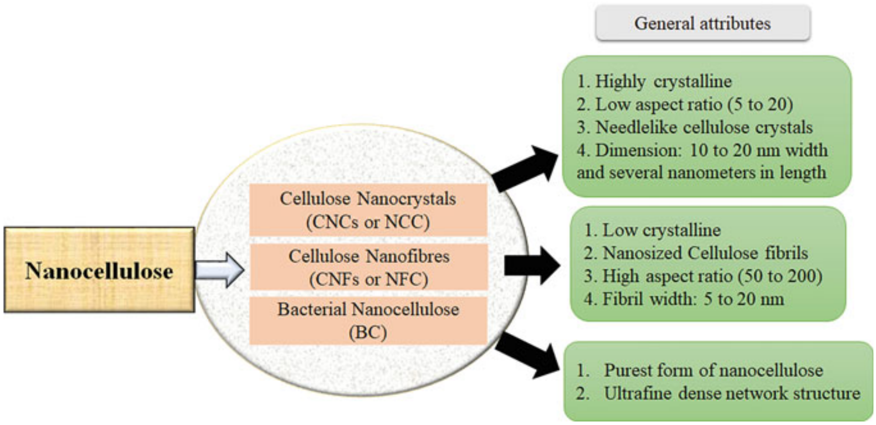

Fig. 3.4 Various forms of nanocellulose

diameter, high degree of polymerization, and high crystallinity produced by using microorganisms via two methods such as fermentation production and reactor-based production. For the fabrication of BNC, several acetic acid bacteria are used [12], where *Komagataeibacter xylinus* has received much interest due to their capability in secreting micro- or nanofibrils. An investigation has been made using high-pressure homogenizer (HPH) for preparation of BNC from pineapple peel waste by fermentation using *A. xylinum*, where the fabricated BNC (by HPH) has a crystallite size of 4.76 nm and crystallinity percentage as 86% [13]. In this regard, Fig. 3.5 details the various techniques for nanocellulose extraction.

3.4.1 Acid Hydrolysis

The first attempt of preparing successful CNC was made in 1947 using the technique of acid hydrolysis of cellulose with sulfuric acid and hydrochloric acid by Nickerson and Habrle [14]. Besides, the techniques for obtaining nanocelluloses such as enzymatic hydrolysis, organic acid hydrolysis, solid acid hydrolysis, oxidative degradation, ionic liquid, and subcritical hydrolysis have been represented in a tabulated form in below section (Table 3.1). The acid hydrolysis is the most commonly used among the other methods including sulfuric acid (H_2SO_4), hydrochloric acid (HCl), phosphoric acid (H_3PO_4), hydrobromic acid (HBr), and their mixed acids [15]. Among all, H_2SO_4 has been used mostly due to the negative surface charges and producing more stable suspension of nanocellulose. In general, a concentration of 60–65%, with reaction time and temperature of 30–60 min and 40–50 °C, respectively, is required to develop CNC (30 wt% CNC yield). The decreased yield may be related to the high acid concentration, temperature, and time, which can be tailored further for increased yield of CNC. However, the yield of CNC, its properties, characteristics such as crystallinity and aspect ratio can be monitored by varying the acid concentration. Further, CNC obtained from eucalyptus with yield of 70 wt% can also be obtained via tailoring the reaction conditions in terms of acid concentration, temperature, and reaction time [16]. The bamboo pulp has been used as the source for cellulose to obtain CNC (rod and porous network form) with yield of 32.3 wt% using sulfuric acid hydrolysis. The thermal stability for CNC presented two-step degradation temperature range at 180 ∼ 320 °C and 350 ∼ 450 °C [17]. Moreover, from oil palm empty fruit bunch pulp, CNC having spherical shape and average diameter 30–40 nm has been obtained using H_2SO_4 solution (64% w/v) at 45 °C in an ultrasound bath for almost 2 h [18], whereas to obtain increased yield of nanocellulose, acid concentration is the key parameter. Further, studies have been made on bleached kraft (eucalyptus pulp), where acid concentration between 58 and 62 wt%, moderate temperature of 50–60 °C, and reaction time between 30 and 180 min are preferred to maximize CNC yield [19]. The extraction of CNCs using hydrochloric acid hydrolysis under hydrothermal conditions (neutralization with ammonia) can provide thermally stable CNC with 93.7% yield, 88.6% crystallinity, maximum degradation

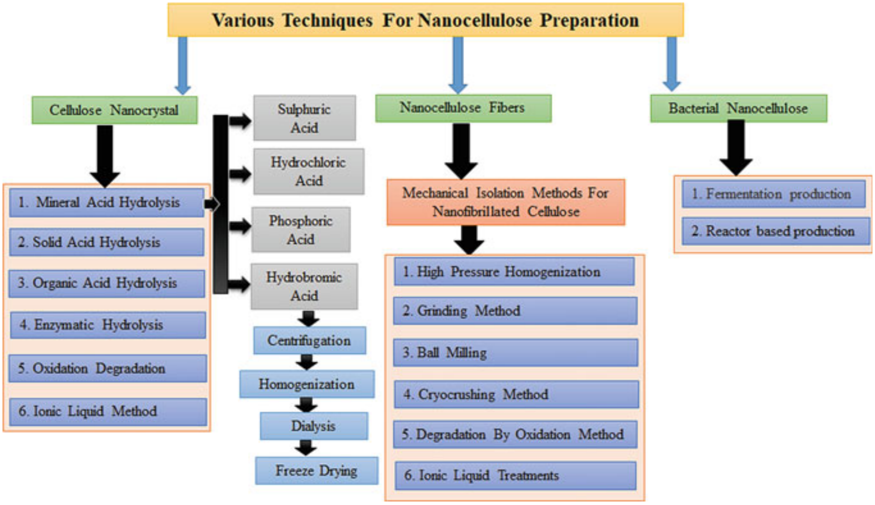

Fig. 3.5 Targeted techniques for extraction of nanocellulose

temperature of 363.9 °C [20]. Further, the presence of ammonia in the hydrochloric acid hydrolysis under hydrothermal condition can provide stability of CNC suspensions. Moreover, a further study has been reported using different mineral acids to obtain tailored morphological structure of nanocellulose, where the CNCs can be utilized to develop poly(lactic acid)-based composites with improved thermal stability as non-edible packaging materials [21]. The fabrication of acetylated CNF from sisal fiber has been done using chemical methods such as strong alkali treatment to swell fibers, bleaching treatment to remove lignin, and acetylation treatment to reduce the intermolecular hydrogen bond [22]. Further, the surface chemistry of nanocellulosic materials can be tailored for various applications. Moreover, among available, CNCs can be widely utilized as a potential nanofiller for the various characteristic attributes [23].

3.4.2 *Mechanical Methods*

In plant cell walls, cellulose fibers are converted to nanofibrils applying high and strong mechanical disintegration power, where the nanocellulosic materials can be obtained with dimensions of 10–100 nm. In this regard, the several mechanical techniques to obtain nanocellulose are grinding, cryocrushing, homogenization, microfluidization, ultrasonication, etc. In case of grinding method, cellulose slurry is introduced between static and rotating grindstones, and the grinding of the materials occurs due to frictional effects which deliver smaller-sized materials with higher surface areas. There are various factors which may affect the obtained

Table 3.1 Fabrication of nanocellulose using various techniques from available sources

Sl. no.	Source	Method	Procedure	Results	References
1	Rice husk	Acid hydrolysis	50 °C, 10.0 mol L^{-1} of sulfuric acid, 40 min	10–15 nm	[6]
2	Sisal fibers	Nitric acid and acetic acid	Boiling temperature 90 min, diluted to (1:5 ratio)	Diameter of 27 ± 13 nm and length of 658 ± 290 nm	[25]
3	Recycled pulp	Enzymatic hydrolysis	84 EGU of endoglucanase per 200 mg, 50 °C for 60 min	33–80-nm widths and 100 nm–1.8 µm in length	[14]
4	Sugarcane bagasse	High-pressure homogenization	Ionic liquid (1-butyl-3-methylimidazolium chloride ([Bmim]Cl))	Nanocellulose diameter: 10–20 nm	[32]
5	Sisal fibers	Enzymatic hydrolysis and mechanical shearing	Enzyme Celluclast (Cclast) (endoglucanase) or ecopulp energy (Eco) (exoglucanase) (minimum 0.1 wt% incubated at 50 °C for 2 h) Homogenized and incubated again for 50 °C for 2 h, manual 10 min mixing an 100 °C for 10 min to stop reaction	Length (Cclast = 251 ± 47 and Eco = 234 ± 63) Diameter = 7.2 ± 0.8 and 6.6 ± 1.3, respectively	[15]
6	Pulp fiber	Cellulase and xylanase	20 u/mL (cellulase: xylanase) = 9: 1	Mean particle size of about 30 nm	[29]
7	Oil palm empty fruit bunch pulp	Ultrasound-assisted acid hydrolysis	With 64% (w/v) of H_2SO_4 solution in an ultrasound bath At 45 °C for 2 h	Average diameter range of 30–40 nm	[18]
8	Banana fibers	Steam explosion in alkaline medium followed by oxalic acid treatment	Pressure 20 lb or 15 min, repeated for 8 times with pressure release	Length 200–250 nm Diameter 4–5 nm	[35]
10	Commercial MCC	Subcritical hydrolysis	120 °C, heating and cooling rate (6 and 3 °C/min), 60 min, pressure 20.3 MPa	242 ± 98 nm in length and 55 ± 20 nm in width	[37]
11	BNC	High-pressure homogenizer	Sugar 10% (w/v), ammonium sulfate 0.5% (w/v), and add acetic acid, bacteria starter 10% (v/v), and incubated for 10 days. at 150 bar and 5 repeated cycle	4.76 nm	[13]

BNC: Bacterial nanocellulose; *MCC*: microcrystalline cellulose nanocellulosic materials. The ball milling such as planetary ball mill, mixer ball mill, and vibration ball mill is utilized to obtain the grinding of cellulosic materials. Further, vibratory ball mill is also used for defibrillation of ramie fibers and others. Another fibrillation method to obtain nanocellulose is cryocrushing, where water-swollen cellulose fibers are first immersed in liquid nitrogen and further crushed using mortar and pestle to rapture the plant cell wall [24]. The cryocrushing method needs very high energy consumption; thus, it is not economic.

Additionally, many researchers have worked on high-pressure homogenization method, where cellulose is passed through small nozzle at high pressure (50–200 MPa), high velocity to generate shear rate to reduce the particle size into nanoscale. This method is mostly used to prepare CNF. The high shear forces, large pressure drop, turbulent flow, the number of homogenization cycles, and inter-particle collisions are the main criteria to achieve reduction of cellulose fibrils. Few disadvantages of this method are homogenizer clogging, high energy consumption, and damage of crystalline structure due to excessive mechanical shear. Thereby, various other mechanical treatments have been performed like grinding, milling, cryocrushing, etc [25]. Also, ultrasonication has attracted a considerable interest to isolate CNF, which involves application of sound energy, physical as well as chemical systems. Here, cavitation occurs with introduction of intense shear forces, shock waves, and others. The extreme environment conditions with very high temperatures, pressures, and heating/cooling rates help in cleaving the strong cellulose interfibrillar hydrogen bonding into nanofibers. A work has been made on the fabrication of NCC from oil palm empty fruit bunch pulp (EFBP) using mechanical technique, i.e., ultrasound-assisted acid hydrolysis [18].

3.4.3 Enzymatic Hydrolysis

An interesting method to produce nanocellulose fibrils in contrast to mineral acid hydrolysis methods is enzyme hydrolysis, which does not lead to toxic residues, and further, very mild thermal and pressure conditions are required for value-added applications. An investigation was performed using combination of enzymatic hydrolysis and mechanical shearing to produce NPs from sisal pulp, whereas hydrolysis using enzyme endoglucanase in microwave heating allows faster NP production compared with conventional heating [26]. Further, the morphology of cellulosic NPs can further be tuned by combined processes of mechanical shearing, acid hydrolysis, and enzymatic hydrolysis [27]. The enzymatic hydrolysis is generally obtained using cellulases, which helps in cellulosic fiber hydrolysis. Further, cellulase enzymes are categorized into three types such as endoglucanases (β-1,4-endoglucanases/A-type and B-type cellulases), exoglucanases (cellobiohydrolase, also named as C- and D-type cellulases), and β-glucosidase which can convert cellobiose to glucose [28, 29]. Among all, endoglucanases are the ones with the highest interest as it hydrolyzes the amorphous region, and exoglucanase attacks the cellulose chain from reducing or non-reducing end. Further, synergistic interactions of cellulases on crystalline cellulose in various regions of cellulose microfibril provide better activities than sum of the individual activities [30]. Chen and coworkers have reported spherical-shaped cellulose NPs having mean diameter of 30 nm using enzymatic hydrolysis from pulp fibers [29].

3.4.4 Ionic Liquid Treatments

The ionic liquids for cellulose dissolution include mainly 1-butyl-3-methylimidazolium $[C_4mim]^+$, 1-hexyl-3-methylimidazolium $[C_6mim]^+$, and 1-octyl-3-methylimidazolium $[C_8mim]^+$ cations and anions such as Cl^-, $[PF_6]^-$, Br^-, $[BF_4]^-$, $[SCN]^-$. However, the normal stirring of celluloses in such liquid ions has no dissolution as reported, but heating in a microwave oven at 100–110 °C improved the dissolution rates [31]. Also, these ionic liquids are recoverable using ease methods such as ion exchange, evaporation method, and reverse osmosis for reusability. This is a green way of developing nanocellulose without any pre-treatment method with good mechanical property. Li et al. have reported a work on developing nanocelluloses (diameter range of 10–20 nm) from sugarcane bagasse using high-pressure homogenization method coupled with a homogeneous pre-treatment method using an ionic liquid [32]. Moreover, the highly crystalline nanocellulose materials can also be obtained with the aid of ionic liquids [33].

3.4.5 Steam Explosion

Steam explosion is an effective method for the development of nanofibers from biomass using high-pressure steaming followed by rapid decompression [34]. In this method, dry materials are saturated using steam at elevated pressure and temperature followed by sudden pressure release. Thus, the thermomechanical force generated by the flash water evaporation causes rapturing of the material. This method helps in reducing the non-cellulosic compounds using steam explosion and can be utilized as pretreatment methods. The CNF obtained using this technique has higher aspect ratio than other conventional methods. The principle is based on cooking in vapor phase in a temperature range of 180–210 °C and steam pressure between 1 and 3.5 MPa. The advantages of steam explosion include low energy and chemical consumption, low environmental impact, and lower capital investment. Various sources are used to obtain nanocellulose using this method such as banana fibers followed by oxalic acid treatment [35].

3.4.6 Supercritical and Subcritical Water Hydrolysis

Another method was proposed as an alternative to acid hydrolysis termed as supercritical water hydrolysis, where pure water is heated and pressurized above critical points such as to 374 °C and 22.1 MPa, where the water properties such as density, viscosity, and dielectric constant get decreased. In supercritical reactors, a pressure of 25–30 MPa is used maintaining the temperature and time. Thereby, swollen or dissolved form of the microcrystalline cellulose can be seen in near critical and supercritical water, which enables a homogeneous hydrolysis [36], whereas use of subcritical water (obtained at 120 °C and 20.3 MPa for 60 min) is another greener pathway for CNC production with high crystallinity index of 79.0% and having onset degradation temperature around 300 °C. This process provides lower corrosive nature, low and cleaner effluent, and use of low-cost reagents [37].

3.5 Overview of Cellulose and Nanocellulose in Food Sector

Cellulose has many beneficial properties such as it functions as an anticaking agent and thickening agent, and replaces fat in some food products. However, the materials should not create cytotoxicity and genotoxicity; should follow internationally standardized regulations; and should reduce the calorie value of food products. The nanocellulose forms as nanocomposites are used as food packaging and functional nanocomposites such as gas barrier coatings and food quality analysis sensors. The use of nanocellulose in various applications specifically in food sector includes food packaging (edible and non-edible), as ingredients in food functionalization, as stabilizing agents in food emulsion, as food additives, as dietary fiber, etc. [38]. Further, vegetable nanocellulose is used in salad dressings, sauces, gravies, cake frostings, whipped toppings, etc. The use of nanocellulose has been found in sauce and soy soup, retort food, bean jam, dough based products, etc. As food stabilizers, nanocellulose-based materials are used in the preparation of foams, puddings, dips, whipped toppings, etc. Further, the food applications of BC include nata-de-coco, rheology modifiers, fat replacers, artificial meat, stabilizer of Pickering emulsions, immobilizer of probiotics and enzymes, etc. BC is used as a raw material for developing dessert and artificial meat and further is used as food ingredients for gelling, water binding, emulsifying, thickening, stabilizing agents. The noteworthy health beneficial properties of cellulose and its nanoforms provide a great interest in the current food sector for versatile application. Besides food packaging, cellulose is widely utilized in the sections of (i) renewable energy such as household industry, electricity, and biogas in traffic fuel; (ii) construction materials and industrial applications such as furniture, buildings, and panels; and (iii) development of paperboard, tissue paper, musical instruments, fertilizers, etc.

3.5.1 Use of Cellulose and Nanocellulose in Food Products

Cellulose is used widely in bakery products for obtaining improved properties and healthier food products [39]. In bakery industry, cellulose and various derivatives such as methylcellulose, CMC, MCC, HPC, and others are used to improve the dough rheology and bread texture [40, 41]. Moreover, the textural properties of pasta can be improved using CMC and the solid losses in non-wheat-based pasta can also be reduced [41]. Further, cellulose derivatives such as microcrystalline cellulose, methyl cellulose, CMC, HPC, and HPMC have a great impact in obtaining the tailor-made properties of crumb of gluten-free doughs and breads [42]. The stability of frozen dough during storage can be maintained using CMC and gum arabic, where the use of the biomaterials helps in reducing the ice crystallization in frozen dough [43]. The emulsifiers such as glycerol monostearate, sodium stearoyl-2-lactylate, calcium stearoyl-2-lactylate, and diacetyl tartaric acid esters of mono- and diglycerides are used to improve the crumb properties of gluten-free doughs and breads [44]. Cellulose derivatives as emulsions are used to replace fat in biscuits [45]. In biscuit preparations, trans-fatty acid-free fat replacers such as sunflower oil–water–cellulose ether emulsions are used to reduce fat content of biscuits [44–46]. The fat content in the biscuits need to be reduced for developing healthier food products, where methyl cellulose and HPMC have good emulsifying properties and have an ability to develop stable vegetable oil-in-water-based emulsions. The sensory of rice cakes can be modified using CMC, where the cellulose derivatives help in improving the property of gluten-free cakes [47]. Thus, cellulose holds onto a great impact in the development of various bakery products for improved properties. Further, BNC has a great potential to be used as an additive for wheat bread, where the addition of BNC provides increased average pore size and softer crumb compared to control formulation [48]. In this way, cellulose and its various forms are utilized to deliver improved food attributes. In meat industry, cellulose-based casings are extensively utilized in the preparation of meat and poultry-based ready-to-eat food products such as sausages, frankfurters, and bologna [49]. Additionally, BNC is also used as an additive in tailoring the quality and stability of low-lipid low-sodium meat sausages [50].

3.5.2 Use of Cellulose, Its Derivatives, and Nanoforms in Food Packaging

Cellulose, its derivatives, and nanostructured forms are extensively utilized in the development of edible and non-edible food packaging materials as an alternative for existing packaging materials [51–54]. Cellulose in various forms such as hydrogel, composites, edible films, and edible coatings is utilized for food packaging section. The various properties of nanocellulose in food packaging involve eatable nature, biocompatibility, transparency, antimicrobial, barrier properties against oil and grease, oxygen, water vapor, liquids, mechanical properties, flexibility, etc. The packaging application of nanocellulose is utilized via solution casting, coating, layer-by-layer assembly, electrospinning, and others. The main motivation of using nanocellulose in food industry is extending the shelf life of food products, blocking gaseous elements into food materials, improving food quality, etc. The nanocelluloses have gained extreme popularity to be acted as a nanofiller material for developing bionanocomposites.

Edible Food Packaging. The cellulose delivering various health beneficial properties and further having a capability to improve packaging properties and shelf life of food products is used in the development of edible food packaging materials. The use of cellulose and its derivatives in edible food packaging includes CMC/CS bilayer-based edible coating on citrus fruit [55], methyl cellulose/sodium alginate (SA) for peaches [56], cellulose derivatives for 'Berangan' banana [57], CS/CMC/moringa leaf extract-based edible coating for avocado fruit [58], CMC/Zataria multiflora essential oil/grape seed extract-based edible coating for rainbow trout meat [59], CMC/calcium/ascorbic acid-based edible coatings for fresh-cut apples [60], etc. The application of nanocellulose in edible food packaging includes CS/CNF for cut pineapple [51], carrageenan/cellulose nanowhisker (CNW) as edible films [61], alginate–acerola puree/cellulose whisker (CW) as edible films [62], fish myofibrillar protein/bacterial cellulose nanofiber (BCNF) as edible films [63], agar/nano-BC as edible films [64], etc.

Non-edible Food Packaging. The non-edible nanocellulose-based packaging includes the use of various nanostructured forms of cellulose as a filler material in biodegradable polymers. Several researches report the development of biocomposite-based materials for food packaging applications such as CNC-reinforced poly(lactic acid) [65, 66], CNC-reinforced poly(3-hydroxybutyrate) (PHB), CNW-reinforced poly(lactic acid) [67], paper sheets coated with wheat gluten (WG)/nanocellulose/titanium dioxide nanocomposite as active food packaging [68], and paper coated with modified nanocellulose fiber (NCF)/polylactic acid (PLA) composites [69].

3.6 Market of Cellulose-Based Packaging

The segmentation of market report on cellulose film packaging has been done based on film types, sources of cellulose extraction, targeted end use, application area, and region of development (as shown in Fig. 3.6) [70]. The market is segmented based on the sources such as cotton and wood-based sources for the fabrication of films, and the films can be obtained as colored, transparent, and metalized type depending on the synthesis type. Moreover, the various end use industries for using cellulose film packaging include food and beverage, pharmaceuticals, retail and personal care, etc. The growth of global market of cellulose film packaging is related to the

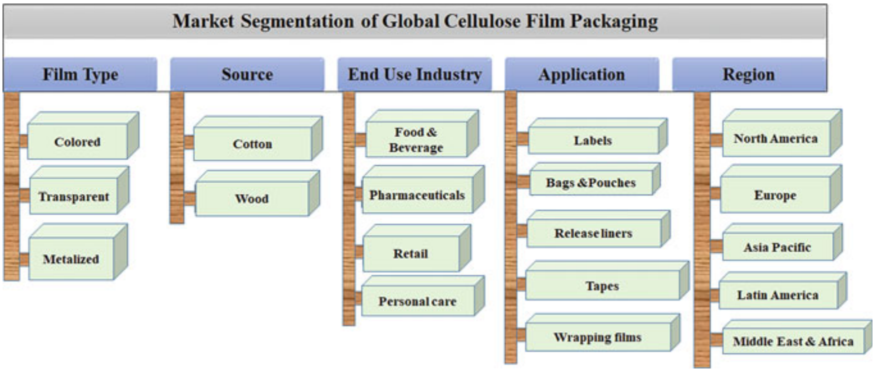

Fig. 3.6 Market segmentation of global cellulose film packaging

easy availability of raw materials and manufacturing techniques. However, the available alternatives and cost of raw materials that are required for the development of cellulose film may change the target market growth of cellulose film packaging. The region of global cellulose film packaging includes North America (USA and Canada), Europe (Germany, UK, France, Italy, Spain, Russia, and others), Asia-Pacific (China, India, Japan, Australia, South Korea, and others), Latin America (Brazil, Mexico, and others), and Middle East and Africa (GCC, South Africa, and others). The cellulose film packaging is developed by Futamura Chemical Co., Ltd., Hubei Golden Ring Co., Ltd., Celanese Corporation, Weifang Henglian Cellophane Co., Ltd., Eastman Chemical Company, Chengdu Huaming Cellophane Co., Ltd., Tembec Inc., Sappi Limited, Rotofil Srl, Rhodia Acetow GmbH, etc. In this regard, oils and fat-resistant cellulose film packaging are developed by NatureFlex^{TM}, where heat-seal resins are used for microwave applications [71].

3.7 Prospective for Edible Food Packaging Application

As discussed in the previous section, cellulose-based materials have obtained a wide usability in food sector for its noteworthy attributes. As shown in Fig. 3.7, cellulose being a non-toxic, biocompatible, widely available, renewable resource has gained an extreme enthrallment in the field of edible food packaging. Cellulose has been used for versatile application from 150 years back. The noteworthy features of nanocellulose include facile surface chemistry, promising health beneficial properties, physical and chemical properties, etc. In this regard, the section will give a brief overview about the various promising factors of nanocellulose to be used in edible food packaging materials.

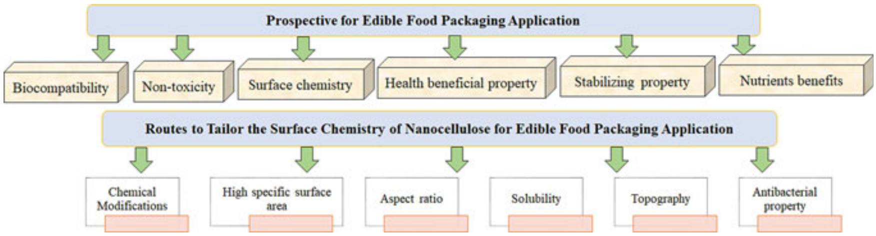

Fig. 3.7 Prospects of nanocellulose in edible food packaging

3.7.1 Biocompatibility and Non-toxicity

The biocompatibility of a material is generally defined as the compatibility of the material with the living tissues. This is a kind of biomaterial behavior, where the toxic or immunological response of the material in human body is not produced. The biocompatibility of a material does not possess toxic effect on biological systems, and the material should perform the targeted function without creating any undesirable effects in the recipient. The biocompatibility of a material is evaluated following ISO 10993 set of standards. The pure form of nanocellulosic materials is non-toxic and biocompatible, and is used widely in edible food packaging materials. The BC has the status of "generally recognized as safe" by Food and Drug Administration (FDA). Further, the compatibility and cell adhesion property of nanocellulose can be promoted by controlling the surface property of nanocellulose.

3.7.2 Surface Chemistry

Cellulose units can form intra- and interhydrogen bonding within cellulose chains and between cellulose chains. There are strong intra- and interhydrogen bonding in the crystalline region of cellulose, which makes it enable to absorb water. On the other hand, in the amorphous region, the bonding is weak, which makes them able to absorb some water. The moisture uptake of cellulose is around 8–14%, and the water uptake rate is very slow. The nanocellulose molecules can be easily functionalized due to the presence of numbers of hydroxyl groups on nanocellulose surface, which helps to provide improved dispersion in composites for acting as edible food packaging materials. The cellulose is composed of both crystalline and amorphous regions, where the CNC molecules have higher crystallinity than CNFs. The fabrication of CNC is done using strong acids, which make them more crystalline in nature ranging from 40 to 90%. The surface modification of nanocelluloses can be obtained via (i) physical modification such as utilizing surfactants (anionic, cationic, nonionic surfactants) and polymers (macromolecules and biopolymers), and (ii) chemical modification such as polymer grafting (grafting from and grafting onto) and derivatization. Further, the surface chemistry of nanocelluloses can be utilized and modified properties can be obtained using cross-linkers and compatibilizers, etc. However, the modification of nanocelluloses can be obtained via functionalization of hydroxyl groups, which increase the interaction and dispersion property, etc. However, the nanocellulosic materials have a tendency to form agglomeration due to strong hydrogen bonding; thus, the fillers need to be dispersed properly via chemical or physical modification to obtain tailored properties of polymer composites for edible food packaging materials.

3.7.3 Health Beneficial Property

The sources of cellulose include fruits with peel, green vegetables, peas, cereal brans which are directly eatable by human being. The cellulose is not considered as essential nutrients; however, the intake of cellulose is considered as health beneficial. In this regard, cellulose, its derivatives, and nanoforms have attained a great interest in the current trend of product development. Cellulose is an insoluble fiber, which has an ability to absorb water in human intestine. However, the beneficial bacteria present in large intestine can break cellulose-based materials which can be absorbed in human body. The health benefits of cellulose-based components include weight loss, lowering blood sugar, glucose level in body, lipid lowering activity, and provide a laxative effect. However, cellulose-based materials may produce excessive gas, loose stools, and allergic reactions.

3.7.4 As Emulsifying and Stabilizing Agents

The nanocellulosic materials have an ability in forming stable emulsions, which helps in improving the texture and suspension of emulsion-based coatings. The nanocellulose can be used for developing water/oil-based emulsions, and it is considered as natural emulsifier and stabilizers. The nanoemulsion-based edible coatings can also be developed using nanocellulosic materials. The functionalized nanocellulose and BNC are also used in stabilizing the oil-in-water-based emulsions.

3.7.5 Nanocellulose-Based Antibacterial Materials and Other Properties

The nanocellulose materials have very less antimicrobial and antifungal activity, which can be modified using various modes of action. The surface of nanocellulosic materials can be modified utilizing different molecules, and conjugation of antimicrobial agents with nanocellulosic materials can also be obtained using different cross-linkers. The general antimicrobial agents that are conjugated with nanocellulosic materials include organic compounds, metal NPs, antibiotics, metal oxide NPs, etc. Further, the nanocellulose-based materials can be provided with antioxidant property, when polyphenolic and active agents are added to it via surface modifications. Further, various bioactive molecules are added to nanocellulosic materials including BC for improved packaging properties.

3.8 Traits of Nanocellulose Biocomposites for Edible Food Packaging

The well dispersion of nanocellulose in various matrix materials helps to improve the characteristic attributes of packaging materials such as barrier, thermal, mechanical, wettability, and other properties to be acted as ideal packaging materials for improved shelf life of food products. Since the past decades, the nanocellulose-based polymer composites are considered as emerging materials to be used in various forms of food packaging materials. The high mechanical strength, water resistance property, stability, barrier properties make it a superior candidate for edible food packaging materials. In this regard, nanocellulose-based materials are widely utilized as a potential alternative for available petrochemical-based packaging materials. In Table 3.2, a brief discussion about the available edible packaging materials has been made.

3.8.1 Barrier Property

The nanocelluloses such as CNC, CNF, CNW, and BCN have a capability in improving the barrier properties of edible food packaging. As discussed earlier, the cellulose-based nanomaterials can form strong network via hydrogen bonding, which make gaseous molecules hard to pass through the composites by enhancing the barrier properties. The improved barrier properties of nanocellulose at its nanodimension and its composites can be related to the formation of dense and percolated network formed by nanocellulose with various dimensions. The formed dense network creates a tortuous path for the gaseous molecules and thus decreases the permeability of the films. Further, it has been observed that the oxygen barrier properties of CNF-based films are better than CNC-based films, as CNF-based films can form a high entanglement within the matrix materials. However, nanocellulose being hydrophilic in nature may provide low water barrier properties. The nanocellulose materials at high relative humidity may have a problem of structural disintegration which decrease the oxygen and water vapor barrier properties [75]. However, the nanocellulose materials can be modified to reduce its hydrophilic

Table 3.2 Packaging properties of some available edible packaging materials in terms of edible films and coatings

Sl. no.	Edible packaging composition	Packaging properties
1	CNF and mgCNF-reinforced CS [51]	**Mechanical properties:** Tensile properties: CS: ~6.27 ± 0.7 MPa; CS/1.5mgCNF: ~57.86 ± 14 MPa **Young's modulus:** CS: ~462.36 ± 64 MPa; CS/1.5mgCNF: ~2348.52 ± 276 MPa **Composition of iron:** **CS-mgCNF1.5: 6.3** ± 0.06 ppm **Thermal properties:** CS: 10% loss in weight: 80 °C; CS: 50% loss in weight: 334 °C CS-CNF0.5: 10% loss in weight: 116 °C; 50% loss in weight: 342 °C CS-mgCNF0.5: 10% loss in weight: 95 °C; 50% loss in weight: 351 °C **Transparency in UV region:** CS: 85%, CS-CNF0.5:71%, CS-CNF1:69%, CS-CNF1.5:64%, CS-mgCNF0.5:42%, CS-mgCNF1:33%, CS-mgCNF1.5:18%
2	CNF-reinforced CS [72]	**3% CNF/CS (1%)** Color: L:86.3, a:−0.64, b:3.02; WVP: 14.15 g.mm/kPa.d.m^2 **3% CNF/CS (2%)** Color: L:71.7, a:−0.60, b:6.08; WVP: 13.62 g.mm/kPa.d.m^2
3	Nanocellulose fibers-reinforced sodium caseinate [73]	**1 wt% nanocellulose fibers-reinforced sodium caseinate** Opacity: 2137.7 ± 434.0 AU nm/mm; contact angle: 32.8 ± 4.2°; Average porosity: 0.116% **3 wt% nanocellulose fibers-reinforced sodium caseinate** Opacity: 3215.2 ± 214.9 AU nm/mm; contact angle: 29.7 ± 1.5°; Average porosity: 0.194%
4	CNC-reinforced pectin [74]	**2% CNC-reinforced pectin films** Glass transition temperature (Tg): 57.9 ± 0.66 °C; melting temperature (Tm): 167.79 ± 5.56 °C; melting enthalpy (ΔH): 128.65 ± 1.27 J g^{-1} **7% CNC-reinforced pectin films** Tg: 57.39 ± 0.39 °C; Tm: 165.36 ± 0.94 °C; melting enthalpy (ΔH): 140.5 ± 7.5 J g^{-1}
5	Bacterial CNF (BCNF)-reinforced fish myofibrillar protein [63]	**2 wt% BCNF-reinforced fish myofibrillar protein** TS: 6.84 ± 0.09 MPa; elongation at break: 90.49 ± 2.93%; WVP : ~2.95 × 10^{-10} g/ms Pa; contact angle: ~82.76° **6 wt% BCNF-reinforced fish myofibrillar protein** TS: 8.94 ± 0.33 MPa; elongation at break : 87.63 ± 2.52%; WVP: ~2.28 × 10^{-10} g/ms Pa; contact angle: ~87.70°

(continued)

Table 3.2 (continued)

Sl. no.	Edible packaging composition	Packaging properties
6	Nano-BC-reinforced agar [64]	**3 wt% nano-BC-reinforced agar films** Moisture content: ~19.50%; WS: ~21.56% TS: ~27.95 MPa WVP: ~8.69×10^{-11} gm/m^2 Pa s **10 wt% nano-BC-reinforced agar films** Moisture content: ~13.79%; WS: ~18.55% TS: ~44.51 MPa WVP: ~6.88×10^{-11} gm/m^2 Pa s

WVP: Water vapor permeability; *TS*: tensile strength; *WS*: water solubility; *UV*: ultraviolet; *mgCNF*: magnetic cellulose nanofibers

property at high-moisture environments via chemical modifications. In this regard, the metal oxides can be used to modify properties of nanocellulose for improved barrier properties. As discussed, CNFs can be developed by mechanical fibrillation method, where the pure films of these kind of CNFs have very good oxygen barrier properties. Interestingly, CNF provides tailor-made barrier properties, where partially acetylated CNF-based films are also utilized for modified atmospheric packaging. The barrier properties of nanocellulose-based composites are better than the available synthetic polymer that are used in the food packaging industry. The CNF-based films are more complex and compact in comparison with the cellulose fiber-based films, which make them a potential candidate for water barrier properties. The ordered structure of nanocellulose and crystallinity behavior of nanocellulose create a percolation network in composites which slower the diffusion of gaseous molecules, thus reducing water vapor permeability (WVP).

3.8.2 Mechanical Property

Crossbonding in cellulose-based nanomaterials provides the maximum strength in composites. CNFs have an exceptional mechanical property and thus are used as a potential candidate in food packaging materials for transportation purpose. The fruits and vegetables are very much prone to get effected by mechanical damages such as abrasion during transportation. The mechanical properties of nanocellulosic materials are better than the available lignocellulosic biomass materials for having uniform morphological structures. The mechanical properties of nanocellulose also depend on the percent crystallinity, which in turn depends on the source. The nanocellulosic materials are available in its various polymorphic forms which further differ the composite properties.

3.8.3 Thermal Property

The nanostructured forms of cellulose are thermally stable than the cellulosic materials. The thermal properties are dependent on the source materials, extraction procedure, condition of processing, etc. The nanocellulosic materials provide higher crystallinity (may get varied for nanofibers, nanocrystals, whiskers, etc.), flexible orientation, and removal of non-cellulosic materials, which in turn improves the thermal stability of nanocellulosic materials. The composites of nanocellulose are influenced by the intermolecular bonding between filler and matrix materials. The surface chemistry of nanocellulose helps to improve the thermal properties due to intermolecular interaction forming percolation network. However, nanocellulose extracted using sulfuric acids contains sulfate groups, which reduces the thermal stability of nanocellulose materials. However, the sulfate groups on nanocellulose can be functionalized to obtain improved thermal stability.

3.8.4 Optical Property

The optical property of nanocellulosic composites is influenced by various types of cellulosic nanomaterials such as CNC, CNF, CNW, and others. The transparency and color coordinates (L, a*, b*, hue, and chroma) are found to influence by the inclusion of functionalized nanocellulosic materials in different matrix materials.

3.8.5 Others

The other properties of nanocellulosic composites such as moisture content, water solubility (WS), wettability, and thickness are affected greatly by the different types of matrix materials.

3.9 Tailored Nanocellulose-Based Materials for Edible Food Packaging

As discussed earlier, edible coating is an environmental-friendly technique for providing a protective coating layer to maximize the quality and shelf life of fresh fruits and vegetables. Focusing on different biopolymers such as polysaccharides, alginates, CS, proteins, gelatin (fish/pork), and lipids has been gaining an increased attention regarding its excellent cost-effectiveness nature and film-forming function [76–80]. The biodegradable, non-toxic, high surface area with high crystallinity nanocellulose including NFC, CNC, and BNC with their unique barrier layer properties such as enhanced oxygen and water vapor properties is used for food packaging. The percolated structure of CNF and its long, flexible fiber structure with good mechanical properties provide proper dispersion in the matrix. Various investigations have been put forward to utilize nanocellulose-derived biopolymers as edible films in food packaging. An article was reported on edible films from pectin, a natural polymer reinforced with crystalline nanocellulose (2, 5, and 7 w/w %) developed using solution casting evaporation method [74]. The most important functional properties in case of edible films and coatings are their barrier properties for water vapor and oxygen, carbon dioxide gases, migration of compound, appearance such as color and gloss, physical and mechanical protection for proper handling of food products. The high surface area feature of nanoscale cellulose demonstrates its application as reinforcements for polymer matrix giving a new research area. However, its poor dispersion is a critical concern, which affects the mechanical properties of the composite materials leading to a challenging task. Thereby, modification of the nanofiller is needed for good dispersion in the polymer materials via physical and chemical methods [81, 82].

3.9.1 Chemical Modification

The chemical modification of nanocellulose can be obtained via utilizing the available hydroxyl groups on nanocellulose. The chemical agents such as compatibilizing agent, coupling agent, acetylating agent, polymer grafting agent, copolymerization, non-covalent surface modification, sulfonation, TEMPO (2,2,6,6- tetramethylpiperidine-1-oxyl)-mediated oxidation, esterification, etherification, and silation are used to improve the properties of nanocellulose for food packaging application. The chemical modifications further help to improve the interfacial compatibility with the various polymer matrices by changing and controlling the molecular interactions. However, in case of surface compatibilization of nanocelluloses, one reactive group should be present. On the other hand, for the copolymerization-aided modifications, at least two functional groups are required for improved interactions. In this case, the functional group in cellulose can react with polymer matrix via grafting, radical reactions, and organometallics for improved properties [83, 84].

3.9.2 Physical Modification

The physical treatment of cellulose is a challenging method to alter the cellulose polymorphs, thereby improving the matrix mechanical bonding and other properties [85]. The various physical methods such as cold plasma (electric discharge), dielectric-barrier discharge (creation of ions, free radicals, and other species generated by high energy electrons), ultrasonification (include no use of organic solvent), and irradiation by gamma rays are used to enhance the CNC's adhesion properties, and further, mechanochemical treatment is applied through shearing and compressing forces, etc [86–89].

3.9.3 Development of Bionanocomposites

The alternative of petroleum-based plastics such as the active-biobased food packaging has received a great attention for improved hydrophobicity, mechanical properties, and antimicrobial activity of individual biopolymers. Li et al. have reported that developing CW with CS matrix displays increase in tensile strength (TS) up to 120 MPa by incorporating 20% CNW along with excellent thermal stability and water resistance [90]. A work has been reported on the preparation of bionanocomposites using casting/evaporation method based on WG, CNC, and titanium NPs (TiO_2), which provide improved breaking length of 56% and burst index of 53% by incorporating 7.5% CNC and 0.6% TiO_2 for 3 coating layers over kraft paper. The bionanocomposite also showed excellent antimicrobial activities almost 100% against yeast *Saccharomyces cerevisiae*, gram-negative bacteria *Escherichia coli*, and gram-positive bacteria *Staphylococcus aureus* [68]. Seoane and coworkers prepared PHB/CNC bionanocomposite that has attained improved mechanical and barrier properties, when optimized concentrations of CNC (6 wt%) have been incorporated, which further act as an alternative for polypropylene in packaging applications [91]. Dehnad et al. studied the shelf life of meat that can extend meat shelf life and provide maintained quality for 6 days when compared with nylon packaging [92]. Jung et al. have investigated developing coating using Fe^{3+}-anthocyanin complexation with CNF/SA using layer-by-layer (LBL) coating method on blueberries and found that leakage of anthocyanin pigments can be eliminated [93]. Another work has been reported demonstrating the tailor-made properties of optical, mechanical, thermal, and texture properties using CS and magnetic CNF as edible nanocoating. The investigation shows improvement in storage quality of the coated cut pineapple using texture and gravimetric analysis. Another study on acerola puree and alginate reinforcing with CW provides improved water vapor barrier property in the form of nanocomposite edible film [62].

3.10 Case Studies on Nanocellulose-Based Edible Food Packaging

In this section, a brief discussion has been made about the use of various nanoforms of cellulose such as CNC, CNF, BNC, and CNW, which are utilized with other biopolymers as edible food packaging materials. However, the characteristic attributes of fabricating composite-based edible packaging can be modified using plasticizers, flavoring agents, and others as shown in Table 3.3. Different types of cellulose-based materials are utilized as primary, secondary packaging, flexible packaging, developing containers, etc. However, in this section, the use of nanocellulose as a component of edible packaging materials has been made.

Table 3.3 Application of cellulose nanostructured materials in edible food packaging

Sl. no.	Filler type	Matrix type	Targeted food product and obtained property	References
1	Nano-BC	Agar	Edible films (targeted food packaging application) **Property:** Tailor-made properties of dispersibility of nanofiller, thermal properties, mechanical properties, moisture content, WS, WVP	[64]
2	BCNC	Gelatin	Edible films (low-cost food packaging materials) **Property:** Improve properties of gelatin and BCNC-based edible films, improve mechanical and thermal properties, reduced moisture affinity of neat films	[102]
3	CNF	Mango puree	Edible films **Property:** Improve mechanical property Improve barrier property	[103]
4	MCC NPs	HPMC	Edible Films **Property:** Better moisture barrier properties, reduced water barrier properties	[98]
5	CNC	Gelatin	Edible coating on fresh strawberry **Property:** Antimicrobial effect Reduce weight loss Prolong shelf life Retention of ascorbic acid	[94]
6	CNF and magnetic CNF	CS	Edible coating on cut pineapple **Property:** Improve mechanical property Improved thermal property Improved optical property Maintained quality of cut pineapple during storage	[51]
7	BCNF	Fish myofibrillar protein	Edible films **Property:** Improved thermal property Reduce WVP Reduced swelling index	[63]
8	CW	Alginate–acerola puree Plasticizers: Corn syrup	Edible films **Property:** Improved TS Improved Young's modulus Improved water vapor barrier	[62]

(continued)

Table 3.3 (continued)

Sl. no.	Filler type	Matrix type	Targeted food product and obtained property	References
9	Crystalline nanocellulose	Pectin	Edible films **Property:** Improved TS Improved elongation at break Improved water barrier properties	[74]
10	CNF	Gelatin Glycerol (A plasticizer)	Edible films **Property:** Tunable mechanical property Tunable optical property Tunable barrier property	[104]
11	CNF Titanium dioxide REO	WPI	Edible films Food products: Lamb meat **Property:** TiO_2 and REO improve the organoleptic property of lamb meat Increase shelf life of lamb meat Inhibit the growth of spoilage and pathogenic bacteria in meat	[101]
12	Nanocellulose	CS Glycerol (plasticizers)	Edible films **Property:** Good dispersion Tunable properties in terms of TS, elongation at break, Young's modulus, barrier properties, thermal properties	[99]
13	CNF	CS	Edible films and active packaging **Property**: Antimicrobial property Barrier property	[100]
14	NCF	Sodium caseinate Glycerol as plasticizer	Edible films **Property:** Less transparency for composite films More hydrophilic than neat sodium caseinate films Improve mechanical property with the aid of nanofiller material	[73]

CNF: Cellulose nanofiber; *CS*: chitosan; *BCNC*: bacterial cellulose nanocrystal; *BC*: bacterial cellulose; *MCC*: microcrystalline cellulose; *TS*: tensile strength; *HPMC*: hydroxypropyl methylcellulose; *CNC*: cellulose nanocrystal; *REO*: rosemary essential oil; *WPI*: whey protein isolate; *CW*: cellulose whisker; *NPs*: nanoparticles; *NCF*: nanocellulose fiber; *WS*: water solubility

3.10.1 Storage Study of Edible Packaged Food Products

Nanocellulose has been widely utilized for the fabrication of composite materials to be used as edible coating materials for enhanced shelf life of food products as given below:

- Storage study of fresh strawberry has been done using edible coating based on CNC-reinforced gelatin plasticized with glycerol (storage condition: under refrigeration for 8 days) [94].

- Storage study of strawberries has been done using apple pectin, CNCs, essential oils of lemongrass (storage condition: 8 days and 10 °C ± 1 °C) [95].
- Storage study of pears has been done using CNC-reinforced CS-based edible coating (storage study: 3 weeks and ambient storage: 20 ± 2 °C and 30 ± 2% RH) [96].
- Storage study of cut pineapple has been done using magnetic CNF (mgCNF)-reinforced CS-based edible coating (storage condition: 12 days at room temperature) [51].
- Storage study of grape fruits has been done using CNF, carrageenan, glycerol, and HPMC (storage condition: refrigerated storage for 41 days) [97].
- Storage study of strawberry has been done using CS and cellulose nanofibril-based composites (storage condition: cold storage for 21 days) [72].

3.10.2 Tunable Packaging Property as Edible Packaging Materials

The nanocomposite based on CNF-reinforced mango puree provides tunable packaging properties for developing edible films. The incorporation of CNF provides improved mechanical properties in terms of TS and Young's modulus for forming a fibrillary network within mango puree. The development of edible films based on MCC and lipid-coated MCC NP (LC-MCC) incorporated HPMC has a promising attribute in the field of food packaging for being flexible and transparent materials. The TS of MCC and LC-MCC-incorporated HPMC films provide increased TS up to 53 and 48%, respectively, in comparison with neat HPMC films [98]. Additionally, the fabrication of CNF and magnetic CNF (mgCNF)-dispersed CS-based edible coating materials can adequately improve the packaging quality and shelf life of cut pineapple in terms of firmness and weight loss [51]. As shown in Fig. 3.8, the tailored surface morphology of CNF (Fig. 3.8a) can be obtained via developing mgCNF (Fig. 3.8b), where iron particles get adsorbed onto CNF via single-step coprecipitation method. Further, the use of CNF, CS, and curcumin can provide uniform edible films (Fig. 3.8c). Further, the appearance of stored uncoated (e″) and dipped coated (e′) cut pineapple using curcumin-loaded CNF-dispersed CS solution at refrigerated storage for 1 month time duration has been shown. In this regard, several types of nanocellulosic materials are used to improve the appearance and quality of various food products during storage time. Further, CW from cotton fibers is used as a filler material in developing composites of alginate–acerola puree with plasticizer which can be used as edible food packaging material with a tailor-made property of overall tensile property and water vapor barrier property [62]. The fabrication of pectin/crystalline nanocellulose-based biocomposite edible films delivers an improved film property in terms of mechanical property, and barrier property [74]. Further, this kind of pectin-based packaging materials can be considered as fully biodegradable and renewable packaging materials for food products. The properties of CS-based edible films can be tailor-made with the aid of nanofiller nanocellulose and glycerol as a plasticizer. With the aid of central composite design considering two variables, CNF concentration (0–20 g/100 g) and glycerol (0–30 g/100 g) are used for optimizing the development of CS-based edible films, where with 15 g/100 g CNF concentration and 18 g/100 g glycerol concentration are considered as optimum condition [99]. The CNF-dispersed CS films have antimicrobial property, with an efficacy in improving barrier property [100]. Further, Pereda and others have developed sodium caseinate/nanocellulose-based edible films, where the TS and tensile modulus have been found to be improved with increased nanocellulose [73]. The application of CNF, TiO_2, rosemary essential oil (REO), and whey protein isolate (WPI)-based edible nanocomposite films helps in improving the sensory quality, organoleptic properties, and shelf life of lamb meat [101]. The synthesis of biocomposite films has a great interest in delivering the controlled release of antimicrobials. The fabrication of WPI/CNF composite films containing TiO_2 and REO is very efficient in preserving quality of meat for maintaining food properties, where the films with TiO_2 and REO have an ability to inhibit pathogenic bacteria in meat, and are considered economic. The nanocellulose and CS-based edible films in packaging ground meat have an ability to decrease the lactic acid bacteria, which helps in improving the shelf life of ground meat [92]. Further, the storage study of grape fruits with edible coating application using CNFs, HPMC, Ꝁ-carrageenan has been reported, where application of spray system is utilized for coating fruits [97].

BC is obtained in its pure form and provides superior mechanical, thermal, and water holding property, which make it a suitable candidate for edible food packaging in comparison with plant-based cellulose. The biocomposite edible films based on BCNF-reinforced fish myofibrillar protein have a positive impact in improving the film properties such as physical property, thermal property, and others [63]. The addition of 6 wt% of BCNF in the preparation of composites improves the TS about 49% than the neat films. The composite films further improve the physical properties and thermal properties, and reduce WVP and solubility indexes. The TS of the composite edible films for BCNF-reinforced fish myofibrillar protein is ~ 6, ~ 6.84, ~ 7.19, and ~ 8.94 MPa for 0, 2, 4, and 6 wt% nanofiller, respectively. Moreover, the WVP of the composite edible films for BCNF-reinforced fish myofibrillar protein is $\sim 3.41 \times 10^{-10}$ g/ms Pa, $\sim 2.95 \times 10^{-10}$ g/ms Pa, $\sim 2.73 \times 10^{-10}$ g/ms Pa, and $\sim 2.28 \times 10^{-10}$ g/ms Pa for 0, 2, 4, and 6 wt% nanofiller, respectively. Interestingly, the swelling index of the developed edible films is also improved ~ 215, ~ 212, ~ 199, and $\sim 172\%$ for 0, 2, 4, and 6 wt% nanofiller, respectively. The use of nanofiller nano-BC in the development of agar-based edible films has an improved thermal stability and mechanical properties [64]. The various contents of nano-BC have different effectivenesses in agar-based edible film preparation such as: (i) 3–5% nano-BC has a good dispersion in agar-based films; (ii) 10% nano-BC decreases moisture content by 60.4% compared with neat agar films; (iii) 10% nano-BC decreases WS by 13.3% compared with neat agar films; (iv) 10% nano-BC decreases WVP by 25.7% compared with neat agar films; (v) 10% nano-BC-dispersed agar-based edible films provide TS and elongation at break of 44.51 MPa and 13.02%, respectively; (vi) maximum mass loss

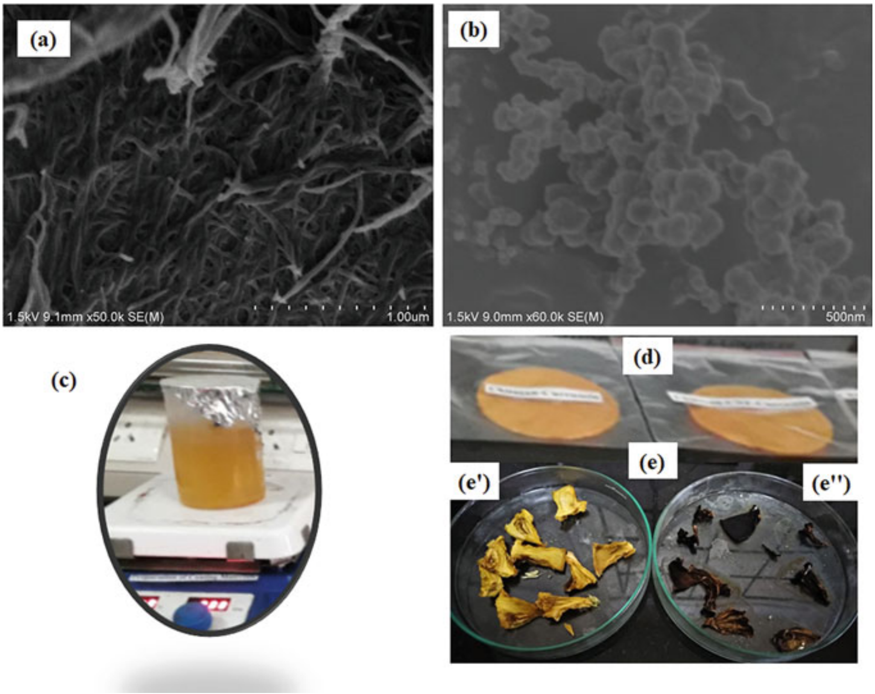

Fig. 3.8 Surface morphology of (a) CNF, (b) functionalized CNF (mgCNF), (c) Preparation of edible coating solution, (d) Edible Films and (e) storage of cutpineapple using CNF-dispersed CS-based edible coating at refrigerated storage for 1 month time duration with edible coating (e') and without edible coating (e'')

temperatures for neat agar and 10% BC-reinforced agar-based edible films are 303.9 and 315.6 °C, respectively [64]. As discussed, BC as long fiber form can be obtained from *G. xylinus* [102]. The BCNC is obtained following acid hydrolysis process at various processing conditions. The use of bacterial cellulose nanocrystal (BCNC) as a nanofiller material in developing gelatin-based nanocomposite forms a percolation network, which resulted in improved mechanical property such as incorporation of 3 and 4 wt% BCNC in gelatin-based matrix having TS of 103.1 and 108.6 MPa, respectively. Additionally, the reinforcements of BCNC improve the thermal stability, degradation temperature, and dynamic mechanical property of gelatin.

3.11 Conclusion

The noteworthy properties of nanocelluloses such as abundancy, renewable resource, reduced carbon footprint, biocompatible, non-toxic, high strength, light-weight, dimensional stability, thermal stability, optical transparency, and reduced oxygen permeability make it a potential candidate to be used in edible food packaging. Moreover, the nanocellulose-based edible coating has been utilized with other applied packaging technology for obtaining improved shelf life of perishable food products. In this regard, the synergistic effect of edible nanocoating and non-edible food products as secondary packaging materials can be utilized for transportation of food items. Further, research is going on to apply various packaging technologies such as reduced oxygen storage, passive and active modified atmospheric storage, controlled atmospheric storage for improved shelf life of food products.

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Chapter 4
Chitosan-Based Nanostructured Materials in Edible Food Packaging

Tabli Ghosh, Kona Mondal, and Vimal Katiyar

4.1 Introduction

The chitosan (CS) nanostructured material is one of the most investigated bio-nanomaterials in developing edible food packaging for perishable food products including fruits and vegetables, fish products, meat, and meat products, and others [1–4]. The increased consumer demands for microbiologically safe and ready-to-eat food products has increased the use of CS, its derivatives, and nanomaterials in developing edible food packaged products. Further, utilization of CS as a constituent for several packaging applications can help in waste management of seafood-based waste and marine waste. Thus, the researchers are continuously investigated for CSNPs as a component in fabricating new types of edible films and coatings for enormous food applications. In this regard, the several aspects of CS nanostructured material-based edible food packaging in terms of packaging aspects (different packaging forms, microbiologically safe food, tailored packaging property), environment aspects (waste utilization, sustainable packaging, biodegradability), and targeted perishable food products are represented in Fig. 4.1. CS, its derivatives, and nanostructured materials are attractive biopolymers for developing food packaging using dipping, wrapping, coating, spraying, etc. Besides, CS-based nanocomposite is also developed to deliver the antimicrobial and other noteworthy properties of CS to other biopolymeric materials and is widely used to improve the storage life of perishable food products as a solution to the waste management obtained from seafood, marine waste, etc. CS being a hydrophilic biopolymer can easily get solubilize in food products providing high water activity and poor barrier

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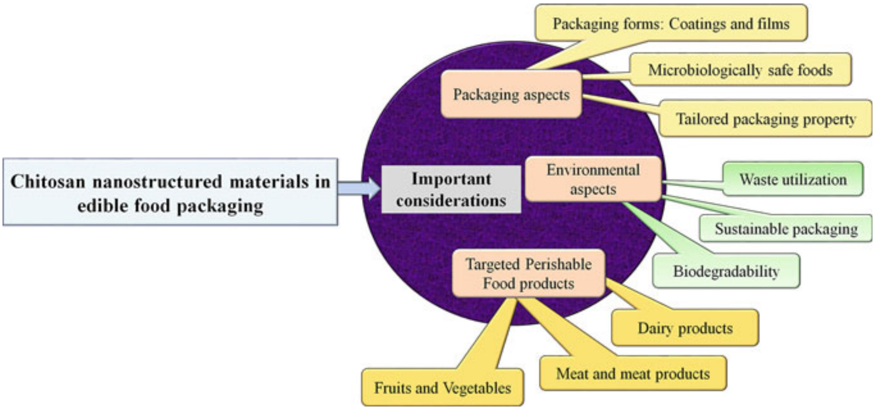

Fig. 4.1 Several important considerations of CS nanostructured materials in edible food packaging

properties, which reduces the use of this biopolymer for commercialization applications. In this regard, the solubility of CS can be improved by fabricating its nanoforms and further helps to offer tunable properties in terms of antimicrobial activity, interactive ability, and solubility [1].

CS is a unique cationic polysaccharide with several functional properties including non-toxicity, biocompatibility, antioxidant, antimicrobial, and other health beneficial property, film-forming property, lipid lowering activity, gelling property, etc. The mentioned properties make CS, its derivatives, and nanostructured form a potential candidate to be used in edible food packaging which is focused to discuss in the present chapter. Additionally, the characteristics attributes of CS, related derivatives, and CS-based nanostructured materials have attained a remarkable importance for several existing noteworthy functional properties. The biological properties of CS are bioadhesive in nature, adsorption enhancers, cytocompatible, antimicrobial active, antioxidant active, macrophage activation, and others, which depend on the degree of acetylation and molecular weight of CS. The physicochemical properties of CS include solubility, crystallinity, viscosity, and others, which vary from source to source. Further, it is worth mentioning that the remarkable biological, antimicrobial, and physicochemical properties of nanochitosan (NCS) provide multifaceted applications specifically in sustainable food packaging.

CS nanostructured materials have some advantageous properties in comparison with commercial CS including high surface area, small particle size, high reactivity, and compactness. In this regard, the fabrication of CSNP is commonly obtained using ionotropic gelation method, which is a simple method and the desired particle size can also be optimized. Besides, the other methods for developing CS-based nanostructured materials include emulsification solvent diffusion, emulsion-based solvent evaporation, microemulsion, and others. However, the degree of deacetylation and molecular weight is the crucial factors to affect the particle size and surface charge of CS nanostructured materials. In this regard, a detailed discussion related to different fabrication techniques to develop CS-based nanostructured materials will be made.

The CS nanostructured material-based edible food packaging provides tailored-made packaging properties in terms of barrier, mechanical, thermal, color, and other properties. Thus, the use of NCS with other biopolymeric materials can also be used to improve the film properties such as barrier properties. The combined use of NCS and pectin provides functional packaging materials with improved mechanical property and reduced water solubility [5]. The other biopolymeric materials which are used in combination with NCS include cellulose, and its derivatives, tara gum, pectin, proteins, etc. The carboxymethyl cellulose (CMC) and CSNP-based composite films provide high mechanical strength and stability [6]. The use of CSNP in developing tara gum-based nanocomposite edible films provide improved mechanical and physicochemical properties [7]. The CS nanomaterials-based edible films and coatings are extensively used for extending the shelf life of meat products, bread products, fresh products, dairy products, etc. Additionally, CSNP is used as edible-coating materials for several fruits and vegetables such as peach fruit [8], banana fruits [2], table grapes [9], cucumber [10], tomato [11], strawberry [12], apple [13], and others. CS and CSNPs are used for providing improved microbiological properties of fish fingers, where psychrophilic bacteria, *coliform* bacteria, total bacterial count (TBC), *coliform* bacteria can be reduced using the specified coating materials [3]. The use of CSNPs as an active edible coating on fish fingers can reduce oil uptake [14]. The edible coating of refrigerated silver carp fillets using NCS helps to improve the quality during storage [15]. Edible coatings based on CSNPs with essential oils (EOs) are used to improve the pork quality such as antioxidant and antimicrobial property [4]. However, the CSNPs with large sizes can be used as a natural preservative for meat and meat products. Moreover, CS is used as antimicrobial packaging, active packaging, intelligent packaging, biodegradable films/coatings and edible packaging [16]. In this regard, the chapter focuses to discuss the use of CS nanostructured as edible food packaging materials as blends, composites and coatings to improve the packaging and food properties. Further, CS having functional groups such as amine, primary hydroxyls, secondary hydroxyls provide an ability for chemical modifying the packaging properties such as mechanical and biological properties.

Based on this discussion, the utilization of CS nanostructured materials in edible food packaging will be discussed in the current chapter. Further, the storage life of edible-coated food products using CSNPs will be detailed. The modification technique of CS bionanomaterials to modify the inherent properties will be discussed in the current chapter.

4.2 History Outline of Chitosan and Its Versatile Application

As discussed in Chap. 2, CS, a deacetylated product of chitin, has attained great interest in several multifaceted advanced applications [17, 18]. In 1811, chitin was first discovered and being extracted from mushroom by the French professor, Henri Braconnot and the name chitin was coined by Odier in 1823 [19]. In 1820, chitin was isolated from insects. Chitin was the first polysaccharide detailed among available, while cellulose was described 30 years later than chitin. In later days, the CS was discovered by Rouget in 1859, when chitin is heated in a concentrated potassium hydroxide solution and was found to be soluble in acid solution [20]. Besides, edible food packaging, CS, and its derivatives are extensively utilized in food and bioengineering application including enzyme immobilization, encapsulation of bioactive compounds, as a delivery agent for drugs, plant growth promoter for its several noteworthy properties. The CS nanostructured materials are further considered as a potential candidate to make use in edible packaging for noteworthy properties. Interestingly, CS is also used as edible films and coatings for the improved shelf life of agricultural products, bread, dairy products, meat products, etc. [16].

4.3 Sources and Synthesis Approaches of Chitosan

In later days, the several sources of chitin, the precursor of CS, include insects (ladybug, wax worm, silk, butterfly), molluscs (squid pen, shell oysters), fungi (*Mucor rouxii, Aspergillus niger, Penicillium chrysogenum, Lactarius vellereus*), and crustaceans (Shrimp, lobster, crabs) as shown in Fig. 4.2a. Interestingly, chitin is widely extracted from seafood waste, crustacean waste, fish waste, shrimp waste, etc. The shells of shellfish are a crucial reason for generating pollution in coastal areas, where the biowaste can be utilized as a source for producing chitin and CS [21]. Additionally, chitin is available as three crystalline forms such as α, β, and γ forms (Fig. 4.2b) [22, 23], where α chitin is the most stable and abundant form of chitin, being extracted from yeast, shrimp shells, insect cuticles, lobsters, and in α chitin, the chitin fibrils are arranged in an antiparallel orientation with maximum bonding and crystallinity index of 80%. The polymer chains in β chitin are arranged in a parallel orientation, and the crystallinity index is found to be around 70%. Additionally, γ chitins are a combination of α and β and can be obtained from cocoons of insects [22].

From the chitinous sources, chitin is extracted following chemical and biological methods which include deproteinization and demineralization process (Fig. 4.2c). The chemical method of chitin fabrication involves three steps of processing such as deproteinization, demineralization, and decoloration. The deproteinization of chitinous waste involves the removal of proteins, where the bonds between protein

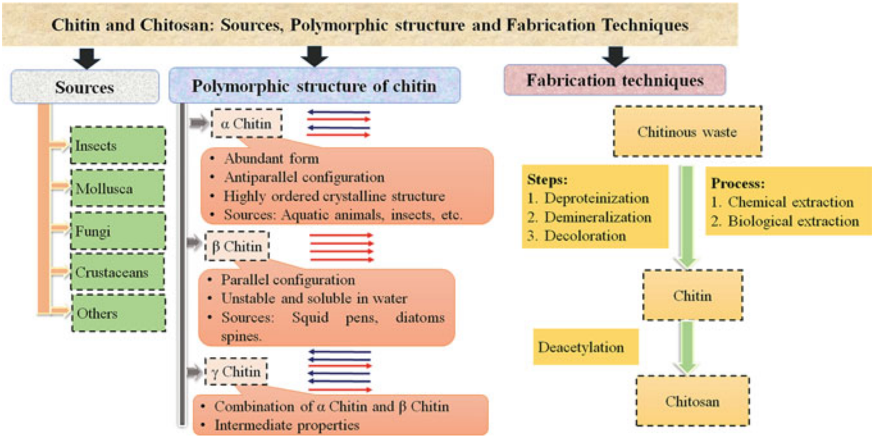

Fig. 4.2 Sources of CS; Polymorphic forms, and Extraction process

and chitin are disrupted via depolymerization. In this process, the chemicals used are sodium hydroxide, potassium hydroxide, sodium carbonate, and potassium carbonate. The demineralization of chitinous waste includes the removal of minerals such as calcium carbonate via acid treatment using hydrogen chloride/sulfuric acid/nitric acid. The decoloration step involves the removal of pigments such as carotenoids and others using acetone or any other organic solvent mixture. The biological method of chitin extraction includes enzymatic deproteinization and fermentation using microorganisms. The protease enzymes for deproteinization of chitinous waste involve the use of enzymes such as papain, trypsin, alcalase, and pepsin. Additionally, the fermentation method of deproteinization includes lactic acid fermentation and non-lactic acid fermentation. The deacetylation of chitin can be obtained using alkalis, where chitin is treated with hot and concentrated solution of sodium hydroxide and deacetylation of 85–99% is obtained [24].

4.4 Prospective of Chitosan Nanostructured Materials in Edible Food Packaging

CS is renewable and non-allergic material which is used in developing edible food packaging materials. CS nanostructured materials are also utilized as drug delivery materials. The CSNP with insulin has an ability to enhance the pharmacological bioavailability. CSNPs have some remarkable attributes such as non-toxicity, small size, high surface reactivity provides additional benefits to be used in edible food packaging. As shown in Fig. 4.3, the NCS is obtained in the form of CSNP, nanofiber, nanocrystals, which have several characteristics attributes which increased its use in edible food packaging.

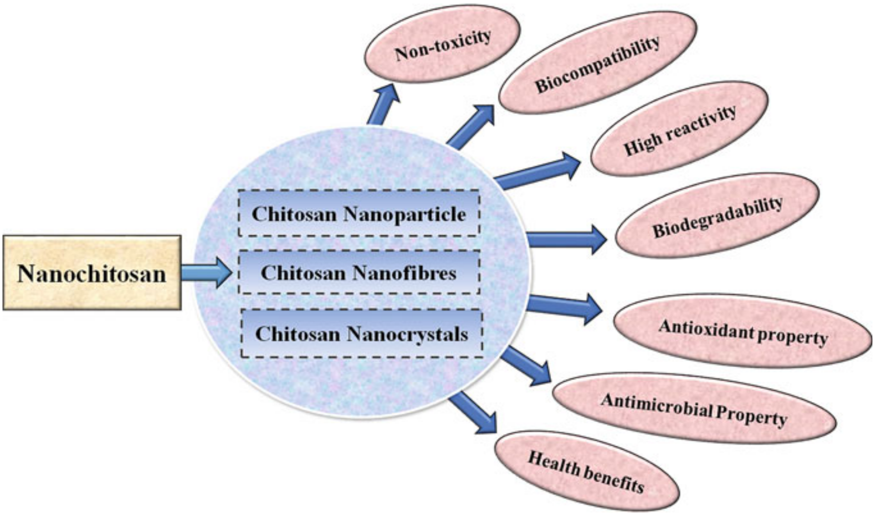

Fig. 4.3 Prospective of NCS in edible food packaging

4.4.1 Biocompatibility and Non-toxicity

The non-toxicity and biocompatible nature of NCS make it a potential alternative to use as food components and to develop edible films or coatings for an improved shelf life of food products. The toxicity determination of CS (source: fungi and shrimp) and NCS (fabrication process: ionic gelation) has been evaluated using brine shrimp bioassay, and rat bioassay [25]. The toxicity evaluation using brine shrimp bioassay shows that there are no toxic components at several concentrations such as 5000, 10,000, 15,000 ppm. The blood parameters such as biochemical property of blood, liver, kidney functions, histopathology changes in liver, stomach and kidney tissues can be determined for toxicity evaluation. The normal functioning of the liver when feed with CS and NCS is observed by alanine amino transaminase (ALT) and aspartate amino transaminase (AST) values. Further, the levels of creatinine, urea, and uric acid levels in the blood can provide the normal kidney functioning. Further, the intake of 100 and 200 mg kg^{-1} bw rat CS and NCS has no effectiveness toward liver and kidney functions. Histopathology of liver, kidney, and stomach also provides no obvious changes in the tissues and inflammation, and fibrosis has not found for the same. The biochemical property of blood and oxidative stress is also not affected by the intake of specified CS. Additionally, the CSNPs (50 µg mL^{-1}) extracted utilizing the fungal enzyme *Trichoderma harzianum* are biocompatible in nature which provides no cytotoxicity effect as evident by cell viability and acridine orange/ethidium bromide staining assays [26]. The CS releases amino sugars in human body by the action of enzyme lysozyme which makes it a biocompatible biomaterial [27]. However, the biodegradability rate of CS depends on degree of deacetylation, where the biodegradable nature of NCS is a critical parameter for acute and long-term toxicity. Thus, the CS nanostructured materials due to its non-toxic, biocompatible, and biodegradable nature make it an attractive biopolymer for microencapsulation and edible food packaging.

4.4.2 Antimicrobial Property

CS and its nanostructured forms have antibacterial property which provides a great interest to be used as edible coating and film materials for several food products. However, CS provides antibacterial activity in the acidic medium due to its insoluble nature at pH above 6.5. Additionally, the antimicrobial property of CS depends on the type of sources, degree of deacetylation, physicochemical property, molecular weights, selected solvent, etc. The CS has a better antibacterial property against gram-negative bacteria than gram-positive bacteria. The CSNP is considered as a remarkable antimicrobial agent against several bacteria, viruses, and fungi, which make it a promising candidate against some of the available bactericides and chemical fungicides [1]. In this regard, the CSNPs (low molecular weight and high molecular weight) have an inhibitory action against *Bacillus cereus, Escherichia coli, Listeria innocua, Staphylococcus aureus, Salmonella typhimurium,* and *Yersinia enterocolitica* [28]. The encapsulated CSNP with polyphenols also provides the antimicrobial activity. NCS has better antimicrobial activity than CS for preserving silver carp fillets [15]. The antimicrobial property of NCS is better than CS due to the larger surface area and better interaction with microbes. Additionally, NCS has a better ability to inhibit total volatile basic nitrogen (TVB-N) than CS. Another research reports the antimicrobial activity of CS (sources: crustaceans and fungi) and NCS against gram-positive bacteria, gram-negative bacteria, yeast, and fungi [25].

4.4.3 Antioxidant Property

Additionally, the CSNPs extracted utilizing the fungal enzyme *Trichoderma harzianum* provide antioxidant activity in a dose-dependent manner [26]. The CSNPs with small size ranges and low molecular weight provide antioxidant activity and have scavenging activity against free radicals [1]. The antioxidant property of CS can be evaluated by radical scavenging activity against superoxide anion and hydroxyl radical. The lower molecular weight CS oligomers have better antioxidant activity than higher molecular weight, where the 50% inhibition concentrations for determining superoxide anion scavenging are 5.54, 8.11, 12.15 mg/mL, respectively, for CS oligomers with molecular weights 2300, 3270, 6120 Da. Additionally, for superoxide anion, the maximum inhibiting efficacy of CS oligomers with molecular weights 2300, 3270, 6120, and 15250 Da are 89, 75, 74, and 41%, respectively. For hydroxyl radicals, the maximum inhibiting efficacy of CS oligomers with molecular weights 2300, 3270, 6120, and 15250 Da are 71, 65, 51 and 7%, respectively [29]. The antioxidant activity of high molecular weight CS-based films provides a way to use it as a natural antioxidant material for food application, where high molecular weight CS can be evaluated for radical scavengers against 1,1-diphenyl-2-picrylhydrazyl (DPPH) radicals, hydroxyl radical, superoxide radicals [30]. However, the antioxidant property of hetero-chitooligosaccharide depends on degree of deacetylation and molecular weight [31].

4.4.4 Other Properties

Besides, the above-mentioned properties, the CSNPs have many health beneficial properties, such as the increased immune response of living system, act as a weight reducing agents, lower the high density lipoprotein, provide antioxidant property, decrease the lipid peroxidation, decrease oxidative stress, reduce pain, bioadhesive property, antitumor property, and antiparasitic effects. CS has an ability to bind with fat more effectively in comparison with other glycans, which helps in lowering the high density lipoproteins. Additionally, NCS-based films are used as a therapeutic agent to treat cutaneous leishmaniasis and provide increased wound contraction rate, re-epithelialization rate, and formation of scar tissue [32]. The nanosystem based on CS-sodium tripolyphosphate (STPP) also acts as a potential candidate for the delivery of active agents for several food-grade properties.

4.5 Fabrication Routes of Chitosan Nanostructured Materials

In 1994, Ohya and coworkers first described CSNPs preparation using emulsifying and cross-linking process. Besides, the various methods for developing CSNPs as shown in Fig. 4.4 include ionotropic gelation, emulsion cross-linking, emulsion-droplet coalescence, precipitation, reverse micellar method, sieving method, spray drying method, and others. The CSNPs formation can be obtained using chemical or physical cross-linking process. The acid hydrolysis is another approach for obtaining chitin nanocrystals and nanofibers with controlled dimensions. The CSNPs are also used extensively in the agricultural sector as pesticide delivery for crop production, fertilizer delivery, micronutrient delivery, nanosensors, etc. [33]. Additionally, NCS is a potential candidate in food sectors and is used in edible films, coatings, encapsulation, carrier materials, antimicrobial active films, dietary supplements, and others.

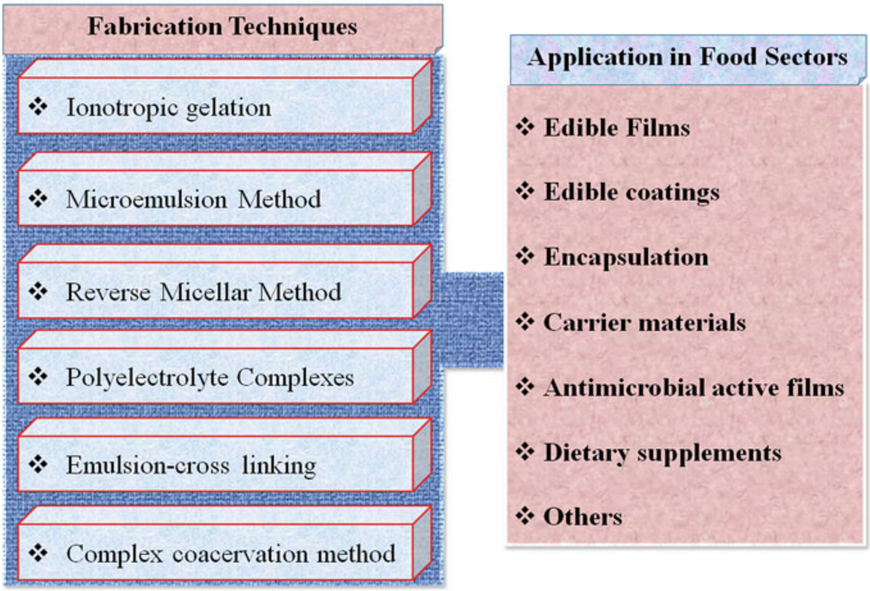

Fig. 4.4 Fabrication techniques of nanochitosan and its application in food sectors

4.5.1 Ionotropic Gelation

The ionotropic gelation is a simple and mild process, which includes no use of chemical cross-linking and also reduces the possible toxicity of chemicals. CS (polycation) having an amino group possesses positive charge, whereas STPP (polyanion) has negative charges, so the component can be self-assembled through the ionic gelation method. In this method, the CSNP extraction is developed through interaction between the oppositely charged molecules as represented in Fig. 4.5. The ionic interactions between the biomacromolecules can be monitored by charge densities of CS and STPP. The STPP has the ability to form gels through ionic interactions. The CSNPs ionically cross-linked with tripolyphosphate (TPP) have the properties of haemocompatibility, non-toxicity, mucoadhesivity, antimicrobial activity which also provide the benefits of incorporating macromolecules, hydrophilicity, etc [34]. For the fabrication of CSNPs via ionotropic gelation, CS is first dissolved in acetic acid solution, and before or after the addition of polyanion, stabilizing agent such as poloxamer can be added in the CS solution. In the next step, STPP as a polyanion is added to the above solution under mechanical stirring at room temperature and formation of CSNPs held. In this process, the processing parameter such as weight ratio of CS and TPP is a crucial parameter to control and obtain higher yield to fabricate CSNPs. In this process, the dimension of fabricated CSNPs can be modified via tailoring the ratio of CS and STPP. The several factors which affect the fabrication of CSNPs are concentration, pH, ratio of components, mixing method, etc. The particle size of CSNPs is 85 nm

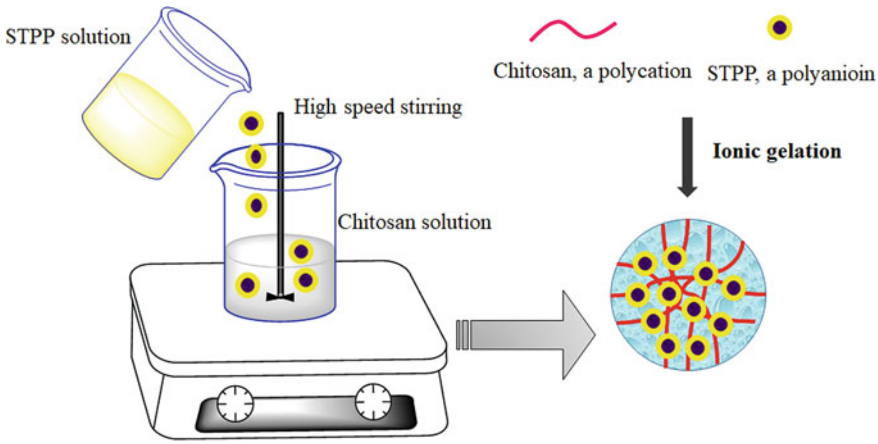

Fig. 4.5 Fabrication of CSNPs through ionotropic gelation

(CS concentration: 2.14 mg mL^{-1}, TPP concentration: 0.3 mg mL^{-1}), 110 nm (CS concentration: 2.14 mg mL^{-1}, TPP concentration: 0.6 mg mL^{-1}), and 221 nm (CS: 3.15 mg $mL^{-1}$and TPP concentration; 0.6 mg mL^{-1}) [35]. The STPP is used in food product development and has the characteristics attributes for acting as a carrier material for the delivery of bioactive components. The biomacromolecules are used as a preservative and food additives for meats, poultry, seafood, etc., where STPP is used to keep the freshness of the products by maintaining the natural color and improving the texture. The mentioned properties are attained by improving the water holding properties of the products. Further, it is used as emulsifier in food products. Additionally, the particle size, polydispersity index, and other properties of CSNPs can be modified by controlling the molecular weight, concentration, mass ratio, pH of CS solution [36].

4.5.2 Microemulsion Method or Covalent Cross-Linking Method

Microemulsion method of developing CSNP is also known as the covalent cross-linking method, where there is a formation of covalent cross-linking between CS molecule and a functional cross-linking agent such as glutaraldehyde, epichlorohydrin, polyethylene glycol, dicarboxylic acid, and others. In this microemulsion method of developing CSNP involves several steps such as (i) firstly, surfactant dissolves in N-hexane and glutaraldehyde is added to the mixture at room temperature and stirred continuously, and on the other hand, CS solution in acetic acid is prepared. The above mixture is stirred continuously, where glutaraldehyde or others acts as a cross-linker as mentioned earlier and a cross-linking between glutaraldehyde and free amino group of CS occurs. After this, the used organic solvents from the systems are removed via evaporation technique. The obtained NP with excess surfactants is treated with calcium chloride to remove the surfactants as precipitant, which is separated using centrifugation. After this, the obtained CSNPs are undergone dialysis followed by freeze drying. The specific process has the advantage of developing NPs with narrow size distribution. However, the several disadvantages associated with this method are long processing time and the use of organic solvents.

4.5.3 Reverse Micellar Method

The fabrication of CSNPs using the reverse micellar process provides thermodynamically stable particle size, with suitable polydispersity index, and particles with narrow size distribution and smaller particle size. However, the process is laborious and tedious. The fabrication of CSNP does not include the use of cross-linking agents and toxic organic solvents. The reverse micelles of CSNPs are formed by adding an aqueous solution of CS to a solution of surfactants under constant agitations. The use of this method can provide CSNP of narrow size ranges with ultrafine polymeric NPs. The lower molar mass of CS provides better control for obtaining targeted particle size and size distribution.

4.5.4 Polyelectrolyte Complexes

The polyelectrolyte method of developing CSNP occurs due to the interactions between anion and cation through electrostatic interactions. At low pH, the CS has positively charged groups and thus can associate with polyanions to form polyelectrolyte complexes. In this method, the resulted polyelectrolyte complex gets self-assembled due to the charge neutralization between cation (CS) and selected anion. The CS-based polyelectrolyte complexes are used for the delivery of bioactive components. The several factors such as molecular weight, pH, and concentration affect the dimension of developed CSNPs. The process does not involve the use of catalysts or initiators, and further, the reaction occurs in aqueous solution only.

4.5.5 Emulsion Cross-Linking

This method is used to develop both CS microparticle and NP. The method includes the fabrication of water-in-oil emulsion via emulsifying CS solution in a continuous phase of oil. The aqueous droplets of developed water-in-oil emulsion are stabilized using a preferable surfactant, and the developed emulsion is further treated with an appropriate cross-linking agent to stabilize the CS droplets followed by washing and drying. In this technique of developing NPs, the size can be controlled by varying the size of aqueous droplets, speed of stirring, amount of cross-linking agents, etc.

4.5.6 Other Methods

The other methods for developing CSNPs include complex coacervation method, incorporation and incubation techniques, acid hydrolysis, and others. The complex coacervation method of developing CSNPs involves the development of coacervates between CS (a cation) and anions (biomacromolecules). The incorporation and incubation technique of developing CSNP is used to deliver protein molecules. Further, nanocrystalline CS is a modified form of CS, which is thermally stable and is more potential in developing biomaterials for various applications.

4.6 Packaging Properties of Nanochitosan-Based Edible Food Packaging

CS nanostructured materials are extensively used to improve the packaging property of several polysaccharides such as hydroxypropyl methylcellulose (HPMC) and protein-based films in terms of barrier property, mechanical property, thermal property, color property, and others. Furthermore, the CSNPs having the significant properties of antimicrobial, antioxidant, antifungal, and cytotoxic activities are used to wrap several food products. The fabrication of CS-based edible films with tailored packaging properties in terms of functionality and performance can help to improve the existing shortcomings of individuals. The utilization of CS nanostructured-based materials for edible food packaging of food products in terms of edible films and coating can help to avail with fresh, seasonal food products, in several regions. The CS nanostructured materials in combination with other polymeric materials as polymer biocomposites are used to provide the improved shelf life of several categories of food products such as poultry products, seafood, and agricultural products. The emulsion-droplet coalescence method of developing CSNP involves both emulsions cross-linking and precipitation methods. Further, the several NCS-based edible films are represented in Table 4.1.

In this regard, the edible films based on HPMC and CS/TPP NP provide improved barrier (water vapor permeability), mechanical properties (tensile properties), and thermal properties [35]. The use of CSNPs in developing HPMC-based biocomposite films has a tendency to occupy the empty spaces of the pores of HPMC matrix, which helps to improve the packaging properties. The CSNPs with a particle size of 85, 110, and 221 nm provide elastic modulus of $\sim$1264, $\sim$1190,

Table 4.1 Packaging properties of CSNPs-based edible films

Sl. No.	Film composition	Type of packaging materials	Packaging property	References
1.	CSNP and HPMC	Polysaccharide-based edible or biodegradable films	Improve mechanical property, barrier property, water solubility, thermal stability, and others	[35]
2.	CSNP and tara gum	Polysaccharide-based edible films	Improved tensile strength, thermal stability, antimicrobial effects, and others	[36]
3.	CSNP, zein film, and CEO	Protein-based active edible films	Provide improved tensile strength, decreased elongation, decreased water vapor permeability, tailored solubility, and others.	[39]
4.	CSNPs and Fish gelatin	Edible films	increased superficial hydrophobicity, thermal stability, and crystallinity	[37]
5.	CSNP, HPMC, guava puree	Edible film	Increased mechanical and thermal property Decreased WVP and water solubility	[40]
6.	Fish gelatin, CSNP, *Origanum vulgare* L. EO	Edible films	Increased crystallinity, flexible films, decreased water vapor permeability Antibacterial film	[38]
7.	CSNPs, banana puree films, glycerol	Edible films	CSNPs improves the mechanical property And reduces the mechanical property	[41]
8.	NCS and pectin	Active edible films	Increased tensile strength, reduced water solubility, hydrophobic films	[5]

CSNPs Chitosan nanoparticles, *HPMC* Hydroxypropyl methylcellulose, *CEO* Cinnamon essential oil, *EOs* Essential oils, *NCS* Nanochitosan, *WVP* Water vapor permeability

~1204 MPa, respectively. Further, the elongations of HMPC/CSNP-based edible films are ~11.1, ~5.2, ~5.7% for particle size of 85, 110 and 221 nm, respectively. The CSNP loaded HPMC films improve the thermal stability of the films and the thermal degradation temperature changed from 232 to 271 °C due to NP loading. Additionally, the CSNPs incorporated tara gum-based edible films improve the mechanical property and physicochemical property [36]. The use of CSNPs helps to reduce the film hydrophilicity and water solubility which is a desirable property for food packaging application. As discussed earlier, the free volume of HPMC edible films can be reduced when CSNPs are used as nanofiller materials due to the compact structure and further, create an obstruction in the path of water diffusion decreasing the moisture content of the films. The tensile strength of CSNP (10% w/w) incorporated tara gum increased to 58 MPa from 23 MPa (neat tara gum films). The incorporation of CSNP in HPMC films helps to increase the tensile strength by 35.73 MPa and decrease the elongation by 7.21%. Additionally, the water solubility and water vapor permeability are reduced by 74.3, and 22.7% for CSNP reinforced HPMC films, respectively. The development of CSNP and fish gelatin-based edible film provide increased superficial hydrophobicity, thermal stability, and crystallinity (due to nucleating effect of nanofiller) in comparison with neat fish gelatin films [37]. Additionally, the addition of *Origanum vulgare* L. EOs develop more flexible films with increased crystallinity and further reduce the water vapor permeability [38].

The edible films based on biocomposites of zein film with CSNP, and cinnamon EO (CEO) provide significant tunable packaging properties in terms of mechanical property, physical attributes, structural and antimicrobial attributes [39]. The zein-based biocomposite films containing CSNPs (2% w/w) and CEOs (4% w/w) increase 112% tensile strength and 45% decrease in elongation at break in comparison with neat zein films. The water vapor permeability of neat zein film and zein biocomposite films (with CSNPs and CEOs) are ~6.4 and ~2.0 g Pa^{-1} h^{-1} m^{-1}, respectively. Additionally, the color parameters of neat zein film and zein biocomposite films are ~36.30 and ~27.05 in terms of *L** value; ~6.28 and ~14.22 in terms of *a** values; ~13.96 and ~19.00 in terms of *b** values, respectively. Interestingly, the zein biocomposite films with CSNP and CEOs have an inhibitory area of ~11.33 and ~27.33, respectively, for *Escherichia coli* and *Staphylococcus aureus*. The edible films based on guava puree, HPMC, CSNP deliver a sustainable packaging material with improved thermal, mechanical, property, whereas the water vapor permeability and water solubility also reduce due to added CSNPs [40]. The edible films based on banana puree extracted from over-ripe peeled bananas, glycerol, pectin, and CSNPs reduce the water vapor permeation by 21% [41]. The active edible films based on NCS and pectin can improve the shelf life of food products which inhibit the growth of *Aspergillus niger*, *Escherichia coli*, *Saccharomyces cerevisiae*, and others [5].

4.7 Application of Chitosan Nanostructured Materials in Edible Food Packaging

The use of CS nanostructured material in developing edible food packaging has attained a great interest due to several remarkable properties including antimicrobial, antibacterial, and cytocompatible nature. The CS nanomaterial-based edible coating also helps to decrease the weight loss of fruits and vegetables, while delaying the ripening of perishable fruit products. In this regard, several research and development are continuously undergoing for its wide consumer acceptance and commercialization purpose. Additionally, CS as an edible nanomaterial added coatings provides significant advantages in order to maintain quality, safety, and enhanced shelf life of highly perishable food products. The edible-coating-based technology has been considered as one of the widely used food preservation techniques, which minimizes the postharvest loss of fresh produces. In this context, this section specifically discusses the application of edible coating incorporated with nanostructured CS on the storage life of food products and their significant properties.

4.7.1 Effect of Nanostructured Chitosan-Based Edible Coating on Fruits and Vegetables

The NCS-based edible coatings are extensively developed on several fruit products such as tomato, strawberry, apples, and cucumber as the coating minimizes the respiration rate and reduces the water loss from fruit products. The fruit products have a short shelf life due to the perishable nature and have a high degradation rate due to the microbial attack. NCS also acts as a protective barrier to fruit products against bacterial contamination for its biocidal activity. Among available fruit products, tomato is a widely consumed fruit throughout the world. However, after harvesting, the storage of tomato for a long period is difficult due to the microbial attack, and further, the quality of tomato gets degraded by the external agents during storage, which creates several problems in transportation such as mechanical damages. In this regard, the work reported by Mustafa et al. [11] studies the keeping quality of tomatoes (*Solanum lycoperiscum* L.) after harvesting, where a reduced ripening rate can be obtained after formulating an edible coating on green tomatoes with CSNPs. Additionally, the fabrication of CSNPs with varying droplet sizes of 800, 600, and 400 nm can be obtained using the ultrasonication treatments. The formulation of edible-coating material using 1% CS reinforced with different size ranges of CSNPs provides tunable properties, where the coated and control (uncoated) tomatoes are kept under refrigerated condition (15 ± 2 °C) with 70–80% relative humidity (RH) for a storage period of 20 days. The specified coating helps in maintaining firmness, chlorophyll content, and soluble solid content in the stored edible-coated tomatoes, which further delays the ripening rate. Further, in coated fruit, the initiation of ripening has been delayed by 5 days and it is observed that the weight loss in coated fruits is found to be reduced in comparison with the uncoated tomatoes with maintained quality parameters.

Eshghi et al. [12] have studied the development of edible coating using NCS with and without copper loading for an extended shelf life with maintained physicochemical properties and bioactive components of fresh strawberry fruits (*Fragaria x ananassa* Duchesne) under refrigerated storage condition (4 ± 1 °C) for 20 days with 70% RH. However, the copper loaded edible coating has no significant effect on strawberry and further reduces the bioactive molecules such as anthocyanin in the fruit sample such as strawberry. In this regard, the NCS-based coating shows better improvement in retaining firmness, reduced ripening rate, and also maintained the weight of fruits during the storage life. Additionally, the antioxidant activity can also be improved in coated strawberry fruits in comparison with the control fruit. However, anthocyanin concentration is observed to be increased for the first 12 days of storage, whereas, in later days, the concentration decreases at a slower rate, and further, there is a minimum loss of ascorbic acid in coated strawberry fruit. Further, both the coating materials such as NCS with and without copper loading have antibacterial and antifungal effects as well as inhibitory effect on the polyphenol oxidase (PPO) and peroxidase (POD) activity, which possess browning effect. Moreover, the sensory appeal toward consumers can also be maintained by preserving flavor, color, and overall appearance through the coating.

Additionally, apple is another nutritious and healthy fruit, but, apple slices have several problems such as browning due to enzymes, spoilage due to microbial attack, and microbial attack may cause several diseases such as black rot, bitter rot, and white rot. Thus, the microbial growth in fresh-cut "Gala" apples can be reduced without altering the quality attributes using CSNPs [42]. The apple slices with maintained quality can be obtained when coated with CSNPs by spray coating technique with different size of NPs (110 and 300 nm) stored under refrigerated condition (5 °C) for 10 days. Besides, the edible-coated fresh-cut apple developed by dipping into the neat CS solution and uncoated cut apple has comparable quality attributes. The browning in apple slices is less, when CSNP is used as a coating material in comparison with CS coated and uncoated sample. Further, significant antimicrobial effect can be obtained in CSNP-based coating, where edible coating with 110 nm dimension provide better effectiveness against mesophilic and psychrotrophic bacteria, as well as reduces mold, and yeast counts and further, the growth of pathogenic microorganisms (*Salmonella* and *coliforms*) also inhibits. This investigation suggests a potential use of edible coating of CSNP in citric acid solvent for maintaining the microbial quality of fresh-cut apple slice. Additionally, apple cv. *Golab Kohanz* is native cultivars with a unique flavor and aroma, but this apple variety is highly perishable and does not have the ability to maintain quality during transportation as compared to other apple variety. For instance, the shelf life of this native apple cultivar can be improved via NCS-based edible coating. In this regard, the development of edible coating on apple cv. *Golab Kohanz* based on NCS emulsion with varying concentrations of CS (0.2 and 0.5%) can maintain the quality (storage conditions: 1 ± 1 °C with 85–90% RH for 9 weeks) [13]. The application of mentioned NCS and CS coating significantly improve the quality and shelf life of stored apple and also a potential candidate in minimizing the postharvest loss. Interestingly, comparing with uncoated fruit sample, a significant reduction in apple respiration rate (CO_2 generation rate), production of ethylene, POD and PPO (enzyme) activity can be obtained in coated sample. However, the weight loss of apple is also reduced with NCS- and CS-based coating during the storage period. Further, the coating is beneficial in maintaining the tartness (slowing down softening process) and color quality of apple after the climacteric period. It is worth mentioning that the formulation of nanocoating with 0.5% CS and NCS emulsion is better in preserving quality and reducing weight loss as compared to the other coating materials. Thus, NCS coating is very effective in reducing fruit ripening and improving the storage life of perishable apple.

The application of NCS-based edible coating in improving the shelf life of whole cucumber can be attained using nanoemulsions of NCS and EO [43]. Moreover, *Zataria multiflora* EO (ZEO) can be added to the CSNP in order to improve the shelf life and antioxidant activity of whole cucumber. In this study, the CSNP has been prepared via ionic gelation method and the developed nanoemulsion is an oil-in-water type emulsion. The edible-coated cucumber samples can be stored (storage condition: refrigerated storage (10 ± 1 °C) with 90–95% RH) for a period of 21 days, where the decaying rate is higher in uncoated cucumber (97.7%) in comparison with coated cucumber (coating materials: NCS (12%) and ZEO incorporated CSNP (2%)). The lower aerobic microbial count is found in coated cucumber as compared to uncoated fruit after 18 days of storage. Further, the addition of NCS and ZEO provides improved physiochemical properties of edible-coated cucumber such as increased firmness, DPPH radical scavenging activity, and reduced respiration rate of cucumber. However, ZEO incorporated coating deliver better property retaining of cucumber in terms of physiochemical as well as microbial quality, antioxidant activity, and further, improve the shelf life of cucumber. Based on the result, the added EO enhances the efficiency of CSNP to act as an edible-coating material. Therefore, the use of edible active compounds (EO) in order to improve the effectiveness of CSNP, deliver more significant characteristic properties to the food products via edible coating.

Additionally, the shelf life and quality of banana fruit (*Musaacuminate* AAA group) can be enhanced via developing an edible coating with the aid of CS and CSNPs [2]. In this regard, an investigation suggests the use of three different edible-coating solutions such as CS (1.15 and 1.25%), and CSNP (fabrication method: ionic gelation). The investigation reports that the banana fruits are coated via dip-coating technique and stored at ambient temperature (25 ± 1 °C) for 15 days. Further, the uncoated banana becomes unacceptable for eating due to high level of softness and initiation of decaying nature after 8 days of storage, whereas the coated banana samples have reduced decaying rate and are eatable till 11 days. However, the shelf life of banana coated with CSNP is less as compared to CS (1.15 and 1.25%) coated sample and the probable reason is due to the thickness of coating, where the banana fruits are completely covered by CS coating, which can be confirmed via scanning electron microscopy (SEM). Further, the uncoated banana has more starch to sugar conversion rate, weight loss, increased pulp to peel ratio in comparison with coated sample. However, the CS, NCS coated and uncoated banana have no effectiveness in terms of sensory evaluation and overall acceptability. The CS (1.25%) coating is also beneficial for reducing the ripening rate of banana in comparison with uncoated sample. This study indicates the positive effect of CSNP- and CS-based edible coating, which can provide improved shelf life and quality of postharvest fruits for long storage.

Luo et al. [44] investigated the coating effect of CS and NCS composites on fresh-cut medicinal plant *Zizania Latifolia*, which is enriched with high protein, amino acids, carbohydrates, and ascorbic acid and considers as a nutritious healthy vegetable in East Asia. The development of edible coating on freshly cut pieces of *Zizania Latifolia* can be obtained by dipping into the coating solution and the dipped coated cut leafy vegetables are kept inside the polypropylene (PP) plastic bag (after draining the excess coating solution) under refrigerated condition (1 °C) for 12 days. The accumulation of lignin and changes in color is rapid in uncoated samples in comparison with the CS and CS/NCS coated samples during the storage period. Further, the activity of PPO and POD can be reduced via developing coated samples, thereby inhibiting the enzymatic browning. Both the coating material with and without NCS can provide improved shelf life of coated vegetables by retarding lignification and browning during the storage period. Based on the result, it can be concluded that coating has a direct inhibitory effect on enzymatic activity and indirect effect on lipid peroxidation.

Another study by Divya et al. [1] reports the coating effectivity of CSNP (fabrication method: ionic gelation) on vegetables. The edible-coating solution with varying concentration of CSNP (1, 2, 3, 4, and 5%) and CS can be used to coat (using dipping method) vegetables such as chilly, brinjal, and tomato (Storage conditions: Ambient temperature for 5 days). The weight loss of selected vegetables is significantly low when coated with CSNPs (4 and 5% concentration) in comparison with CS and uncoated sample. However, CS coated fruits and vegetables are considered effective in reducing the weight loss than uncoated sample. Further, the radical scavenging activity of CSNP is promising (can be found by DPPH test); however, the activity is lower than gallic acid (control). The scavenging activity is due to the presence of nitrogen in the second carbon atom of CS. Similarly, the observed significant result of the total reducing power and superoxide radical scavenging activity of CSNPs is low in comparison with gallic acid as it depends on the amount of doses. The antifungal activity of CSNP against fungi and pathogens (*R. solani, C. acutatum, P. infestans, F. oxysporum*) can be determined by the minimum inhibitory concentration (MIC). The MIC value of CSNPs is higher as compared to Amphotericin B (control), which indicates the higher antifungal activity. Moreover, the smaller dimension of CSNP has a significant effect on absorption and disruption of fungal cells. The cytotoxicity study can be conducted for CSNP using fibroblast cells. The resulting decreasing trend in cytotoxicity confirmed the non-toxic nature of CSNPs and further indicates that the affectivity is dose-dependent. This study also proves the improved activity of CS due to its nanoform, and further, CSNP is applicable as an edible-coating agent for extending the shelf life of vegetables due to mentioned characteristic properties such as antifungal, antioxidant, and non-toxicity.

4.7.2 Effect of Nanostructured Chitosan-Based Edible Coating on Fish Products

Fish is another highly perishable product which can also be protected from spoilage using CS- and NCS-based edible coating. In this regard, Abdou et al. [3] have reported the microbiological characteristics of fish fingers after coating with CS and CSNPs (fabrication method: ionic gelation using TPP, where CS and TPP concentrations are taken in the ratio of 1:1). The study investigates the characteristic behavior of edible CS and CSNP coated fish fingers with uncoated (T_1) and commercially available edible-coating material (T_2). Further, CS coating with various concentrations (2, 2.8, and 4%) are used to coat fish fingers for obtaining the best output (T_3, T_4, T_5). Similarly, CSNP is varied in concentration such as 2, 2.8, and 4%, are represented as T_6, T_7, $T_{8,}$ respectively. The fish finger has been prepared from carp fish. All the samples have been stored under frozen condition (−18 °C) for 6 months. The average particle size of developed CSNP is 10 nm. Further, the rheological characteristic of the mentioned edible-coating solution follows non-Newtonian pseudoplastic behavior. The TBC of all coated fish fingers and uncoated sample is found in the range of 3.66–4.72 log cfu/g. The TBC of T_1 is lower than T_2 count, because of commercial coating material. Moreover, the samples coated with different concentrations of CSNPs (T_6, T_7, T_8) show lesser TBC when comparing with different concentrations of CS coating (T_3, T_4, T_5) followed by uncoated and commercially coated samples. The obtained results indicate higher antimicrobial activity of CSNPs, and the probable reason can be decreased particle size. The observation further reveals increasing the concentration of CS and CSNPs, which reduce the TBC value during the storage time of two months for T_1 and T_2, four months for T_3 and T_6 and five months for rest of the samples (T_4, T_5, T_7, and T_8). Additionally, a slight increment in TBC has been found after five months of frozen storage till the end of the storage period. In addition, the formation of intra and extra cellular ice crystals aids irreversible injury to the cytoplasmic membrane of bacteria thereby reducing TBC. However, the occurrence of increased count can be due to the hydrolysis of fat and protein by fish enzymes which provide favorable condition for bacterial growth and the byproducts forms are fatty acids, amino acids, and nucleotides. Moreover, during the frozen storage, the lowest TBC is found in T_8 (2.87 log cfu/g) followed by T_7, T_5, T_6, T_4 and T_3 (3.71 log cfu/g), respectively. Similar observation has been observed in psychrophilic bacteria count, where CSNP shows better activity in lowering the psychrophiles. The psychrophilic count is higher in T_1 followed by T_2, T_3, T_4, T_5, T_6, T_7, and T_8 during the complete storage period. The bacterial counts further reduced till three months in T_1, T_2, T_3, T_4 and T_5, four months in T_6 and T_7 samples and five months for T_8 fish finger sample and further, there has been noticed a slight increment till the end of the storage life. In this context, similar trend is observed for *coliform* and proteolytic bacteria count, where CSNP shows better reduction as compared to CS coated and others. Nevertheless, throughout the microbial study CS coated samples have shown better reduction than commercial coated and uncoated sample. The T_8 sample shows best results among others based on the NP concentration. However, no *salmonella*, *staphylococcus aureus,* yeast, and molds counts are observed in all coated and uncoated samples during the complete storage period. Moreover, this study indicates effective application of CSNP-based edible coating in order to minimize microbial count for extending the shelf life of the fish products during storage.

The NCS-based edible coating provides improved shelf life and quality in comparison with the CS for refrigerated silver carp fillets [15]. The obtained zeta potential value of CSNP is +49.50 mV, which indicates the stability of nanoemulsion. In this study, the comparison of samples can be done between 2% CS and 2% NCS along with uncoated control (fish fillet treated in distilled water) and glacial acetic acid treated control. The development of edible coating on fish fillets can be obtained via dipping the fish fillets into the edible-coating solution, which is further stored under refrigerated condition (4 °C) for 12 days. The initial value of total mesophilic count (TMC) and total psychrotrophic count (TPC) of fish fillet are 3.36–3.61 log cfu/g and 3.27–3.56 log cfu/g, respectively, at zero time of storage. Moreover, the samples treated with CS and CSNP are found to have less TMC and TPC in comparison with uncoated control and acid treated control. Further, no significant difference is found in CS and CSNPcoated sample till 9 days of storage, however, on the 12th day of storage the TMC and TPC is significantly lesser in NP coated sample than CS coated samples. This result indicates higher antimicrobial activity of CSNPs in comparison with CS. Moreover, the CS and NCS coated samples have not crossed the permissible limit of 7.0 log cfu/g during the complete storage period, while the controlled values exceeds the permissible limit on 9th day of storage. Further, the evaluation of physiochemical properties of entire samples is done through analysis of TVB-N, Thiobarbituric acid reactive substances (TBARS), changes in pH, and sensory evaluation. The TVB-N value indicates the spoilage of samples which is occurred due to the formation of trimethylamine, dimethylamine, and ammonia from degraded protein and non-protein nitrogenous compound via microbial and enzymatic activity of spoilage bacteria and endogenous enzyme. During the storage period, the TVB-N value increases; however, the rate of increment is slower in CS and CSNP coated sample as compared to control. Moreover, significant differences of TVB-N values are observed among CS and CSNP on the storage days of 9th and 12th. The CSNP coated sample has TVB-N value of 24.6 and 30.8 mg *N*/100 g of fish on 9th and 12 days, respectively, and the values of 39.3 and 44.4 mg *N*/100 g are obtained for CS coated sample for 9th and 12 days of frozen storage, respectively. The outcome indicates higher antimicrobial activity of NCS. Similar findings are observed for TBARS analysis which provides the TBA value countable for degree of lipid oxidation and the auto-oxidation (occurrence of TBARS substances) during which ketones and aldehydes are formed due to the oxidation of peroxides. The TBA values followed increasing trend throughout the storage period for coated and uncoated sample; however, slower rate is observed in CS- and CSNP-based coated samples. Further, no significant differences are found between the CS- and CSNP-coated samples. The initial reduction and then increment in pH value is observed in entire sample during the storage period. No significant changes are observed till 6th day of storage, whereas, on 12th day, uncoated sample has shown higher value as compared to others due to occurrence of volatile basics amines in the muscle tissue. The sensory attributes of entire sample till the 6th day is acceptable for treated and untreated sample, whereas only CS- and NCS-coated samples are acceptable till the end of refrigerated storage. However, the study indicates higher impact of CSNP on the edible-coated fish fillet for maintaining the quality and improving the storage life.

Tapilatu et al. [45] have reported the effect of NCS for the preservation of fresh yellowfin tuna (YFT). NCS can be utilized as a coating material for extending the shelf life of freshly caught wild YFT. The edible coating on YFT can be developed via immersing YFT in 1% NCS solution for 30 min followed by drying at ambient temperature for another 5 min. After drying, the samples are wrapped with plastic and stored at two different temperatures (4 and 28 °C) for 24 h. The TVB-N value is found very less in the NCS-coated sample stored at 4 °C as compared to higher temperature storage and uncoated sample. Similarly, the microbial count is found to be lower for the coated samples stored at 4 °C. This study shows better effectiveness of NCS coating for extending the shelf life at 4 °C storage temperature.

4.7.3 *Edible Nanocomposite Films Based on Nanostructured Chitosan*

The nanostructured CS has been utilized as a reinforcing agent or nanofiller material in order to develop nanocomposites with tailor-made properties. The several approaches to develop NCS-based biocomposites are represented in Fig. 4.6. The

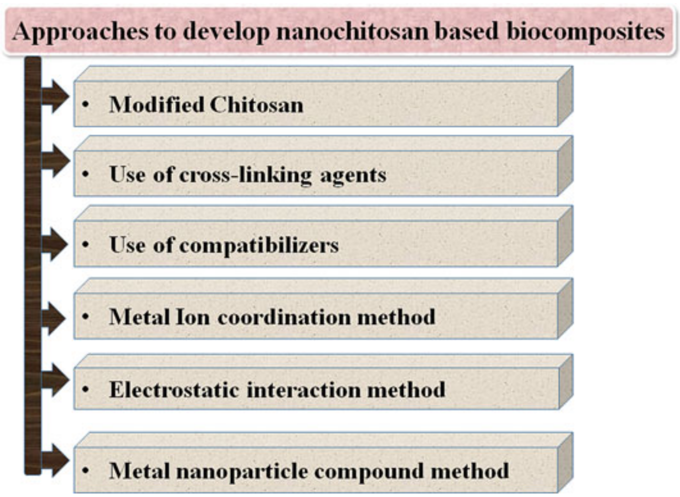

Fig. 4.6 Several approaches to develop nanochitosan-aided biocomposites

utilization of CSNPs (developed via ionotropic gelation method) as a reinforcing agent in developing fish gelatin-based edible bio-nanocomposite film, deliver improved packaging property [46]. The CSNPs have spherical morphology with the size ranges of 40–80 nm and a stable zeta potential value of +10 mV. The edible films can be developed for optimized properties with varying the concentration of CSNPs. The higher concentration of CSNPs (8%, w/w) may have aggregation problems. The fish gelatin and CSNPs-based films have increased crystallinity in comparison with fish gelatin film due to the nucleating effect of CSNPs in the nanobiocomposites. Moreover, the nanofiller is also beneficial in providing the improved mechanical stability to the nanobiocomposite films in terms of increased tensile strength and elastic modulus. Besides, CSNPs contribute to the remarkable barrier properties by reducing the water vapor permeability around 50% due to the addition of 6% (w/w) nanofiller. Furthermore, the gelatin biocomposite edible films are transparent in nature with an excellent barrier against ultraviolet (UV) light. Thus, gelatin and NCS-based edible films can be used as an edible nanobiocomposite film for food packaging. However, these films are not a good choice for high moisture food as the film solubility and the water vapor permeability are higher as compared to the commercial non-edible films such as low density polyethylene, polyvinylidene chloride, and others.

The antimicrobial and physicochemical properties of chitin nanowhiskers (CNWs) incorporated maize starch-based edible nanocomposite films can also provide improved starch properties [47]. The CNWs have needle-like morphology with the length of 100–400 nm and a diameter of 10–50 nm. However, the distribution of CNWs in the starch matrix can be observed via morphological analysis such as SEM and transmission electron microscopy. The agglomeration effect of nanofillers in CNW-based biocomposite can be visible with increased CNWs concentration. Further, a stable suspension can also be obtained using CNW as it has colloidal behavior due to the presence of positive charges of ammonium ion on the surface of crystallites. The edible nanobiocomposite films offer improved relative crystallinity via increased CNWs concentration in comparison with the maize starch film (control). Besides, the CNWs reinforced film exhibits improved mechanical properties, where the tensile strength increases from 1.64 to 3.69 MPa (0–1%), on the other hand, the elongation at break decreases with increasing CNWs concentration. A better interfacial interaction between the filler and polymer matrix can help to keep similar thickness of the films. The another important parameter of packaging materials is water vapor permeability, decreases from 5.32×10^{-12} to 2.22×10^{-12} $gm^{-1}s^{-1}Pa^{-1}$ with the increased CNWs loading from 0 to 2% as compared to control, indicating good barrier properties of the film. The swelling degree has shown decreasing order with increasing the CNWs concentration (0–5%) from 103.11 to 82.15%, indicating lower water uptake of films (water registrant property) due to the strong hydrogen bonding among CNWs and CNWs/maize starch. Further, the nanocomposite films of CNWs and starch have improved opacity and transparent behavior. Besides, these films have improved thermal properties and degradation behavior. Most importantly, the nanocomposite films have stronger antimicrobial activity against *L. monocytogenes* and *E. coli,* when

CNWs are added to starch films. Thus, the utilization of chitin NPs in food packaging application can aid better properties in terms of improving the packaging characteristics. Besides, chitin also acts as a good edible reinforcing agent in developing good food packaging films.

In a recent study, the reinforcement effect of CSNPs in developing biodegradable zein film composites along with CEO has been reported [39]. The edible nanocomposites of zein, CEO (2% w/w) and CSNPs (4% w/w), fabricated via solvent casting approach has improved packaging attributes in terms of water vapor permeability, mechanical property, and antimicrobial effect. The zein-based biocomposite incorporating CEO and CSNPs deliver reduced water vapor permeability by ~41%. In case of mechanical properties, the addition of CSNPs increase the tensile strength and decrease the elongation at break, whereas both tensile and elongation can be increased in zein-based films by the addition of CEO. Further, a ~45% decrease in elongation at break and 112% increase in tensile strength can be obtained by the incorporation of CSNPs along with CEO in zein film. Besides, the nanocomposite films incorporated with both CSNPs and CEO have shown significant antimicrobial activity against *E. coli* and *S. aureus,* whereas CSNPs incorporated zein film has no significant effect. However, the addition of CEO is beneficial for providing improved antimicrobial activity in nanocomposites films. Therefore, the addition of CEO along with CSNPs helps to provide synergistic effects and improved film properties for food packaging applications.

4.7.4 Applications of Nanochitosan in Food Sector and Other Areas

Nanostructured CS has an immense impact in developing edible food packaging for several food products, which has been discussed in the earlier section. Besides, NCS can also be useful in several non-food areas for protecting the product life and environment from adverse effects. In this regard, some of the versatile applications of NCS has been mentioned in the following section. CSNPs are used in food packaging as active, edible films, antimicrobial, intelligent, and improved biodegradable packaging. Further, CS and NCS are used in food products to develop functional food products to improve the organoleptic property, nutraceutical enrichments, etc. CS and NCS have wide applications in several areas such as biomedical, water treatment, cosmetics, agricultural, etc.

The presence of heavy metal pollutants such as copper, lead, zinc, cadmium, chromium, and nickel in water is one of the reasons for causing water pollution, which is also considered as the cause of environment hazards [48]. Zinc is a common pollutant in wastewater obtained from various industries as zinc is utilized in many industries for manufacturing automobiles, batteries, medicine, casting, dyeing, production of fungicide, and others. In addition, zinc is a trace element which is required by the human body enzyme for completing the metabolic pathways with a very small quantity. Therefore, excess consumption of zinc can cause some adverse effects such as genetic mutation, fatal deficiencies, skin irritation, and digestive problem. Nevertheless, the deficiency of zinc is responsible for weakness during growth, reduction in sensitivity of taste, and adverse effect on generation system. Thus, it is essential to eliminate zinc as it is a hazardous pollutant available in water resource. The various methods are available for removal of heavy metals from wastewater including ultrafiltration, reverse osmosis, sedimentation, electrodialysis, activated carbon, adsorption, and others. However, adsorption is the simplest and widely used high efficient method for water and wastewater with the use of different adsorbents such as clay, activated carbon, bentonite, algae, bacteria, and others. Interestingly, in recent days' biopolymers are selected as adsorbents due to low toxicity, environment-friendly, and cost-effective nature. In this context, CS and its derivatives have been used by the researchers as one of the potential adsorbents for metal ions such as arsenic, gold, copper, nickel, lead, and cadmium. The studies predict that the presence of amine and hydroxyl groups can have an influence on the removal of heavy metals. In this regard, Seyedmohammadi et al. [49] reported the effectiveness of NCS and CS particles (microsize) for protecting the environment by the adsorption of pollutant zinc (Zn(II)) from aqueous solution. CS, a cationic hydrophilic polymer, is derived from chitin via acetylation, where CS can react with metal ions via adsorption phenomena. NCS (mean diameter 19.84 nm) has the maximum significances with Zn(II) adsorption capacity of 370.37 mg/g in comparison with the macrosize CS particles (196.07 mg/g), where the better effectivity of CS is obtained with nanosize CS. The highest removal of zinc ions can be obtained at pH 7 and temperature of 25 °C.

The anticorrosion properties of NCS can be obtained by developing epoxy-NCS nanocomposite coating [50], where the nanocomposite coating can be applied over mild steel to make it corrosion resistant. CS is a versatile biopolymer due to its significant characteristics including non-toxicity, biodegradability, biocompatibility, chemical reaction, and the presence of hydroxyl and amino groups. The conversion of CS to NCS improves the crystallinity, degree of degradation, and decomposition temperature. The decrease in molecular weight of CS usually held under acidic conditions, wherein glycosidic bonds are hydrolyzed. The NCS can also act as an organic inhibitor due to the presence of more polar functional groups such as N and O, which is further effective for providing anticorrosion characteristics. Additionally, few studies have been done related to the anticorrosive properties of CS against copper and mild steel under acidic environment [51] and the properties are further improved with increased CS concentration. The NCS added composites have also effective nature for providing better corrosion resistant property along with improved thermo-physical and physicochemical properties of coated film. The best barrier and anticorrosion properties are obtained by a 0.5% loading of NCS [50].

4.8 Synergistic Effect of Chitosan-Based Edible Food Packaging

The inherent antimicrobial properties of CS and its derivatives can be improved further by chemical modification via endowing CS with more positive charges. The functionalization of CS can be obtained by the presence of reactive amino (*N*-substitution) and hydroxyl groups (*O*-substitution). In this regard, the various chemical and biological modification techniques to modify CS and NCS has been represented in Fig. 4.7. The several CS conjugates (addition or cross-linking with other active components) for the tailored antimicrobial property includes CS-catechin conjugate, CS gallic acid conjugate, CS lauric acid conjugate, and others. Interestingly, the activity and physicochemical characteristics of CS can be tailored-made by developing nanostructured CS. Additionally, the CS structure, physical and chemical properties including mechanical strength, emulsification property, viscosity, etc., are improved through several routes. From the previous discussion, it is clear that CS is a potential versatile polymer after cellulose and has gained remarkable interest due to the attributes including edibility, biocompatibility, biodegradability, and others. Moreover, these properties can also provide significant effectiveness by the addition of other food additives and application of hybrid technologies. In this context, various EOs, organic acids and others as food additives and modified atmosphere packaging, controlled atmosphere packaging, gamma irradiation, and others have been utilized to obtain the synergistic effects when added into the CS-based films and coatings. Further, the term synergistic defines occurrence of combined remarkable effect due to the interaction of two or

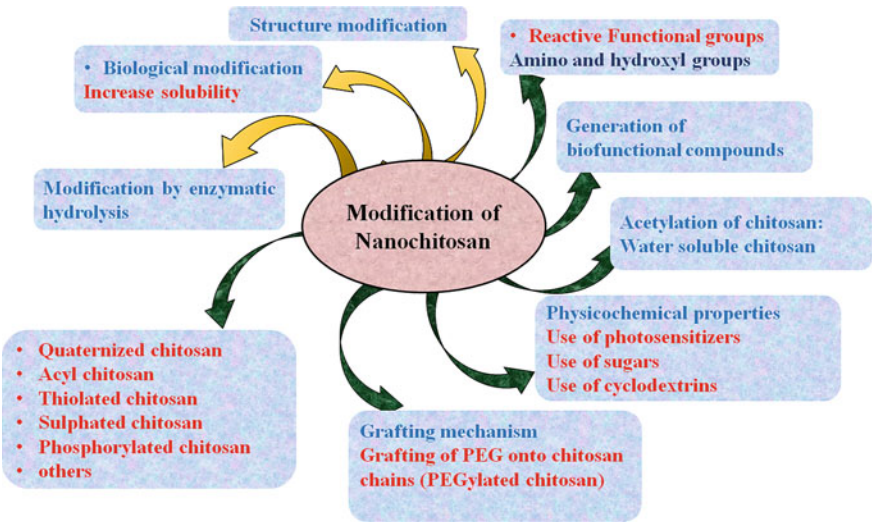

Fig. 4.7 Strategy to modify chitosan and chitosan nanoparticles properties

more substances, which is better than the individual effects. In addition, several reports reveal the improved antimicrobial activity, mechanical properties, barrier properties, etc., which are most important parameters of food packaging along with various physicochemical properties of edible packaging films/coating and help in extending the shelf life of food products due to synergistic mechanism on the packaging film. Furthermore, NCS has shown a synergistic effect when used in packaging material. In this regard, the various synergistic effects of CS-based films/ coating due to the application of one or more additives and technology has been represented in Table 4.2.

Table 4.2 Synergistic effect of CS and its derivative to various properties of food packaging material

Edible and non-edible film/coating matrix material	Synergistic agent (food additives/ technology)	Food products	Synergistic effects	References
Wheat starch (edible film)	CS Lauric acid	NA	• Enhanced antimicrobial activity against *B. subtilis* and *E. coli*	[52]
Gelatin (edible film)	CS nanofiber ZnO NPs	NA	• Improved mechanical, barrier, color and antimicrobial properties against *S. aureus, E. Coli* and *P. aeruginosa* • Improvement in quality and shelf life of food products	[53]
CS (edible coating)	Hot water dipping (42 °C, 30 min)	Wolfberry	• Reduction in loss of ascorbic acid and total phenolic content • Improved antioxidant capacity • Minimization of microbial count while storing the coated fruits at 2 ± 0.5 °C, 90% RH for 28 days. • Extended storage life and maintained postharvest quality of the fruit	[54]
CS (edible coating)	Nisin, gallic acid HO-MAP (80% O_2 + 20% CO_2)	Pork loin	• Improved antioxidant and antimicrobial activity and maintained color properties, and tenderness	[55]

(continued)

Table 4.2 (continued)

Edible and non-edible film/coating matrix material	Synergistic agent (food additives/technology)	Food products	Synergistic effects	References
			• Reduction in lipid and protein oxidation and retardation of microbial growth during storage of pork sample under refrigerated condition (2 ± 1 °C, 20 days)	
CSNP-based coating on the inner surface of LDPE (non-edible)	CSNP CEO	Pork	• Showed remarkable antioxidant and antimicrobial activity during the refrigerated storage at 4 °C, for 15 days	[4]
Agar film (non-edible)	Halloysite, CS	Not specified	• Improved mechanical properties, reduction in moisture uptake, water solubility and degree of swelling, improved transparency	[56]
PLA film (non-edible)	NCS, Medicinal plant extracted EO (*Polylophium involucratum*)	Chicken fillet	• Prolonged shelf life of refrigerated sample more than 10 days • Improved chemical, microbial and sensory properties	[57]
Quinoa protein/CS edible film PET containers coated with developed film	CS thymol NPs	Blueberries Tomato cherries	• The edible coating significantly inhibited growth of *Botrytis cinerea*, reduced water vapor permeability • The coated PET container showed reduction in weight loss of fruits while storing at 7 °C, 85% RH for 10 days	[58]
PLA matrix (Non-edible)	Rosin modified cellulose nanofiber, CS	Not specified	• Improved mechanical properties, remarkable antimicrobial activity against *E. Coli* and *B. subtilis*	[59]

(continued)

Table 4.2 (continued)

Edible and non-edible film/coating matrix material	Synergistic agent (food additives/ technology)	Food products	Synergistic effects	References
CS-based film	Silver NPs, Purple corn extract	Not specified	• Improved highest barrier, mechanical, antioxidant and antimicrobial activity • Capable to enhance the shelf-life of food product and can be used as intelligent packaging material due to occurrence of pH sensitivity	[60]
CS-based coating	Nanoemulsion of mandarin EO, Three non-isothermal treatment (γ-irradiation, UV-C and ozonated water treatment)	Green beans	• Extremely improved antimicrobial activity against *Listeria innocua* with color and texture during 14 days storage	[61]
CS-based coating	Nanoemulsion of EOs (carvacrol, mandarin, bergamot and lemon) MAP, γ-irradiation	Green beans	• The combined effect improves the entire shelf-life of refrigerated beans under 4 °C for 14 days • No detection of *E. coli* and *S. typhimurium* till the end of the storage days	[62]
NCS-based coating	Nisin, natamycin	Egyptian cheese	• Improved remarkably antimicrobial activity	[63]
CS-based edible film	Grapefruit seed extract	Bread	• Enhanced antioxidant antifungal activity	[64]
CS-based film	Natural EOs (clove bud oil, cinnamon oil, and star anise oil)	Not specified	• Improved antimicrobial activity without phase separation via CS-cinnamon film	[65]
CS-based edible coating	Gallic acid, CS	Pacific mackerel fillets	• The combined effect significantly reduced microbial growth, protein and lipid oxidation, biogenic amine formation and	[66]

(continued)

Table 4.2 (continued)

Edible and non-edible film/coating matrix material	Synergistic agent (food additives/ technology)	Food products	Synergistic effects	References
			nucleotide breakdown and maintained sensory characteristics during refrigerated storage at 4 °C for 12 days • Extended shelf-life of sample by 6 days	
CS-based coating	Pomegranate peel extract	White shrimp	• Suppression in color and pH changes, inhibited melanosis and aerobic microbial count, reduced generation of total volatile nitrogen values and improved quality during the ice storage for 10 days	[67]
HPMC-based edible biocomposite	CS, lysozyme, nanosilver	Meat	• Remarkable improvement in antioxidant and antimicrobial activity	[68]

CS Chitosan, *NP* Nanoparticle, *MAP* Modified atmosphere packaging, *CSNPs* Chitosan nanoparticles, *LDPE* Low density polyethylene, *CEO* Cinnamon essential oil, *NCS* Nanochitosan, *CS* Chitosan, *EO* Essential oils, *PLA* Poly(lactic acid), *HO-MAP* High-oxygen modified atmosphere packaging, PET Polyethylene terephthalate, *HPMC* Hydroxypropyl methylcellulose

4.9 Chitosan and Its Derivatives-Based Edible Packaged Food Products: Storage Study

The biodegradable edible films and coatings of CS and its derivatives can extend the storage life of various food products specifically perishable food products via retarding the moisture loss, reduced water vapor and oxygen permeability rate of packaging, reduced respiration rate of fruits and vegetables, reduced ethylene production rate, etc. The application of CS coating further provides restricted loss of aroma, volatile components, and firm texture and also provide the characteristics attributes of antimicrobial active, antioxidants property, which inhibit the growth of microorganisms and extend the stability time of food products. In this regard, the effects of CS and its derivatives such as NCS in the edible coating/films has been the extensively investigated edible packaging materials for improved quality and storage life of food products. Furthermore, the postharvest loss of major agricultural produce is a critical concern, where the losses can occur during processing, storage, and transportation and the use of CS-based packaging system in the form of edible films and coating in reducing the postharvest loss is considered as a potential candidate. As mentioned in the previous section, the shelf life of several available fruits and vegetables including tomato, cucumber, strawberry, mango, leafy vegetables, and many others can be enhanced due to the application of CS- and NCS-based edible coatings and films. However, the refrigerated storage has a major impact in the improved storage life of fruits and vegetables as compared to ambient storage. In addition, various cheese and fish products can also be protected using CS biopolymer-based coatings. The shelf-life of coated food products also depends on the storage and transportation conditions such as temperature, humidity, time, and mechanical abrasion. The inherent antimicrobial characteristics of CS and its nanostructured materials aid potentiality for enhancing the product life and deliver the safe quality food to the consumers. In this regard, Table 4.3 describes the remarkable effects on storage life of food products packaged with CS and its derivative-based film and coatings. However, the fabrication of edible coating based on CS and modified CS with bioactive agents, proteins (such as whey) are widely used for tailored quality attributes and enhanced shelf life of food products such as tomato, strawberry, blueberries, ricotta cheese, salmon fillets, cooked pork sausages, silver crap, radish shreds, eggs, bananas, and others. Additionally, the surface property of chitosan can be improved by adding bioactive agents such as curcumin, which can be analyzed by field-emission scanning electron microscope (FESEM)-based micrograph (Fig. 4.8). Besides, nowadays, CS nanostructured materials are focused to develop novel edible packaging system in a small- and large-scale production.

Table 4.3 Effectiveness of several CS and its derivative-based edible and non-edible films and coatings on the storage behavior of food products

Films/coating	Food items	Storage conditions	Effect on shelf-life	References
PP coated with CS and pectin layers (non-edible bag)	Tomato	Samples stored in coated PP bag under refrigerated condition at 4 °C	The sample kept in coated PP bag had a shelf life more than 13 days without deteriorating quality as compared to control	[69]
Modified CS-based edible coating with bioactive agent	Strawberry	Stored at 4 °C for 14 days	Extended shelf life of more 14 days were obtained	[70]
Acid-soluble CS edible coating	Blueberries	Commercial storage (first treated sample stored at 2 °C, 88% RH in dark room for 1 week then kept at ambient temperature at 20 ± 3 °C, 30% RH under normal light for 16 days)	No reduction in quality and shelf life during the storage period	[71]

(continued)

Table 4.3 (continued)

Films/coating	Food items	Storage conditions	Effect on shelf-life	References
CS/whey edible coating	Ricotta cheese	Stored under MAP and refrigerated condition 4 °C for 30 days	Extended shelf life of sample for more than 30 days without altering the quality	[72]
CS-based edible coating	Salmon fillets	Stored under refrigerated condition (0 °C) for 18 days	Extended shelf life by more than 3 days after storing period with reduction in lipid oxidation and microbial load	[73]
CS-based edible coating	Cooked pork sausages	Stored at refrigerated temperature (4 ± 2 °C) for 25 days	Extended shelf life of sausages till 20 days as compared to uncoated sample based on sensory and microbiological analysis	[74]
CS-based coating	Silver carp	Stored under frozen condition (-3 °C) for 30 days	Improved acceptable shelf life during the whole storage period due to the reduction in microbial count, lipid oxidation, maintained pH and barrier properties	[75]
CS-based powder coating	Radish shreds	Refrigerated storage at 10 °C for 10 days	Extended shelf life by 3 days as compared to control via maintained pH, weight loss and moisture, storing of phytochemicals and reduction in respiration rate, microbial activity	[76]
CS-based coating	Eggs	Stored at ambient condition (24 °C) for 4 weeks	Enhanced shelf life throughout the storage period for coated eggs with improved quality in comparison with uncoated egg with ended storage life after 1–2 weeks	[77]
CS–based edible coating	Bananas	Stored at 22 °C, 85% RH for 8 days	Extended shelf-life by 4 days in coated samples without color loss and browning	[78]

PP Polypropylene, *CS* Chitosan, *RH* Relative humidity, *MAP* Modified atmosphere packaging

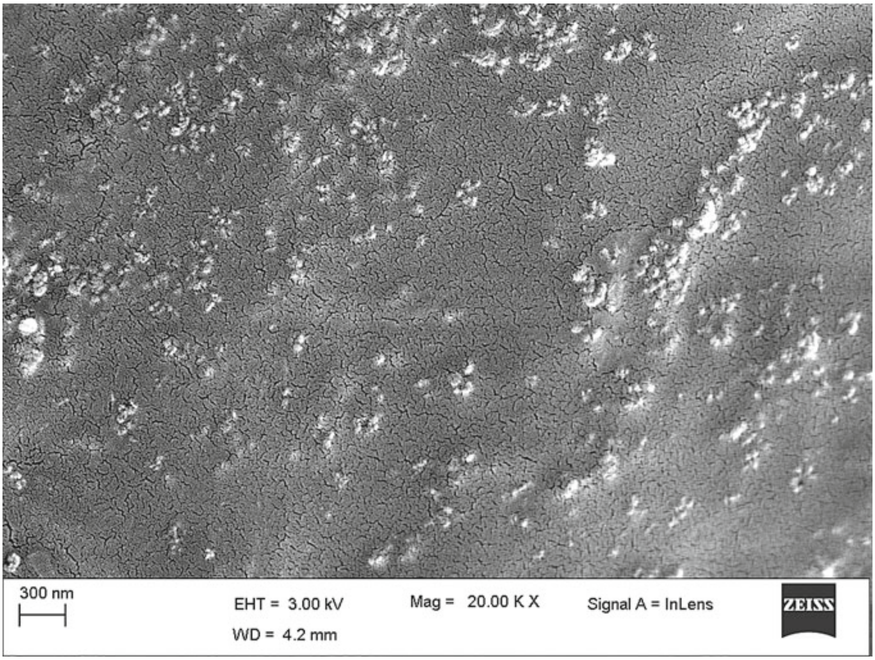

Fig. 4.8 FESEM micrograph of curcumin added chitosan-based edible films

4.10 Conclusion

In view of the raised concern of plastic-based environment issues, there is an increased interest of using edible and biodegradable films obtained from non-toxic and renewable sources. In this context, CS is a natural biopolymer derived via deacetylation of chitin, which is investigated widely for its wide availability in the recent years. This biopolymer is widely extracted from fish, crustacean, seafood materials, and others. Further, the perspective of using CS and its nanostructured forms in developing edible food packaging are biocompatibility, non-toxicity, antimicrobial property, antioxidant property, stabilizing property, nutrients property, health beneficial property, lipid lowering activity, and others. Besides, it can form a transparent film which is suitable for a wide range of food packaging application. Further, studies on CS and its blends displayed the improved properties which can be a promising candidate of active edible bio-based packaging material for extending and preserving the shelf life of food products and arresting the postharvest loss. The CS nanostructured materials in developing edible food packaging for various food products are beneficial in maintaining the product quality such as reduced respiration of fruit products, delayed ripening, and others. The characteristics attributes of CSNPs can also be tailored in terms of the dimension, size and shape, surface property, and others to obtain improved shelf life of food products.

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Chapter 5
Starch-Based Nanostructured Materials in Edible Food Packaging

Tabli Ghosh, Munmi Das, and Vimal Katiyar

5.1 Introduction

From early days, starch and its derivatives are a potential candidate in various food industries including beverages, food packaging, confectionary, bakery products, instant meals, and others. In food packaging industry, the increasing environmental concerns related to the consumption of plastics based on petroleum resources have led to the development of healthier products prepared with sustainable and environment-friendly materials [1, 2]. The eco-efficient products are expected to meet with the ecological, environmental and economic requirements, and thus, focus has been emphasized on the production of edible films, using starch-based materials for existing noteworthy property [3]. The conventional plastics used in food packaging are generally contaminated by food matter, and thus, their recycling becomes impractical. Thus, fabrication of edible food packaging using starch nanostructured materials with tailored properties using novel strategies can resolve the problems related to recycling and waste generation. Considerably, starch-based nanostructured materials provide a new platform in the development of novel edible packaging systems [4]. Considerably, the development of sustainable packaging has become the foremost requirement using naturally available materials as a replacement due to the environmental concern [5]. Starch, a promising natural polysaccharide, is used as an efficient and sustainable edible packaging material because of its abundancy, renewability, low cost, biodegradability, and thermosplasticity compared to other natural resources.

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Moreover, the use of starch-based packaging materials is growing day by day due to water solubility and easy biodegradation. The various sources of starches are cereals (rice, wheat, maize), root vegetables (potatoes and cassava), stems, pith (sago), and others. The various categories of starches are obtained based on sources, and amylose and amylopectin content (waxy starch, normal starch, high amylose starch), and others. Among the available sources, cassava starch is extensively used category of starch due to the low cost, colorless, good oxygen barrier, and other features. The rich sources of starch-based foods are fast food, French bread, brown rice, potatoes, whole wheat pasta, sweet corn, bananas, apple pie, etc. Based on this discussion, starch is safe for consumption, thus targeted to be used as an edible food packaging material. However, the commercialization of starch films is restricted due to poor mechanical property, moisture sensitivity, and others. In the presence of dense interchained H-bonds network, starch films exhibit brittleness which thereby lead to cracking. To increase the elasticity and mechanical property of these films, plasticizers or blending agents are incorporated into the polymeric network, consequently increasing the molecular movement, which further decreases the glass transition temperature (T_g). However, safety measures should be followed to ensure the developed edible packaging to be non-toxic in nature [6–8].

The present-day demand to produce novel materials with improved shelf life and quality has led researchers to a new dimension in the food packaging industry using nanotechnology [9]. Nanostructured materials have at least one of its dimensions in nanometer range and are usually classified as polymeric nanoparticles (NPs), liposomes, nano and microemulsions in the food technology and also exist as layered films, or in cluster forms [10]. These nanostructured materials contribute as functional materials by improving the mechanical strength, barrier property, antimicrobial and antioxidant activity which in turn increase the quality and shelf-life of the food [11]. In this chapter, a discussion on the preparation and property analysis of starch-based nanostructured materials will be discussed with appropriateness of their applications as a noteworthy candidate for edible food packaging. Moreover, the effect of starch-based nanostructured materials with other added agents will also be discussed. In this regard, the improved packaging features of starch nanomaterials can be modified using plasticizers, cross-linkers, biopolymers, and others.

5.2 Effectiveness and Categories of Starch-Based Resources

5.2.1 Components of Starch

Starch is used as a versatile material in the food industry in the form of gelling component, stabilizer, thickener, water retainer, which enhances the texture, adhesiveness, gel formation, moisture retention, miscibility between various components, and viscosity of the end products [11]. The basic structure of starch is $(C_6H_{10}O_5)_n$, and it mainly comprises two biomacromolecules amylose and amylopectin, along with the presence of a small amount of protein, lipid and phosphorus. The dimension of starch granules is within the range of 2–100 μm consisting of crystalline and amorphous lamellae. Amylose is a linear glucose molecule with few branches connected by (1–4) α-D-glycoside bonds and comprises around 20% of normal starch, whereas amylopectin consists of highly branched α-D-(1–4) glucopyranose units interconnected by 5% α-D-(1–6) glycosidic linkages [12]. Depending on the ratio of amylose/amylopectin content, starch is again classified as waxy, normal, and high amylose starch where waxy starch comprises a higher amount (90% or more) of amylopectin and relatively less amylose. Similarly, normal starch constitutes around 15–30% amylose, and high amylose starch contains an amylose content of more than 50% as shown in Fig. 5.1. The amylose and amylopectin molecules are present in an alternating interwined manner in the starch granule [11, 13, 14]. Starch is usually insoluble in water at room temperature but when heated swells and disintegrates. Further, the water solubility property of starch depends on the composition of amylose and amylopectin. The swelling phenomenon of starch takes place by the formation of H-bond between the water molecules and free hydroxyl group of the glucan chains at a granular level [15].

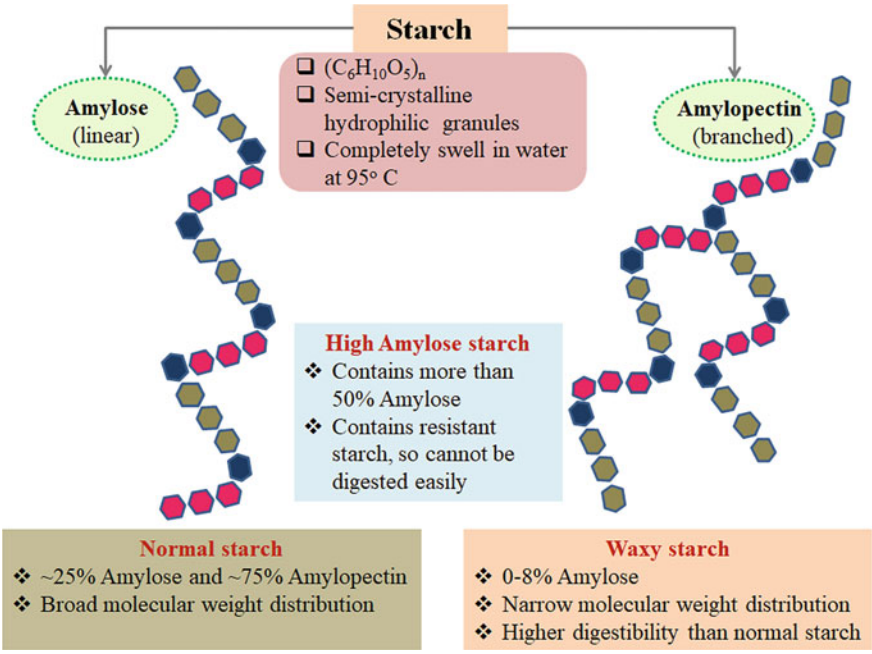

Fig. 5.1 Classification of starch, based on amylose and amylopectin content

5.2.2 *Sources of Starch*

Starch is extracted mainly from plant sources as an energy reserve, specifically from seeds, roots and stems, crop seeds, tubers, and also from some fruits. Some common examples of starch-based sources are rice, wheat, potato, corn, maize, cassava, apple, tomato [12, 16]. Table 5.1 illustrates some common types of starch based on their botanic origin, size, and amylose content [12, 17]. The amylose content varies from 0.5 to 83% based on their botanical origin, which further, influence the rheological, thermal, processing characteristics of starch as well as their structural features. As mentioned in earlier section, the ratio of amylose/amylopectin also determines the mechanical strength, swelling behavior, oxygen permeability, gelatinization, viscosity, water binding capacity, and texture of the starch-based materials. These properties are source-dependent and an increase in amylose content increases the thermal stability of starch-based materials [18]. Additionally, resistant starch, another type of health beneficial starch, is obtained from beans, legumes, lentils, peas, whole grains (oats, barley), cashews, green bananas, cooled rice, cooked rice, raw potato starch, hi-maize flour, etc. The beneficial features of resistant starches are increase stool weight, reduce secondary bile acids, stimulate immune system, reduce intestinal pathogen levels, reduce risk factors relating large bowel cancer, etc.

5.3 Starch for Targeted Food System and Edible Food Packaging

From very early days, food preservation maintains the nutritional level and quality of foods intact, by increasing the shelf life of the packaged edible food. Food preservation technique is a complete cycle starting from growing the food to distribution of the food to the end users. Food preservation protects the food product by improving its shelf life, antimicrobial activity resulting in a sustainable edible packaging. Commonly used food preservation processing techniques are drying, freezing, chemical preservation, chilling, pasteurization, fermentation as shown in Fig. 5.2 [19, 20].

Starch-based materials are also used for developing food products and packaging materials to improve the shelf life of food products and others. The intake of corn starch has many health benefits such as reduce blood pressure, boost immune system, source of vitamins and minerals, and antioxidants. The starch-based derivatives and nanostructured materials have various noteworthy applications including (1) Food packaging sector as matrix and filler materials in edible and non-edible packaging for improved product life; (2) Pharmaceutical industry: Starch is used for developing colloidal formulations; (3) Food Products: Sauces, bread, candies, baby foods, sausage, thickeners, and others. The main functions of starch in food products are thickener (puddings, sauces, pie fillings), binder (breaded items, formed meats),

Table 5.1 Properties of different starch, based on their botanic origin

Source	Maize	Barley	Wheat	Rice	Waxy starch	Waxy barley	Arrowroot	High amylose	Cassava	Sweet potato	Potato	Smooth pea	Wrinkled pea	Ginger
Average granule size (µm)	30	8–26	30	2–7	15	8–26	>30	5–25	3–30	3–27	40–100	2–40	17–30	>20
Apparent amylose content (%)	25–28	29.8	21.5–26.6	29.1	0.5	9.1	20.8	60–73	19.8	22.6	26.9	33–48	60–80	26.5

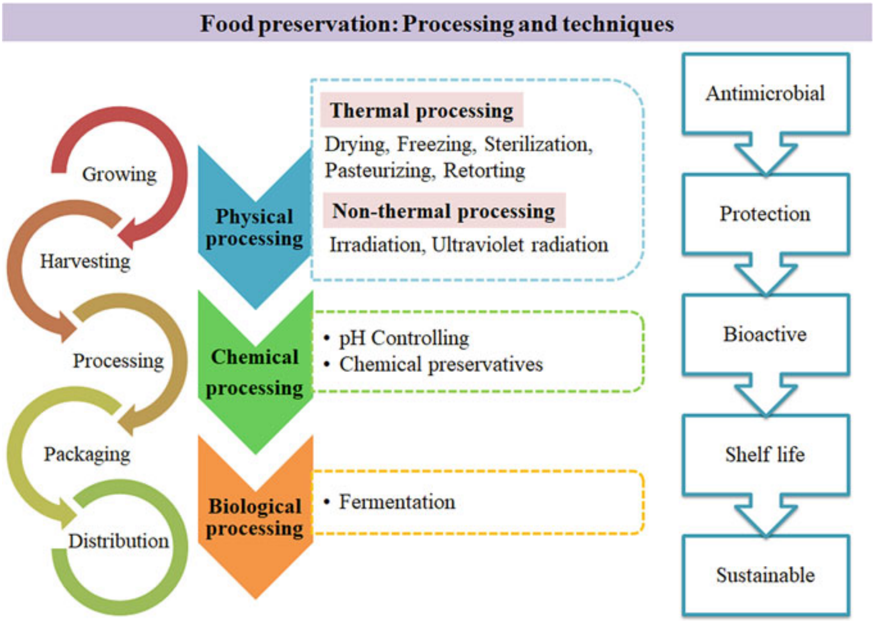

Fig. 5.2 Food preservation techniques in edible packaging

coating (toppings, candies, glazes), water binder (cakes, jellies), releasing agents (candy making), texture modifier (meat products, processed cheese), fat replacer (dairy products, baked goods, salad dressings), etc. Additionally, the functions of starches in food products are adhesion, thickening agents, shaping, molding (gum drops), gelling, glazing, binding, and others. The various forms of starch to be used in food systems are nanostructured materials (NPs, nanowhiskers, nanocrystals (NCs), etc.), nanofilms, hydrogel, microbeads, microsphere, tablets, thermoplastic foam, and others.

5.4 Global Industrial Starch Market Based on Region, Source, Application and Industry

The global industrial application of starch-based resources is food industry, chemical industry, beer industry, modified starch industry, biopolymers, medicine, animal, and others. Besides, the main sectors of starch-based non-food industrial applications are adhesives, paper industry, construction industry, cosmetics, metals industry, textiles, mining, etc. Interestingly, food preservation holds a crucial position in the global economy, and it is predicted that the industrial starch market will grow from USD 72.51 billion (2016) to 106.64 billion USD by 2022.

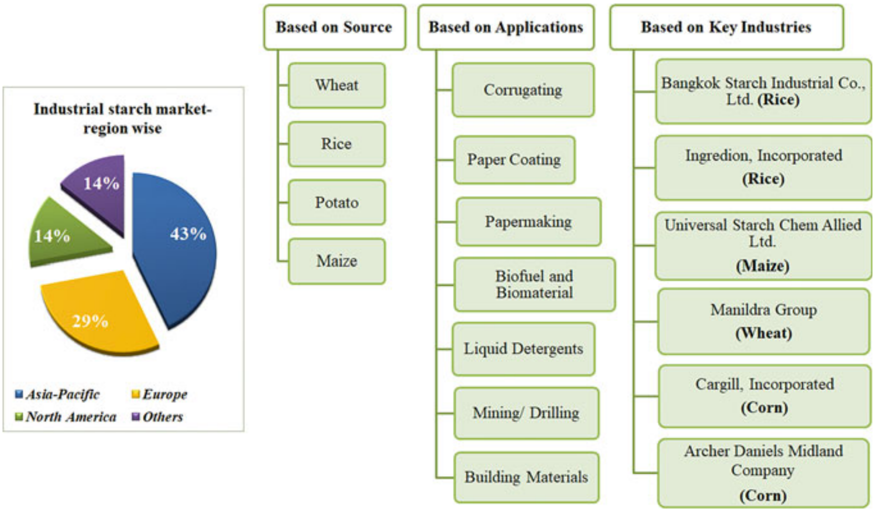

Fig. 5.3 Global industrial starch market based on region, source, application, and industry

Figure 5.3 elaborates the division of the industrial starch market globally among North America, Asia-Pacific, Europe, and other parts of the world. China alone contributes to half of the global starch consumption resulting in the emerging Asia-Pacific market with a wide range of applications. The industrial starch market is classified according to the type of native starch and their derivatives and sweeteners [21]. Figure 5.3 showcases various industrial starch manufacturers across the globe, which includes Cargill (USA), Archer Daniels Midland Company (USA), Universal Starch Chem Allied Ltd. (India), Ingredion Incorporated (USA), Manildra Group (Australia) and Bangkok Starch Industrial Co., Ltd. (Thailand) and their specialized applications as biofuel and biomaterial, mining and building materials as well as pharmaceutical, corrugating, and paper-making materials [22].

5.5 Extraction of Starch from Available Sources

The pure starch extracted from plants appears as a white-colored powdery material in the form of distinct semi-crystalline granules within 1–100 μm size range, insoluble in cold water and alcohol and is particularly used as a thickener or binding agent in paper and food industry [12, 16]. Commonly used extraction methods are alkaline extraction, aqueous extraction, ethanol extraction, ultrasound extraction, microblending and homogenization. The starch structure and morphology vary with every extraction process. The extraction methods of various starch based on their botanic origin are mentioned in Table 5.2. The starch extraction has been reported from available sources such as rice beans, white garland lily rhizomes, pigmented rice, *pachyrhizus ahipa* roots, radix puerariae, plantain (*Musa paradisiaca*) peel,

Table 5.2 Extraction of starch using various methods from different sources

Si. No.	Source	Pretreatment methods	Extraction methods	Starch granule size/yield%	Reference
1.	Rice beans	Milling and sieving followed by hydrothermal processing	a. Aqueous extraction b. Ethanolic extraction	a. 15–25 μm b. 25–35 μm	[23]
2.	White garland lily rhizomes	Peeled and cut	Aqueous extraction	Thickness = 2–6 μm Length = 12–38 μm	[24]
3.	Pigmented rice (white, red, and black)	–	Aqueous extraction	White rice = 44.0%, Red rice = 47.0% and Black rice = 35.7%	[25]
4.	*Pachyrhizus ahipa* roots	Roots are washed with water and immersed in NaClO solution to sanitize for 10 min, peeled and cut	Aqueous extraction	Starch yield = 15.7%	[26]
5.	Radix Puerariae	Peeled, cut, and grinded	High-intensity ultrasound	High amylose starch yield of 44.5%	[27]
6.	Plantain (*Musa paradisiaca*) peel	Washed and sanitized with NaClO and cut	Aqueous extraction	Yield = 16.6–48.5%	[28]
7.	Sorghum	Washed and dried to remove impurities, followed by milling	Homogenization	Yield = 26%	[29]

sorghum and others [23–29]. The pretreated rice beans (pretreated method: milling and sieving followed by hydrothermal processing) are reported to develop starch granules using aqueous and ethanolic method having granules size of 15–25 and 25–35 μm, respectively [23]. Additionally, starch granules having thickness and length of 2–6 and 12–38 μm, respectively, can be extracted from white garland lily rhizomes using aqueous extraction approach [24]. The yield of starch varies based on the available source and starch extraction methods. In this regard, the yield of starch extracted from white rice, red rice, and black rice using aqueous extraction is 44.0, 47.0 and 35.7%, respectively [25]. On the otherhand, the extraction of starch from *pachyrhizus ahipa* roots using aqueous extraction method can provide starch yield of 15.7% [26]. The peeled, cut and grinded radix puerariae is used to extract

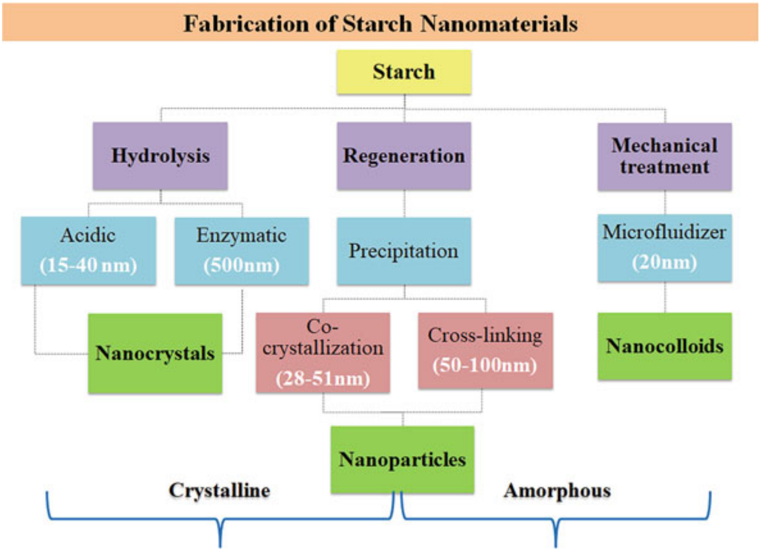

Fig. 5.4 Fabrication methods to obtain starch nanomaterials

high amylose starch (yield of 44.5%) using high-intensity ultrasound [27]. Additionally, using the aqueous extraction method, starch with a yield of 16.6–48.5% can be extracted from plantain (*Musa paradisiaca*) peels [28]. The several extraction methods of starch are aqueous extraction, ethanol extraction, ultrasound extraction and homogenization.

5.6 Fabrication of Starch Nanomaterials

Starch nanomaterials are categorized into different forms, such as starch NPs, NCs, and nanocolloids based on their size, shape, crystalline behavior, and preparation method. Various methods including chemical, physical, enzymatic, and blending are used to fabricate starch nanomaterials from the native starch granules. The commonly used methods in the fabrication of starch nanomaterials are discussed below and shown in Fig. 5.4.

5.6.1 Starch Nanocrystals: Acid Hydrolysis and Enzymatic Hydrolysis

Pure starch granules are semi-crystalline in nature with an onion-like structure in which the amylose content constitutes the amorphous region, and amylopectin content contributes to the crystalline region. Upon hydrolysis, the amorphous content is discarded to give NCs with higher crystallinity. Starch NCs (STNCs) contribute in increasing the mechanical properties when combined with a polymer matrix and are dominantly used as emulsion stabilizers, in bioplastic packaging and as drug carriers [30, 31].

Acid hydrolysis. Starches with higher amylopectin content, for example, waxy maize starch is mostly used for the preparation of NCs due to its higher degree of crystallinity. The extracted pure starch material usually undergoes a pretreatment prior to hydrolysis, where moisture content of starch is adjusted by the addition of distilled water, aqueous alcohol or alkaline solution. Further, these starch suspensions are washed, centrifuged, neutralized followed by drying of the precipitated starch. The obtained starch powder is then grinded and filtered through a sieve [31, 32]. In a recent study, STNCs from Quinoa starch were obtained by dispersing the starch powder in a sulfuric acid solution, followed by incubation for 5 days with continuous stirring at 200 rpm at a fixed temperature. The hydrolysis continued for 5 days, and then, the starch solution was centrifuged, neutralized to obtain a suspension which was further homogenized and freeze-dried to give STNCs of 22.8 and 6.8% yield, respectively, at 35 and 40 °C. The characteristics and yield percentage of STNCs vary depending on the starch source and hydrolysis conditions, such as acid type and concentration, reaction temperature and time [33].

Enzymatic hydrolysis. The optimum longer duration (5 days) of acid hydrolysis to obtain STNCs led to the utilization of enzymes in order to lessen the hydrolysis time and keep the inherited characteristics of STNCs intact. The starch modifying enzymes, for example, α-amylase, amyloglucosidase, aspergillopepsin1, and glucoamylase are generally used to improve the qualities of starch in enzymatic treatment [30]. Starch converting enzymes are usually classified as amylases and transferases, where former hydrolyzes the glycosidic bonds and the latter helps in transfer of the bonds. Enzymatic hydrolysis, with the use of amylases, helps in modifying the viscosity and degree of polymerization of starch. In a study, α-amylase enzyme enabled the fragmentation of starch granules in just 3–6 h leading to the formation of crystalline STNCs [34, 35]. In a study, waxy rice starch was hydrolyzed enzymatically for 3 h using α-amylase, followed by ultrasonication in ethanol to obtain 500 nm-sized STNCs [36]. In a typical enzymatic hydrolysis pretreatment, initially the enzyme is dissolved in a pH buffer (pH = 5.5–6.9) solution. To this enzyme solution, a weighed quantity of starch powder is added and allowed to incubate at 37 °C for a day. Further, the enzyme activity is stopped with the addition of ethanol, and the obtained hydrolyzed product is centrifuged and washed and dried overnight at 35 °C to give STNCs [35, 36].

5.6.2 Starch Nanoparticles: Regeneration and Cross-linking

Regeneration. The formation of amorphous starch NPs by precipitating the gelatinized starch in a chosen solvent usually refers to the regeneration process.

Development of starch NPs for utilization in nanocomposites preparation has grabbed attention due to its unique and improved mechanical and barrier properties. The most recently explored processes to develop starch NPs efficiently are (a) precipitation of gelatinized amorphous starch, (b) combination of enzymatic hydrolysis and complex formation, and (c) reactive extrusion or microfluidization [37].
Ma et al. reported the synthesis of starch NPs by preparing a gelatinized starch solution and further precipitating this solution in ethanol [38]. However, this process is not considered economical as it involves the use of a large quantity of solvent for precipitation and difficulty in controlling the proper size of NPs. To reduce the consumption of solvent, Hebeish and coworkers used aqueous alkaline (NaOH) solution as the solvent system to disperse the maize starch. This starch suspension was then stirred continuously at 25 °C for 120 min to obtain a uniform gelatinized starch. Absolute ethanol was then added as a precipitating agent in a dropwise manner to this gel suspension under continuous stirring to obtain spherical starch NPs of 135–155 nm size range [39].

Cross-linking. Starch NPs possess reactive surface which tend to agglomerate, and thus, a chemical modification is required to maximize its dispersion in aqueous medium. Ren et al. modified the surface properties of starch NPs with the use of sodium hexametaphosphate (SHMP) as a cross-linker. The modification was carried out below gelatinization temperature (40 °C) of the waxy maize starch which resulted in a stable and uniform dispersion of starch NPs in water [40]. Also, Ma et al. used citric acid as a cross-linker to stabilize the NPs, and the modified starch exhibited the properties of resistant starch with various health benefits. These citric acid modified starch NPs when incorporated into the polymer matrix resulted in improvement of glass transition temperature, barrier properties, and tensile strength [38].

5.6.3 Starch Nanocolloids: Mechanical Treatment/ Microfluidization

Microfluidization enables the preparation of nanocarriers by reducing microsize to nanometer scale without disturbing the crystal structure of the starch granules. The particle-size distribution can also be tuned by adjusting the processing conditions. Some mechanical treatment involves the use of ultrasound waves, which converts to energy by the formation of cavity in the starch microemulsion. This results in the formation of bubbles which collapse and produce more energy thereby reducing the droplet size in the emulsion [41].

A microfluidizer uses high-pressure homogenization process which basically manipulates the steady flow of liquid via microfabricated channels. Various external factors activate the flow of liquid through the channels, such as pressure, mechanical pumps, and micropumps. Microfluidization is carried out without any chemical or physical pretreatment. Thus, the particle size of a high amylose corn starch slurry containing sediment and dispersion in water is reduced from 3 to 6 μm to a nanometer scale of 10–20 nm, under 207 MPa pressure by this technique. This high-pressure homogenization transformed the starch sol into a stable gel-like suspension and increased the viscosity of the starch nanocolloid without affecting its thermal stability [42].

5.7 Features of Starch and Its Derivatives for Potential Application in Food System

As discussed in Fig. 5.5, the features of starch and its derivatives for potential application in food systems are biological properties, thickening property, rheology enhancer, film-forming property, water binding capacity, and others. In this regard, a brief discussion on the various beneficial properties of starches for edible food packaging has been discussed in the below section.

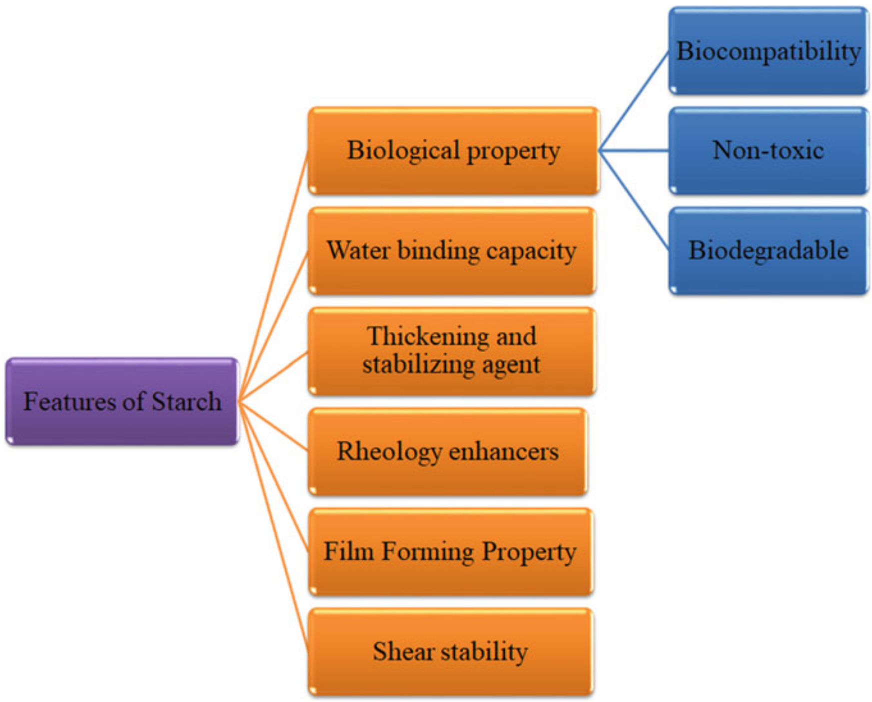

Fig. 5.5 Features of starch for application in food system

5.7.1 Biological Properties: Biocompatible, Non-toxic and Biodegradable

Starch is a biobased material having non-toxic, biocompatible, and biodegradable nature, making it a potential candidate for edible food packaging and others food systems. The starch polymers do not induce toxicity over different cell types and further reported to offer important cell behavior with immune potential [43]. The biocompatibility test of starch-based composite can be done via cell adhesion test and cytotoxicity test, where the starch polymers proved to exhibit cytocompatibility to be used as a biomaterial [44]. Additionally, the biodegradable starch-based materials have potential application in biomedical section as the materials do not provide any adverse effect in in vitro and in vivo models [45]. The biodegradable nature of starch and its derivatives make it a promising candidate to develop sustainable packaging materials in the form of foam, films, hydrogel, coatings, and others. In this regard, improved oral delivery of poorly water-soluble drugs can be obtained using biodegradable porous starch foams [46]. The several biodegradable polymeric materials which are used with starch for focused applications are proteins, cellulose, gums, which help to increase the shelf life of food products when used as a food packaging material [47]. The preparation of bionanocomposites of polymers has some additional benefits to be used in food packaging (edible and non-edible) are (1) Soil composting due to microbial action; (2) Controlled degradation using nanomaterials; (3) Use of renewable resources with improved property; (4) Degradation by water and sunlight; and others.

5.7.2 Water Binding Capacity

The thermo-mechanical properties of starch-based films are influenced by water binding capacity of starch, where the water binding capacity of starch is dependent on the hydrophilic sites, which can be modified through plasticizers and molecular substitution. The water binding capacity of starches can be measured using differential scanning calorimetry (DSC) method or filter paper suction pressure method. The water binding capacity of starch depends on the amylose/amylopectin ratio, which further influenced by the starch type or sources such as high amylose rice, waxy rice, and others. The increase in amylose content of starch reduces the water binding property of starch molecule. Considerably, the pregelatinized starch has a disruptive internal structure of granules which increase the water binding capacity of starch. However, the chemical modification of starch has an ability to block the route to water flow in the granules, which in turn reduce the water uptake ability of starch. Additionally, molar substitution of hydroxylpropyl maize starch also influences the water binding capacity [48]. The water binding capacity of starch can be decreased by developing cross-linked starch with phosphorus oxychloride [49]. The type and concentration of plasticizers effect the hydration property of plasticized starch film [50].

5.7.3 As Thickening and Stabilizing Agents

The foam/emulsions can get stabilized using thickening agents such as starch, gums, and others. The starch and protein systems as foaming and stabilizers are used. Furthermore, starch is used as thickeners and stabilizers in various food products including pudding, soups, gravies, salad dressings, pastas, and others.

5.7.4 Rheology Property

The rheological property of starches is pasting property, viscosity of starch paste, and rheological features of starch gel. The addition of food additives modifies the rheological properties of starch such as lipids, salts, sugars, and others. The rheological behavior of starch is a crucial parameter to control the properties required for food applications. The thermal transition of starch-based films is also influenced by rheological property such as molecular mechanism and enzyme digestibility. Additionally, the rheological property of starch helps to offer carbohydrate concentration, heating rate for developing products, and other processing condition.

5.8 Modification of Starch and Nanostarch for Edible Films and Coating

Starch is a second most abundantly available biomass materials in nature, which is available as a source of stored energy. Native starch has many limitations such as limited digestibility, and poor functional properties. In this regard, the different types of modified starches are acid-treated starch, alkaline-treated starch, dextrin, oxidized starch, bleached starch, starch acetate, acetylated oxidized starch, starch sodium octenyl succinate, hydroxypropyl starch, hydroxypropyl distarch glycerol, hydroxypropyl distarch phosphate, enzyme treated starch, and others. The various routes of modifying starch-based films have been represented in Fig. 5.6. The starch has a wide application in the field of packaging materials such as biocomposite films, edible films, coatings, water absorbent polymers, and encapsulation. Thus, starch has gained a considerable attention in attaining a wide usability for versatile applications due to the complete biodegradable nature and provides potential functional and nutritional properties. However, the thermoplastic starch is widely used for developing commercial packaging materials in the form of carry

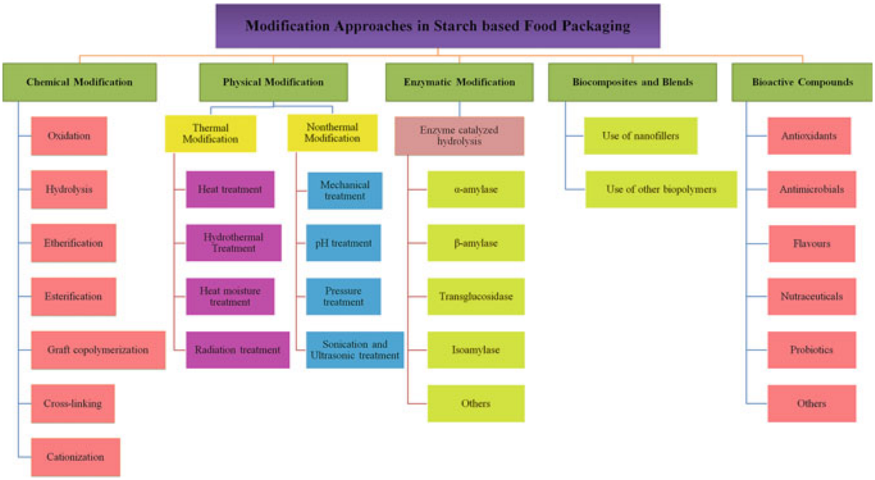

Fig. 5.6 Modification of starch and nanostarch for edible films and coating

bags and foams. Interestingly, thermoplastic starch has some noteworthy properties in comparison with starch such as improved plasticity and thermally processability, where the type of plasticizers in developing thermoplastic starch effects the mechanical property, glass transition temperature of the materials, and the use of glycerol provides less rigidity. The functional property of starch can be tailored-made by varying the dimensions (shape and size) of starch, proportion of amylose and amylopectin, distribution of chain length, etc.

5.8.1 Chemical Modification

The hydrophilicity of starch-based films is a main constraint in limiting its use in the field of edible food packaging. The chemical modification of starch helps to improve the hydrophobicity by substituting the hydrophilic groups of starch molecules. The chemical modification of starch includes the formulation of starch derivatives through functionalization technique such as etherification, esterification, cross-linking, and grafting. The cross-linking agents used in starch modification include glutaraldehyde, boric acid, sodium trimetaphosphate, hydroxypropylation-acetylation, hydroxypropylation-cross-linking, etc. Additionally, the decomposition reaction of starches includes acid hydrolysis, enzymatic hydrolysis and oxidation. The chemical modification approaches help to modify the structure of native starch, which in turn influence the starch film property. The use of plasticizers can improve the properties of starch-based films. In this regard, the several plasticizers for developing edible food packaging include glycerol, sorbitol, formamide, xylitol, etc. The plasticized sago starch using modifying agent such as sorbitol can offer improved thermal features such as improved heat sealability and reduced onset temperatures. The citric acid is used as a modification agent in starch/hydroxypropyl methylcellulose-based films such as reduced glass transition temperature. The use of hydrochloric acid in fabricating acid hydrolyzed starch offers increased heat resistance. Starch nanostructured materials have surface hydroxyl groups and high specific area, which provide the potential to tailor the packaging property via surface chemical modification, providing surface functionality. The modified starch in the form of starch nanocrystal (STNC) has high surface area and thus provides better dispersion property in water than starch granules. Further, the oxidation of starch produces starch oxides, starch radicals; esterification of starch produces starch acetylation; and etherification of starch produces carboxymethylation, hydroxypropylation, and hydroxylethylation.

5.8.2 Physical Modification

Starch modification using physical methods is obtained using thermal and non-thermal methods. The thermal method for starch modification includes heat treatment (simple oven heating, super heating, extrusion, fluidized bed heating), hydrothermal treatment, heat-moisture treatment, and radiation treatment (microwave irradiation, ultraviolet irradiation, gamma irradiation, electromagnetic irradiation). On the otherhand, the mechanical treatment for starch modification includes mechanical method (simple milling/grinding, mechanical activation by stirring), pressure treatment (high-pressure treatment, pressure treatment, osmotic pressure treatment, hydrostatic pressure treatment), ultrasonic treatment (simple, ultrasonication, high-pressure sonication), cold plasma, deep freezing, etc. The physical modification techniques of starch include the use of ultrasound waves, microwave-radiation, osmotic pressure treatment, pulsed electric field, moist-heat treatment, annealing, gamma radiation, dry heating, and others. This approach includes the treatment of starch granules under different pressure, moisture combination, shear, and others. The physical modification of starch using ultrasonic waves provides reduced reaction time, improved degree of substitution, and others. The application of dry heat to starch provides improved water binding capacity; application of gamma irradiation offers improved mechanical and swelling property [51]. The physical modifications of native starch can deliver tailored-made features of water solubility and reduced particle sizes. The physical modification of starch is attained via several approaches such as heat-moisture treatment, annealing (annealed starch), extrusion, freezing, microwave treatment, retrogradation, pregelatinization (obtained by roll-drying, spray, thermal), radio treatment (radio treated starch), and others.

5.8.3 Enzymatic and Genetic Modification of Starch

The enzymatic modification of starch produces various constituents such as maltodextrin, cyclodextrin, and amylose. The enzymatic or genetic modified starch provides novel functionality in starch due to stability and bioavailability. The enzymes for starch modification include α-amylase, β-amylase, transglucosidase, isoamylase, and others.

5.8.4 Biocomposites and Blends of Starch and Its Derivatives

The starch-based composites and blends are developed to provide tailored-made attributes such as chitosan, protein, and cellulose. The attributes of nanostarch such as reaction activity and unique surface area make it a potential nanofiller to develop biocomposites with tunable properties. The nanostructured starch can be obtained as STNCs, starch NPs, and nanocolloids. STNCs can be extracted using acid hydrolysis process such as acidic and enzymatic hydrolysis. The starch NPs can be obtained via regeneration and cross-linking process, whereas nanocolloids can be obtained via mechanical treatment and microfluidizers. The limitations of starch include hydrophilicity, thermal decomposition, and brittle in nature. To combat these limitations, starch and its nanostructured forms are modified via following several routes such as chemical, physical and enzymatic methods. The properties of starch are also improved by making blends with biopolymers. The oxidized starch includes hydroxypropylation, sodium hypochlorite, hydrogen peroxide, persulfate, etc. The oxidized starch is used to coat food products to maintain the food properties. This specific biopolymer has a matrix forming properties to develop biocomposite-based edible food packaging. The fabrication of starch-based biocomposites can be done by dissolving starch at higher temperature followed by mixing of the subsequent biopolymeric solution as shown in Fig. 5.7. As represented in Fig. 5.8, the surface of starch-based biocomposites can be modified using gum arabic and other filler materials as evident by field-emission scanning electron microscopy (FESEM). The tailored-made features of starch biocomposites are obtained by using different types of nanofiller materials such as (1) Polysaccharide-based nanoreinforcements from cellulose, starch, chitosan, chitin, etc.; (2) Protein-based nanoreinforcements; (3) Carbonaceous nanofillers; and (4) Phyllosilicates and others [52].

5.8.5 Addition of Bioactive Compounds

The bioactive starch-based films and coatings can be fabricated with the aid of antimicrobial and antioxidant agents. Additionally, nanosystems of starch are also used in encapsulating different bioactive compounds such as caffeine, curcumin, ciprofloxacin, and others for increased bioavailbility and stability. The inclusion of bioactive such as antimicrobials, antioxidants, probiotics, and flavors to starch

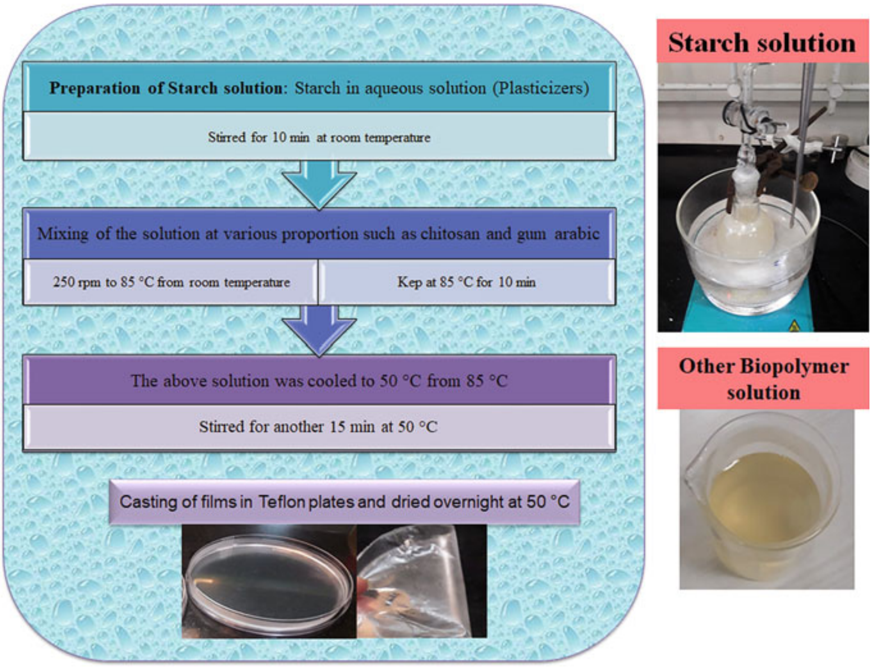

Fig. 5.7 Fabrication of starch-based biocomposites using chitosan and gum arabic

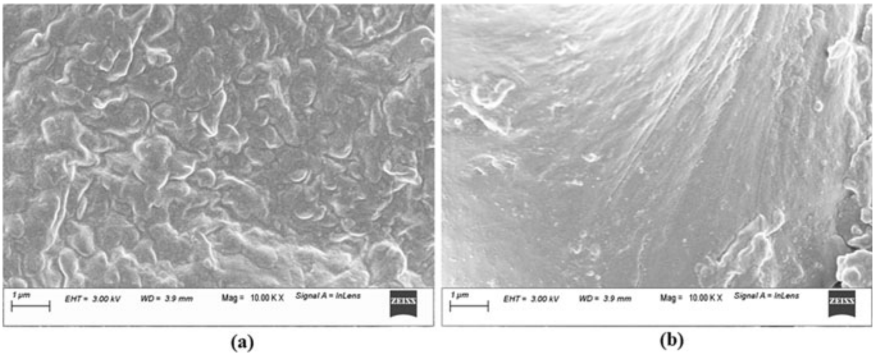

Fig. 5.8 FESEM micrograph of **a** starch/chitosan and **b** starch/chitosan/gum arabic biocomposites

provides improved film properties in terms of functionalized starch-based edible coating assisted by bioactive materials. Additionally, starch is used to encapsulate bioactive compounds. The used bioactive compounds in starch films are antimicrobials, antioxidants, probiotics, nutraceuticals, flavors, and others with a focus to develop functional food products.

5.9 Case Studies on Nanostarch-Based Edible Food Packaging

As mentioned in the earlier section, the acid hydrolysis of starch granules for a long period can hydrolyze the amorphous region, which helps in separating the crystalline part of the starch by fabricating STNCs. However, the acid hydrolysis is generally held at temperature below gelatinization temperature. The STNCs fabricated from waxy maize starch granules via acid hydrolysis (sulfuric acid) can provide relative crystallinity of 63% after 6 days of hydrolysis. The size ranges of STNC fabricated from waxy maize starch granules are 40–80 nm (as evident by transmission electron microscopy (TEM)). The nanocomposite films based on STNCs and carboxymethyl chitosan can provide maximum tensile strength of 29 MPa; and further, the water absorption and water vapor permeability can also be improved using STNC; whereas, the percentage elongation at break decreases with increased STNCs [53]. The STNCs from potato starch granules (fabricated using sulfuric acid-based hydrolysis) and sour lemon peel extract can be used to prepare edible coating on chicken fillets [54]. The application of the edible coating on chicken fillets improves the physicochemical, texture, and sensory properties during 12 days of cold storage. Additionally, high antioxidant ability (against oxidative deterioration) and antimicrobial property (against microbial spoilage) in chicken fillets can be obtained using coating solution of STNC (4.0%) and sour lemon peel extract (5.62%) prepared at 51.17 °C for 43.29 min. The STNC can be fabricated from waxy maize starch via acid hydrolysis at 35 °C, and the fabrication of STNC-reinforced sorbitol plasticized pullulan-based edible films can deliver improved packaging properties in terms of crystallinity, water uptake behavior, mechanical properties, and others [55]. The nanocomposite films based on STNCs and cross-linked cassava starch can be used as an edible coating for Huangguan pears, where the nanocomposite films can maintain texture, color, soluble solids, cell membrane permeability, and other properties of pears [56]. The films based on cross-linked cassava starch with 6% STNCs provide better mechanical and water vapor barrier properties.

The gelatin and starch have the characteristics properties of eco-friendly, safe, abundance, cost-effective, edible and thus can be used in combination to develop edible films. Additionally, the fabrication of smart edible films using starch-butanetetracarboxylic acid dianhydride-N-hydroxysuccinimide (SBN) cross-linked gelatin can provide improved surface hydrophobicity, tensile strength, elongation at break and is applicable for improved quality of postharvest shelf life [57]. The tensile strength of neat gelatin, 5% SBN-gelatin, 10% SBN-gelatin, 15% SBN-gelatin is $\sim 35.73 \pm 0.41$ MPa, 43.36 ± 0.38 MPa, 45.37 ± 0.88 MPa, 46.65 ± 0.63 MPa, respectively. The edible films based on quinoa protein, starch NPs, crude phenolic compound extracts (luria leaves/pomegranate peels) also provide enhanced packaging attributes with a safe packaging system for improved food product life [58]. Furthermore, the composite film based on banana starch, aloe vera, curcumin loaded starch NPs provides improved water vapor permeability and tensile strength due to the hydrophobic nature of curcumin [59]. Additionally, the composite film delivers control release profile of curcumin, which provides application in various food stuff. The mung bean-based native starch has smooth surface with oval shape, can be used to fabricate nanostarch using acid hydrolysis methods, having the average particle-size distribution of 141.772 nm. The bio-composite films based on nanostarch from mung bean (concentrations: 0.5, 1, 2, 5 and 10%) and native starch can be used as edible coating in food applications having renewable and biodegradable nature. Further, this specific biocomposite films provide improved film attributes such as (1) Burst strength increases from 943.56 ± 18.1 to 1265 ± 18.9 g; (2) Thickness of films increases from 0.043 ± 0.006 to 0.063 ± 0.006 mm; (3) Water vapor transmission rate (WVTR) decreases from $5.558 \times 10^{-3} \pm 0.25$ to $3.364 \times 10^{-3}\ g^{-2}\ s^{-1}$; and (4) Water solubility decreases from 37.99 ± 0.47 to 34.11 ± 0.40% [60]. The nanocomposite based on thermoplastic starch (glycerol as a plasticizer) and waxy maize starch provides strong interactions between filler materials and filler–matrix materials due to the hydrogen bonding [61]. The fabrication of starch films incorporating vacuum freeze-dried and spray-dried starch NPs provides denser films, enhanced film roughness, where the incorporation of starch NPs lowers the water vapor permeability (by 44%), glass transition temperature (by 4.3 °C) and crystallinity (by 23.5%), Young's modulus and toughness [62]. Additionally, the incorporation of starch NPs in the biocomposite films of starch offers increased storage moduli, loss moduli, and others [63]. Further, in Table 5.3, use of starch-based nanostructured materials in edible food packaging has been detailed for improved features of edible food packaging.

Table 5.3 Use of starch-based nanostructured materials in edible food packaging

Sl No.	Form of nanostarch	Other film components	Materials property	References
1.	STNCs (Fabrication process: Acid hydrolysis at 35 °C from waxy maize starch)	Pullulan Sorbitol (Plasticizers) (biocomposite edible films)	Increase in crystallinity with increased STNC Water uptake decreased with increasing filler content Improved Young's modulus and tensile strength	[55]
2.	Starch-butanetetracarboxylic acid dianhydride-*N*-hydroxysuccinimide (SBN) (a cross-linker)	Gelatin	Improved surface hydrophobicity Improved tensile strength and elongation at break Enhanced shelf life of peeled apple Is applicable for improved quality of postharvest shelf life	[57]

(continued)

Table 5.3 (continued)

Sl No.	Form of nanostarch	Other film components	Materials property	References
3.	Starch NPs	Quinoa protein Crude phenolic compound extracts (luria leaves/ pomegranate peels) (edible films)	Extend shelf life of food Good barrier properties against oxygen, water vapor, and carbon dioxide. Increased strength due to STNPs	[58]
4.	STNCs	Cross-linked cassava starch	• Can be used for preservation of Huangguan pears • Cross-linked cassava starch with 6% STNCs provides better mechanical and water vapor barrier properties • Inhibit peroxidase, polyphenol oxidase enzyme activities of pears.	[56]
5.	STNCs (fabrication process: Acid Hydrolysis of waxy maize starch granules using sulfuric acid)	Carboxymethyl chitosan	• Maximum tensile strength: 29 MPa • Water absorption and water vapor permeability can be improved using STNCs • The percentage elongation at break decreases with increased STNCs	[53]
6.	Starch NPs	Banana starch, aloe vera, curcumin (loaded in starch NPs)	Incorporation of curcumin reduces the water vapor permeability due to hydrophobic nature. Improved tensile strength due to incorporation of curcumin in the composite films.	[59]
7.	Nanostarch (concentrations: 0.5, 1, 2, 5 and 10%) (Source: Mung bean)	Native starch	• Renewable, biodegradable, and edible coating in food application. • Burst strength increases from 943.56 ± 18.1 to 1265 ± 18.9 g	[60]

(continued)

Table 5.3 (continued)

Sl No.	Form of nanostarch	Other film components	Materials property	References
			• Thickness of films increases from 0.043 ± 0.006 to 0.063 ± 0.006 mm • WVTR decreases from $5.558 \times 10^{-3} \pm 0.25$ to 3.364×10^{-3} g^{-2} s^{-1}	
8.	STNCs (fabricated via acid hydrolysis using sulfuric acid)	Lemon peel extract (ultrasonic extract)	Bioactive preservative coating on chicken fillets Improved quality of chicken fillets during 12 days cold storage	[54]

STNCs Starch nanocrystal, *NPs* Nanoparticles

5.10 Conclusion

The starch is extensively obtained from (1) Cereal grain seeds (maize, wheat, rice, sorghum); (2) Roots and tubers (potato, sweet potato, tapioca, arrowroot); and (3) Stems and pith (Sago). The available starchy foods are cereals, pasta, bread, rice, potatoes, beans, chestnuts, etc. In different categories of food preservation techniques and to improve food properties, starch and its various derivatives are also used. Starch NPs are utilized as a nanofiller material to develop biocomposites to modify mechanical and barrier properties. The STNCs are used in developing biocomposite films due to the crystalline nature for improved mechanical and barrier property. The use of plasticizers can improve the mechanical properties of starch; however, the hydrophilic nature of starch makes it sensitive toward moisture. In this regard, several modifications of starch-based films are required to act as a proper food packaging system.

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Chapter 6
Protein-Based Nanostructured Materials in Edible Food Packaging

Naba Kumar Kalita, Tabli Ghosh, and Vimal Katiyar

6.1 Introduction

Proteins are the building blocks of life and have received a great interest in versatile food-based applications for having a remarkable nutritional property, biocompatibility, non-toxic nature, and others. Further, proteins offer improved food texture when used as an ingredient in food product development, and is extensively used in the forms of macro- to nanoscales in foovd sectors [1]. Additionally, the protein-based nanostructured materials have attained a great deal of interest in developing edible and non-edible food packaging materials and to be used as a delivery system for bioactive components. Proteins play an integral role as a macronutrient and in the structural formation of foods through several functional properties such as emulsification, foaming, gelation, dough formation, and water-holding property. As shown in Fig. 6.1, the protein-based food products have many benefits including increased weight loss, muscle development, increased calorie burn, controlled sugar levels, prevent heart-related diseases, makes healthy skin, help in hormone balance, prevent hair damage, etc. These properties make proteins as one of the potential candidates for several food sectors including edible packaging applications in the form of edible films and coatings. Nowadays, modern food technology generates new food structures having characteristic attributes that consumer demands and further has the ability to soothe the consumer at first sight. In this process, there is a liberty of using only a limited range of ingredients, due to various health factors and protocols labeled by the standard organizations [2, 3]. As discussed, the proteins are considered as one of the available classes of macro-

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Fig. 6.1 Features of protein in food sectors

molecules that provide desired textural attributes to food products with tailored-made properties such as stability, gelling property, emulsifying property, and antibacterial property. Further, its cross-linking and aggregation properties are considered as the most important properties that enable engineering of food structures with desired mechanical properties. Interestingly, the protein cross-linking influences many other properties such as texture, viscosity, solubility, emulsification, and gelling properties [4]. The functionalization of protein nanostructured with aided cross-linking agents, plasticizers, etc., can further improve the packaging properties to obtain tunable edible packaging properties. Interestingly, protein-based materials are also widely utilized as matrix materials for incorporating various nanofillers to formulate biocomposites-based edible food packaging as proteins have the status of generally recognized as safe [5]. In this regard, the chapter is focused to discuss the use of protein nanostructured materials in edible food packaging for obtaining improved food properties. Moreover, the several strategies to modify protein are also discussed in the current chapter.

In today's world, the involvement of protein nanostructured materials-based food packaging plays an important role in food preservation, which ultimately leads to food security. On the other hand, the petrochemical-based plastic packaging creates health issues due to the migration of plastic intermediates in the food and creates a lot of wastes in our homes which end up in landfills [6]. These factors prompted researchers to contribute toward the expansion of new processing technologies such as protein-based edible food packaging for food products for enhanced shelf life and improved health benefits. As discussed in previous chapters, polymers from natural resources play an important role in edible-coatings and packaging systems [7]. However, the edible polymers are comprised of any of the four categories, namely hydrocolloids; polypeptides; lipids; synthetic and composite edible polymers. Among them, polypeptide (protein)-based edible polymer can be used for the packaging of food such as nuts and beans [8]. They can serve as carriers for antimicrobial and antioxidant agents in the body. Protein-based edible packaging systems can also serve as multilayer food coater [9], which might have high mechanical as well as functional properties. Proteins have good oxygen barrier properties, however, are not completely hydrophobic and may sometimes create a mess in high humid conditions by limiting their moisture barrier properties [10]. These limitations can be overcome by tailoring the packaging properties with the aid of other agents. Moreover, the tailoring of the component is necessary for the prevention of deterioration and migration in various foods such as burgers, pizzas, and pies. In this regard, the proteins-based edible films also function as antimicrobial agents and help in supplying the antioxidant agents. These kinds of protein-based films are generally formed from solutions or dispersions through solvent/carrier evaporation, where the solvent is generally limited to water, ethanol, and their mixtures thereof. In developing edible films, protein chains are generally bonded by covalent bonds, ionic bonds, and through hydrophobic bonds. These interactions are stronger, however, are not flexible with excellent barrier properties [3], [11].

Based on the above discussion, the current chapter is related to nanostructured protein, protein biocomposite-based edible food packaging, and its related application domain for improved product shelf life. This chapter also provides details of the specific food preservation techniques and the role of protein in improved property of food products, sources of proteins (animal sources and plant sources), and theirs nanostructured forms to be used as an ingredient in the fabrication of edible food packaging.

6.2 Food Preservation Techniques in Improved Product Life

From very early days, various food preservation methods are used for food security. Among available methods, drying is one of the oldest food preservation methods, which reduces the water activity, and the weight of the food products. In drying-based preservation technique, there is obtained a reduced water activity, which inhibits the bacterial growth enhancing shelf life [12]. Pickling is another process of preserving food by anaerobic fermentation providing extended shelf life of food products with a salty or sour taste. In pickling method, the most unique property is to attain a pH of less than 4.6, which inhibit the growth of most kinds of microbes. Canning is another method for preserving food, which involves the preservation of raw or cooked food under sterile conditions. Sterilization helps in killing various microbes, which enhances the shelf life of the food products. Additionally, the modern world's most famous technique of food preservation is freezing and refrigeration. Refrigeration helps in enhancing the shelf life of food, where growth of bacteria becomes slower with increased shelf life of foods by weeks. In today's world of ready-to-eat food, vacuum packaging is widely used for manufacturing foods in industry both on a large and small scale. Vacuum packaging is generally done for storing food in airtight bag and bottle. It generally cuts off the supply line of oxygen from the package that is needed for the survival of microbes. This technique is very much appropriate for keeping dried foods and other foodstuffs for months [3, 12, 13].

6.3 Use of Protein in Edible Food Packaging

Apart from all the discussed methods of food preservation, edible food packaging is one of the recent attractions, being used as a major preserving technique with improved shelf life and functional properties. Protein-based films are mostly used because they have excellent oxygen barrier properties and most importantly they are edible. In this way, it also reduces plastic pollution caused by food wrappers, films, and strips. There are various types of protein-based edible films, which are widely studied and commercialized in recent times. Among them, collagen is used to make the most popular edible packaging films which are commercially available around the world. Collagen films are biocompatible and have various immunological properties. Similarly, gelatin, a hydrocolloid, has a unique sequence of amino acids such as high content of proline, hydroxyproline, and glycine. It is mostly used for encapsulating oil-based food stuffs, as packaging materials and in many pharmaceutical drugs. Zein is another corn-based protein, having excellent film-forming properties, where the film matrix containing zein chains form hydrogen bonds. Zein has better barrier properties and is thermoplastic in nature. Wheat protein is another class of proteins, which makes excellent edible films. The high cohesiveness and elasticity of wheat protein offer a better film formation capacity, however, have a relatively higher water vapor barrier properties. Another important type of protein-based film is soy protein films. Soy protein is abundant in nature and is very nutritional. They form edible films due to their hydrophobic interactions, which increases yield point and tensile strength offering a good candidate for the edible food packaging. Another prominent protein is casein, which is obtained from milk. It comprises about 80% of the milk proteins and on acidification and neutralization, forms an edible film which is used to protect dried fruit and vegetables for reduced moisture absorption [4, 9, 11, 14, 15]. Therefore, protein-based edible packaging films are extensively developed for increased shelf life of food and also help in improving the nutritional qualities of the foodstuffs.

6.4 Sources of Proteins

Proteins are obtained from both plants and animals sources [2, 16]. In this regard, Fig. 6.2 represents some of the available plants and animal-based protein resources. Recent studies suggest that trends of protein intake are changing worldwide according to the needs and diseased conditions. The nutritional and functional properties of proteins have been studied thoroughly for many years. This quality has prompted researchers to study amino acid content of various proteins and their health benefits. These benefits depend upon the source of the proteins such as animal source or plant-based sources. Recent trends and research activities suggest that there is an increased production of plant proteins in order to replace animal-based proteins. This trend has been increased due to the recent reports about the increased production of greenhouse gases from animal husbandry and use of excess water impacts the sustainability of these resources.

6.5 Plant-Based Protein Sources

Various kinds of proteins are extracted from plant sources and are extensively available at a commercial scale. Additionally, the production of animal proteins requires 100 times more water than producing same amount of plant protein. These limitations make plant-based proteins an ultimate solution for protein security [2, 15].

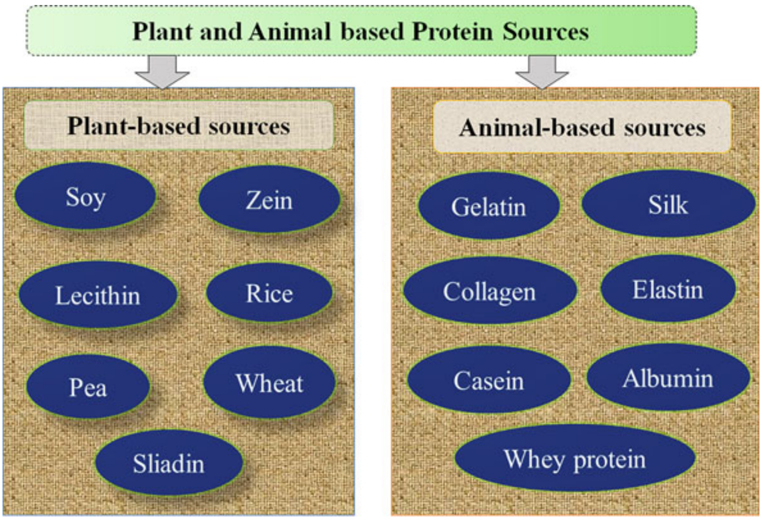

Fig. 6.2 Available sources of proteins

6.5.1 Soy Protein

Soybean contains very high protein due to which it is recognized as one of the most important seed oil crops and as a protein-based food source across the world. It is one of the most used sources for producing vegetable oil after palm oil. After oil extraction, the defatted soybean meal is used as animal feed in many countries, of which some part is also used for protein production for human consumption. Although soybean-based foods are already in use in many parts of Asia from hundreds of years, recent trend of consuming soy-based proteins in the form of milk has increased its market value as well as help in maintaining food security in many parts of the world. Different kinds of soy proteins are available around the world including soy hydrolysates, soy proteins, and soy flour. Nowadays, textured soy proteins are produced, which resembles like meat products. Perhaps, the increased uptake of soy proteins and soy flour is largely due to the intensive research and public recognition of the potential health benefits, such as alleviation of chronic diseases. Application of soy protein is found in various areas such as inexpensive protein rich foods, and edible films which make it as one of the most potential candidates among the available protein-based food sources from plants or land-based [3, 17, 18]. Recent research trends also suggest that soy proteins are used for developing edible protein films. Soy proteins forms good films due to their capacity to get polymerize and are also highly biodegradable. Moreover, they contain both polar and nonpolar side chains, which form strong intra- and intermolecular interactions. These forces restrict molecular mobility within the molecules, which provide increased stiffness.

6.5.2 Wheat

The most abundantly cultivated crop across the world is wheat and is consumed by more than billions of people as a primary food in various processed form. Wheat contains around 9–15% protein, which marginally depends upon grain type. Gluten is the main protein present in wheat and has some unique physical properties. Gluten has some unique functional properties and forms cohesive and proteinaceous network that helps in the production of leavened food stuffs from wheat. Further, simple water wash helps in gluten separation from wheat proteins and 70–80% of gluten can be separated from wheat. Interestingly, the major uses of gluten involve in producing pasta, noodles, and cereals. In these types of products, protein concentration plays an important role in maintaining the texture of the products. Nowadays, vegetarian meat products are produced using texturized wheat proteins [2, 19–21].

6.5.3 Rice, Maize and Barley

Rice is the most commonly consumed staple food in Asia, and further, most of the South Asian countries consume rice-based food products. However, the protein content in rice is around 5–9%, which is less than wheat. The rice proteins are used as animal feed. Sometimes, rice powder is also used as gluten free food stuffs [15]. Maize is one of the important agricultural and industrial crops. Corn or maize contains around 9–15% protein content. Corn flour is another product of corn, which is used in variety of foodstuff. Corn gluten meal is used for producing zein protein, which is another very common source of land proteins. Major applications of zein are producing materials for film and coatings. Barley is another major crop historically. Nowadays, it has been the primary source for animal feed and malting processes. However, barley contains around 10–17% protein, which is higher than rice varieties. It is used in various food stuffs from cereals to soups, and further, β-glucan content in barley helps in controlling blood cholesterol [3].

6.6 Classification of Plant Proteins

Plant-based proteins were first extracted and identified on the basis of solubility and extractability in various solvents. The four major fractions of proteins are albumins, prolamins, globulins, and glutenins, which are having different solubility in various types of solvents like polar and nonpolar solvents. These fractions exhibit different functionality and molecular structure with changes in plant species [22]. In this regard, the proximate analysis of different classes of plant proteins is presented in Table 6.1.

6.6.1 Albumins

Albumins are present in small quantities as part of the storage proteins in legumes and oilseeds. They are widely present in sunflower and canola. They are also found in small fractions in pulses. They are globular proteins having molecular weight of 4000–9000 Da. Albumin is soluble in water and coagulates when heated

Table 6.1 Proximate analysis of different classes of proteins from major crop-based plant sources

Plant source	Albumins (%)	Prolamins (%)	Globulins (%)	Glutelins (%)
Soybean	0–5	0.5–1	40–70	16–25
Wheat	6–10	35–40	5–8	40
Rice	2–6	4	12	80
Maize	4	60	4	26
Barley	3–5	35–45	10–20	35–45

6.6.2 Prolamins

Prolamins got its name from presence of high content of proline and glutamine in cereal proteins. Prolamins account for more than 50% of the total storage proteins in cereals. They are not soluble in water or saline solutions and can only be extracted with 60–70% v/v of concentrated aqueous solutions. With scientific advancements, various modifications were made in order to understand the molecular structure and relations among individual proteins. One of the most studied proteins is gluten proteins derived from wheat.

6.6.3 Globulins

Globulins are storage proteins found in the soybeans, peas and are very low in cereal grains. Their concentration varies accordingly (Table 6.1). Globulins represent the amino acids having very low level of sulfur containing amino acids such as cysteine and methionine. Unlike albumins, globulins are soluble in saline solutions.

6.6.4 Glutenins

They are the major storage proteins of rice and constitute about 80% of the total rice protein. They have very high molecular weight and are very much difficult to extract due to their high cross-linking, which are only extractable under certain alkali concentrations.

6.7 Animal-Based Proteins Sources

Athletes around the world mostly prefer proteins from animal sources. This may be due to the completeness of the proteins from the sources (such as meat, fish, poultry, eggs, and milk). Various studies suggest consumptions of animal proteins, which tend to have higher values than plant-based proteins although there are other studies, which suggests that consumption of animal protein in large quantity can cause cardio vascular diseases [8, 15, 16].

6.7.1 Whey-Based Proteins from Bovine Milk

Whey is milk-based protein and is one of the most prominent milk-based proteins consumed worldwide due to its unique functional properties such as rich in vitamins and minerals. In sports nutrition sector, whey protein is the most commercialized and consumed protein worldwide. Whey protein comes in different forms including whey powder and whey concentrate, where both the forms are used as food additives in many foodstuffs. Unlike whey powder, whey concentrate contains more biologically active substances. Whey protein isolate is another protein variety consumed by those who are having the problems of lactose intolerant. However, its protein concentration is highest, but the denaturation leads to loosing of effectiveness of the proteins [3, 9–11].

6.7.2 Casein

Casein is the abundant portion of the bovine milk concentration accounting nearly 80% of the total milk protein. Bovine proteins are the most important proteins for consumption as well for food security as they are the source of essential vitamins, minerals, and biologically active peptides. Casein exists in milk in the form of micelle and is of colloidal origin. Its attractive property is its ability to form gel-like structure, which is also the driving force in the formation of curd or yoghurt from milk. This helps in nutrient supply. This also provides better nitrogen retention in the body as sustainable supply of amino acids in the body is made possible by the gel or clot in the foodstuffs [3, 23] (Table 6.2).

6.8 Extraction of Protein from Available Sources

Protein extraction involves various solvent-based methods, physical and chemical methods or combined methods. In this chapter, some of the most common protein extraction process are shown in Figs. 6.3 and 6.4 for soy protein and wheat protein-based sources. For the extraction of soy protein, soy protein is cracked and dehulled into full fat flakes and solvent is used for the extraction of the defatted flakes. These defatted flakes are grinded into soy flour, which is then mixed and extruded into texturized soy protein. For the production of soy protein concentrate, defatted flakes are mixed with solvent for cell wall removal which yields more than 60% protein. Defatted flakes are solvent dried, and soy protein concentrate is

Table 6.2 Proximate analysis of various whey-based protein forms

Component	Whey isolate (%)	Whey concentrate (%)	Whey powder (%)
Protein	90	25–89	11–14.5
Lactose	0.5	10–55	63–75
Milk Fat	0.5	2–10	1–1.5

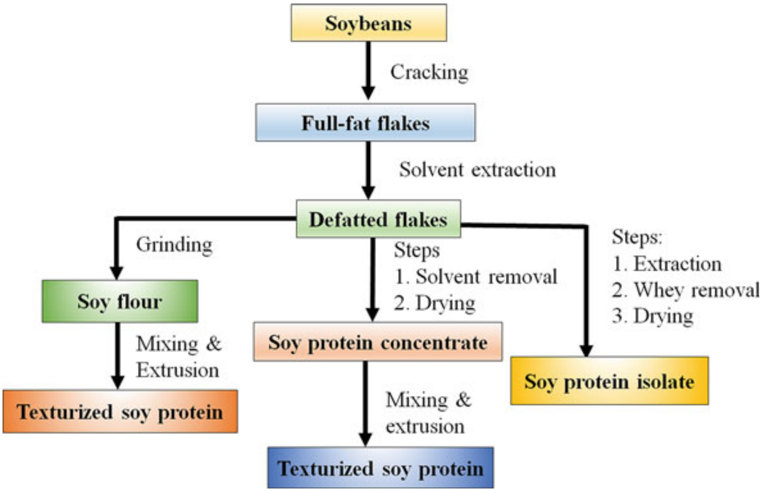

Fig. 6.3 Development of textured soy protein

obtained, which is further mixed and extruded to get texturized soy protein. These kinds of texturized soy protein are mixed in various food stuffs like vegetable meats, sausages, etc. Another major protein is soy protein isolate, which is obtained by alkali extraction, and whey removal followed by drying to get the necessary isolate. They are often replaced with animal proteins [18, 21, 24].

Wheat contains gluten and is the major proteinaceous network, which when washed yields gluten, and contains more than 75% of protein. Wheat flour is consumed around the globe and when mixed with water it forms dough having various rheological properties. The gluten can be obtained by simply washing the dough with water followed by drying. Nowadays, various modifications are done to this process to yield better products. Wheat gluten is then mixed with alkaline solution and dried to obtain the wheat protein isolate. Wheat protein can be extruded further to produce texturized wheat protein. Gluten can also be extruded directly for obtaining the texturized wheat protein. Wheat gluten can hydrolyzed using enzymes like hydrolysates for obtaining hydrolysed wheat gluten. This enzyme treatment also increases functionality and protein content. Gluten is also used as replacement of meat products in vegetarian foods around the world [3, 19, 20].

6.9 Use of Protein in Food Industry

Protein science and engineering has a massive role to play in food and pharmaceutical industry. From supplements to edible food packaging films, proteins have a great contribution in the industry in terms of sustainability as well as food security. Protein like insulin was the first protein, which was sequenced. Protein application

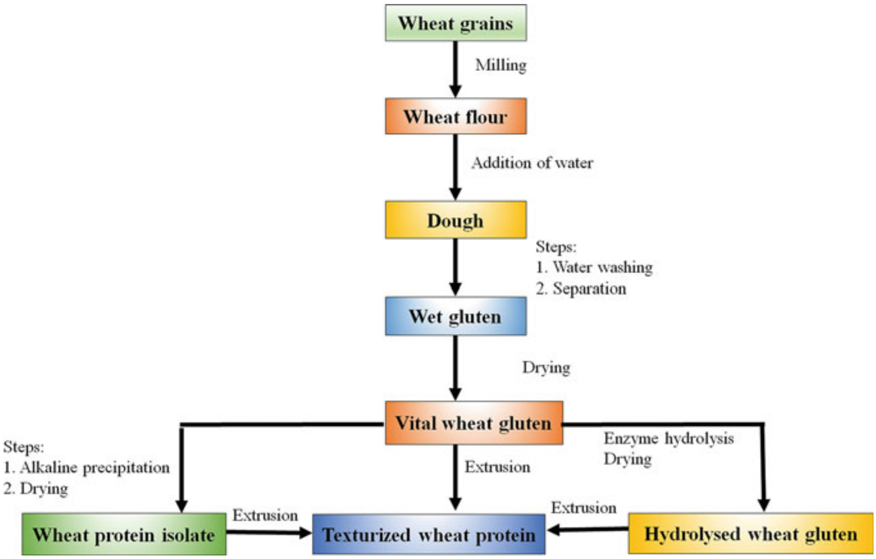

Fig. 6.4 Development of textured wheat protein

in food industry has roles in nutrition security, functional properties by application in food packaging, supplements, etc. In food industry, protein processing or processing of protein-based food stuffs is based upon its structure and chemistry. Different food-based proteins or proteins in food industry needs different processing treatments like thermal analysis and enzymatic hydrolysis. [7, 25].

6.9.1 Effect of Industrial Processing on Food Proteins

Industrial processing techniques such as thermal processing or non-thermal-based techniques directly affect the protein functionality. However, the current foods industry mainly focus on consumer demand and sensory elements. Further, the various elements of food processing affect the functionality of food products starting from enzymatic browning to loss of enzymatic activity and nutritive value such as development of off-flavors. These properties are altered through processing techniques, from thermal, non-thermal, biological, or chemical processing of foodstuffs in food industry. Processing also helps in forming structure and to rapture structure accordingly to support nutritional benefits.

6.9.2 Thermal Processing of Proteins

The thermal processing technique is the world's oldest technology used at home and food industry. Thermal processing helps in increasing shelf life of foods through reduction in microbial load and food safety. In industry, it also helps in improving the sensory properties of food. It helps in cleavage of disulfide bonds, protein oligomer formation, and aggregation. Thermal treatment is provided to proteins in order to modify functionality, stability, and formation of emulations in foodstuffs [25–27]. Interestingly, thermal treatment is required for denaturing the protein, which helps in providing the tailored-made structures in order to form the films.

6.9.3 Non-thermal Processing of Proteins

In industry, non-thermal processing is becoming an alternative for thermal processing. Among the available non-thermal processing of proteins, fermentation and biocatalysis, and high-pressure processing are commonly used methods in food industries. Ultrasound is also used for cell rapture and protein size reduction, which increases the functional properties of the protein extracts. Ultrafiltration, electrodialysis, and reverse osmosis are another emerging technologies, which increases functionality of food-based proteins [3, 25].

Treating food proteins with appropriate processes or their combinations can provide the desirable nutritional, functional, and sensory properties of food products. Therefore, understanding the molecular basis of the functionality is important in determining the specific structural transitions that occur in food proteins during processing. The knowledge of the factors and influence of the processes are critical to controlling and meeting the increasing consumer and market demands for novel physical and sensory attributes. The significance of the modifications is dependent on nature of the proteins, processing methods, food matrix, and process-induced alterations achieved. Therefore, further research should capitalize on the limitless opportunities offered by this diversity in generating new protein functionalities for improved and novel applications in food formulations.

6.10 Traits of Proteins in Edible Food Packaging

The formation of edible films from proteins depends upon the surface film formation method and the deposition method. Various factors affect the protein-based edible films such as raw materials, polymer chemistry, pH, drying method, concentration, relative humidity, and film additives. In this regard, various kinds of edible films and coatings have been successfully developed from whey proteins, caseins, zeins, and others; and the materials have the ability to serve other functions, viz. carrier of antimicrobials, antioxidants, or other nutraceuticals, without significantly compromising the desirable primary barrier and mechanical properties as packaging films. Besides, it provides additional values for eventual commercial applications. The advancement in whey protein production increased significantly due to the improvement in membrane and ion exchange technologies. Various researcheres have used structuring agents, plasticizers, fillers, and others for betterment of film properties such as reduced brittleness with the aid of plasticizers. Polysaccharides and lipids are used for improvement of barrier properties. Further, a research reports the development of protein layer having excellent barrier properties and is comparable to ethylene vinyl alcohol (EVOH) copolymers barrier layer, which is conventionally used in food packaging, with an oxygen barrier (OTR) of <2 [cc/m^2/24 h] when normalized to a thickness of 100 μm [10]. Further, reports are also available, where rosemary, oregano, and garlic essential oils are incorporated to whey proteins in order to boost antimicrobial activity of whey-based edible films [11]. Edible soy proteins and poly(lactic acid)-based bilayer films are also prepared for active food packaging. In this type of films, the layers are incorporated with natamycin and thymol, which impart the properties of antifungal and antibacterial films and the films showed inhibition of molds, yeast, and some of the bacteria [17].

6.11 Extraction and Importance of Protein Nanoparticles

The development of various protein-based nanostructured materials as available in the forms of nanoparticles, nanohydrgels and nanoemulsions can be obtained using various techniques such as gelation mechanisms (denaturation of globular protein, thermally induced gelation, acid-induced gelation, ionic-gelation, enzymatic-gelation), water-in-oil heterogeneous gelation (inverse emulsion method, reverse micellar method, membrane emulsification, etc.), and others such as electrospray, physical self-assembly, associative separation, and spray-drying as shown in Fig. 6.5. In this regard, some of the available techniques for developing protein nanoparticles are discussed in the below sections.

6.11.1 Desolvation

In 1978, the desolvation process is developed by Marty and co-workers. The process involves the use of desolvation agents such as salts and alcohol in the proteins. After addition of the desolvation agents, proteins molecules start to change its shape slowly. This helps in the formation of proteins clumps eventually forming protein nanoparticles, due to cross-linking mechanisms. Additionally, the separation of the nanoparticles is obtained by increasing the turbidity of the solution [28].

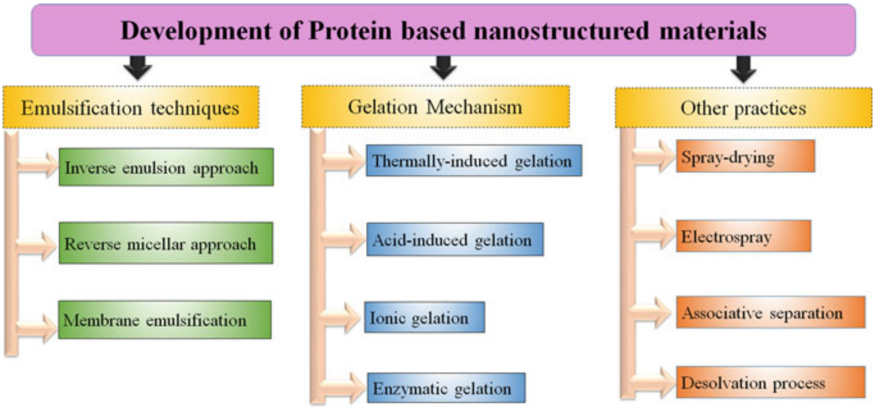

Fig. 6.5 Several extraction processes for protein nanoparticles

6.11.2 *Emulsification*

Emulsions are a dispersion of two or more immiscible liquids in which one of the liquids is dispersed in another liquid as droplets of various sizes (0.1–100 μm). Interestingly, the amphiphilic nature of proteins allows them to reduce the interfacial tension at the surface. Further, a research reported the incorporation of protein particles in the oil–water interface for utilization in drug delivery to the food industry. The emulsions are formed in the presence of emulsifiers, which comprise both hydrophobic and hydrophilic components [29].

6.11.3 *Electrospray Drying*

Electrospray is another technique used to produce protein nanoparticles. In this method, high voltage is applied to the protein solution, which emits a liquid jet stream. Further, the jet stream passes through a nozzle, which helps to form an aerosolized size liquid consisting of drug and nucleic acid. This technique is generally used for developing gliadin and elastin peptide nanoparticles [30].

6.11.4 *Nanospray Drying*

From very early days, nanospray drying is basically used for developing nanoencapsulates of protein concentrates or other protein materials. It is also considered as one of the cheapest technologies available for developing protein-based encapsulates, which has received a great interest in food- and protein-based industries.

Nanospray drying involves the use of spray drying technique, where the protein materials are dispersed in a carrier polymer solution and further, atomized into droplets of nanometer size range in the spray drier. This technique generally involves the use of aqueous solution, which can be a green alternative for organic solvents in developing protein nanostructured materials [30, 31].

6.12 Modification of Proteins and Related Nanoparticles for Edible Food Packaging

The various strategies for protein modifications have been represented in Fig. 6.6. The protein and its nanoforms are modified in terms of improved physical (dissociation, solubility, optical activity, emulsifying effect, foam stabilization), chemical, and biochemical properties to be used in developing edible films and coatings. However, the use of several proteinaceous materials and their function depends on the protein extraction process, digestibility, nutrition value, functional properties, organoleptic properties, etc. In food systems, protein modification is carried out for increased protein stability, improved texture and other attributes. The protein modification via chemical modification includes the addition of chemical groups such as hydroxylation, phosphorylation, methylation, acetylation. The amino acid modifications for protein and nanoform include deamidation (removal or conversion of amide functional group in the side chains of asparagine and glutamine), and eliminylation. On the other hand, protein modification via addition of complex molecules are glycosylation, prenylation (addition of hydrophobic molecules), PEGylation. Further, proteolysis (cleavage irreversibly) is another approach for protein modification. In this regard, the denaturation of whey proteins offers nanostructures of whey protein for improved functionality. The potential of protein nanostructures has several benefits such as enhanced stability, improved food functionality in terms of physical, biological, and chemical functionality. Whey protein nanostructures are used as a delivery agent for transferring nutraceuticals to food products [32]. In this regard, several approaches for protein modifications in regards to targeted applications have been discussed in the below section.

6.12.1 Chemical Modification

The chemical modifications of proteins and related nanoparticles (NPs) include alkylation, acylation, acetylation, esterification, glycosylation, deamidation, PEGylation, thiolation, cross-linking, and lipophilization. Additionally, the chemical modifications of proteins mainly involve the use of chemical agents or pH-driven modifications. The alkylation-based protein modification occurs at the protein side

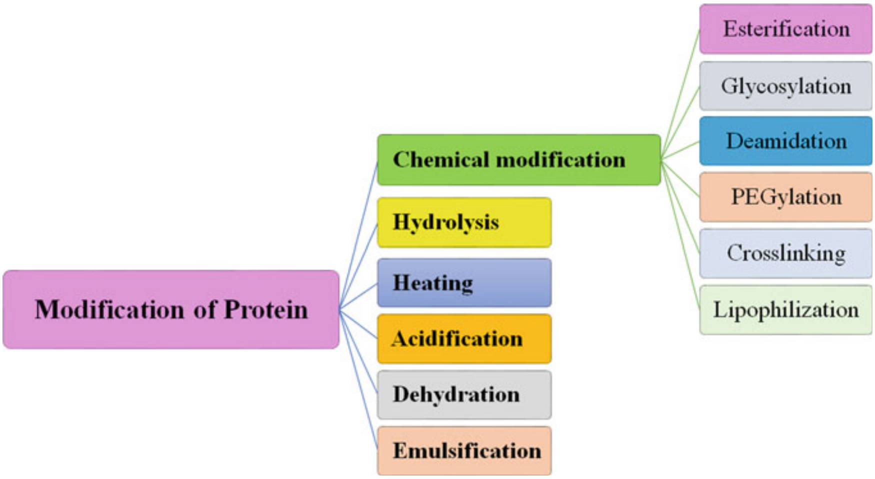

Fig. 6.6 Available strategies for protein modification

chains such as amino acids. The deamidated proteins are obtained as by-products from protein-based food processing, which are obtained by acid, alkaline, and enzymatic treatment. Interestingly, deamidation of proteins influences the protein functionality in terms of foaming property, solubility, emulsifying property, etc. PEGylation is another approach to modify protein, where chains of polyethylene glycol are attached to protein molecules through covalent linking. This chemical modification approach further helps in the delivery of bioactive molecules. The chemical cross-linking of proteins is obtained by using several chemical agents such as formaldehyde, glutaraldehyde, α-hydroxyadipaldehyde, and others. The protein cross-linking using glutaraldehyde provides chemically and thermally stable cross-link molecules. However, the use of glutaraldehyde cross-linked protein has not been done in food sectors for the toxicity levels. The chemically modification of protein using lipophilization approach includes the esterification of lipophilic moiety on proteins. The lipophilization of proteins provides many significant benefits such as improved hydrophobicity, altered surface property, and improved water barrier property. Additionally, the increased hydrophobicity of protein molecules further helps to develop stable protein-based foams and emulsions, which help in providing improved interactions with hydrophobic substances. The widely used fatty acids (saturated and unsaturated) in lipophilization of proteins are capric acid, lauric acid, caproic acid, stearic acid, and others.

6.12.2 Hydrolysis

The protein hydrolysis is an important tool to modify the protein properties including lower hydrophobicity, improved stability, emulsifying property, and viscosity for targeted application in food sectors specifically edible food packaging. The hydrolysis of several types of proteins is carried out via chemical method and enzymatic methods. In enzymatic hydrolysis, the various enzymes targeted for enzyme hydrolysis are obtained from different sources such as (i) Plant sources: bromelain (source: pineapple), papain (source: papaya), ficin (source: fig); (ii) Animal source: pepsin, pancreatic; and (iii) Microbial source: Alcalase. Additionally, enzyme protein hydrolysis technique is one of the widely used techniques in the recovery of amino acids, proteins, and peptides from by-products of food processing units. The proteolytic enzymes act on protein at optimum conditions (temperature and pH), where protein hydrolysis occurs when enzyme cleave on the specific peptide bond. The proteolytic enzymes from animal sources provide more specific action in comparison with plant-based enzymes. In this regard, researchers use several methods to measure the degree of protein hydrolysis, where some of the techniques are time-consuming and low reproducible ability, laborious, etc. However, Fourier transform infrared spectroscopy (FTIR) method is a kind of rapid and advanced technique used for characterizing protein hydrolysates. The underutilized proteins (sources: seafood and fishery by-product) can be functionalized using protein hydrolysis approach. The enzymatic protein hydrolysis method becomes one of the most recommended techniques to functionalize protein and to produce bioactive peptides. Further, the functionalized protein has attracted a great attention to fortify food, nutraceuticals, edible packaging materials, and others. The functional protein products obtained by protein hydrolysis have many health beneficial attributes, high nutritional value, good digestibility, balanced amino acid, etc. Additionally, the enzyme-based protein hydrolysates are used in developing texturized food product, emulsifying property, water retaining capacity, plasticizing protein films, and others. The plasticity and barrier properties of fish myofibrillar protein films can be modified using fish gelatin hydrolysate, where the degree of hydrolysis and protein hydrolysate concentration, type of enzyme used effect the film property. Additionally, the emulsifying property of proteins can be improved by tailoring the overall protein solubility which are obtained through controlled enzymatic hydrolysis.

6.12.3 Heating

The protein thermal aggregations are utilized for developing bio-based edible nanocoating networks, where the aggregation of protein molecules are dependent on several factors such as molecular interactions, and other factors (temperature, protein concentration, ionic strength). The application of thermal treatment can enhance protein functionality, and structure–property relations. The mechanical properties in terms of tensile strength of protein-based films can be improved aiding heat treatment to denature proteins. In food products, bread with softer crumb texture is developed by incorporating heat-treated protein in wheat flours. The gelation and film-forming property of proteins can be improved with the aid of heat treatment, which break non-covalent bonds and form sulfhydryl bonds. Further, thermal treatment to protein products influence emulsion stability and foam stability. Interestingly, the heating of protein may have influential effects on protein modifications and protein digestibility [33].

6.12.4 Acidification

The various degrees of deamidation and surface hydrophobicity can be obtained via acid modification of proteins. Acidification of protein is generally done in food beverages, to improve shelf life, taste and viscosity. The acidification of proteins in food products helps to reduce microbial growth. The acidification of milk is a very important factor in developing yogurt and other protein products. The acidification of casein micelles can provide tailored size, where acidification at pH below 5.5 increases the micelles size, and at pH 5.5, provides reduced micelles size [34]. Additionally, slow acidification and cold acidification of milk can produce acid-induced milk gels. Proteins are widely used in developing edible films and coating, where acidification of protein denature the protein by extending the protein structures required for film formation. The acidification and homogenization of collagen develop protein-based moisture gel, which is needed for developing some protein-based edible films. The addition of citric acid to gelatin-based films can improve the film functionality. The use of fatty acids can improve the barrier properties of protein-based films.

6.12.5 Emulsification

Proteins, consisting of both hydrophobic and hydrophilic group, can be used as an excellent emulsifier. Protein-based emulsifier formulations are obtained in protein-in-oil or oil-in-protein-based systems, which provide various noteworthy properties. The functions of protein emulsifiers are (i) Modify texture, (ii) Stabilize protein emulsion, (iii) Control agglomerates of fat globules, (iv) Form a complex with starch and protein components to provide improved rheological property, (v) Control the polymorphism of fat, which in turn improve the texture of fatty food products, and others. The common protein emulsifiers are milk (casein), cream, egg yolk (lecithin, lipoprotein), mustard, etc. The emulsion stability can be improved by several approaches such as strong interfaces, electrical repulsion, and increased viscosity. The development of emulsion-based films can provide improved attributes of protein-based films such as wheat gluten, whey protein-based films due to incorporating lipids (stearic acid and palmitic acid) and fat materials. The emulsifying attributes of proteins are commonly described as emulsion activity, emulsion capacity, and emulsion stability. The properties and application of dietary protein sources in food formulations can be improved by improving the emulsifying properties. The emulsifying properties can be improved using enzyme hydrolysates such as peanut protein hydrolysates, potato protein hydrolysates, amaranthus protein hydrolysates, fish protein hydrolysates, etc. The foaming and emulsifying properties of proteins are two important attributes to improve the protein functionality in food products including ice-cream, margarine, whipped toppings, mousses, etc. The emulsifying property of proteins, and its hydrolysates are determined by emulsifying stability index and emulsifying activity index. The protein folding-unfolding property and ratio of hydrophobic–hydrophilic ratio influence the emulsification property of protein. Moreover, foam stability, capacity and expansion influence the foaming property of proteins. In this regard, the high protein content and hydrophobicity can provide improved foam stability of proteins. Additionally, the nanoemulsion of proteins has attracted a great deal to develop edible films and coatings for improved shelf life of high perishable food products.

6.13 Nanosystem in Protein-Based Edible Food Packaging

The nanosystem-assisted protein-based edible food packaging include the use of protein-based nanostructured materials such as whey protein isolate nanofibrils (WPNF), zein NPs, silk fibroin, gelatin NPs, egg albumin NPs, casein-silver conjugated NP, and others. However, the inclusion of plasticizers, and compatibilizers with protein-based nanostructured materials can further improve the edible films and coating properties. The noteworthy attributes of protein NPs include non-antigenic property, biodegradability, surface chemistry, surface modifications, and further, controlled dimensions of protein NPs can also be obtained [35]. Additionally, protein-based biocomposites are also used to develop edible food packaging materials and incorporation of nanofiller materials can improve the protein films properties in terms of barrier property, mechanical property, thermal property, and others. In this regard, a detailed discussion related to nanostructured protein-based materials has been done in the below sections.

6.13.1 Protein-Based Nanostructured Materials in Edible Films and Coatings

As detailed in Table 6.3, the inclusion of proteins, its derivatives, and nanostructured form in developing edible food packaging has several beneficial attributes including antimicrobial activity, antioxidant activity, reduced gas exchange rate, improved mechanical property, and others. The nanostructures of whey proteins are used as carrier materials for antimicrobials, nutraceuticals, antioxidants, etc. In this regard, the edible-coating properties of whey protein isolate (WPI) can be modified with the aid of whey protein isolate nanofibrils (WPNF), glycerol, and trehalose.

The edible coating based on WPNF, glycerol, and trehalose holds the ability in keeping quality of food products in terms of product weight loss, browning effect, total phenolic content, and others. Additionally, the use of WPNF can improve the film properties such as increased hydrophobicity, smoothness of film surface, transparency, homogeneity, and others. On the other hand, WPNF is also beneficial in providing decreased moisture content and water solubility. The use of WPNF provides improved antioxidant activities and hydrophobic properties in comparison with WPI [36]. The development of edible coating to salted duck egg yolks (SDEY) using WPNF, glycerol, and carvacrol is helpful in reducing weight loss of SDEY and hardness increase rate (18.22%) [37]. Interestingly, the antibacterial activity (against *Listeria monocytogenes, Staphylococcus aureus, Salmonella enteritidis, Escherichia coli*), smooth surface, and better transmittance can also be obtained for combined use of WPNF, glycerol (plasticizer) and carvacrol (antimicrobial agent) to edible coating. The edible-coating materials consisting WPNF with glycerol and carvacrol has improved antioxidant property in comparison with WPI with glycerol and carvacrol. Additionally, the emulsions of WPNF, glycerol, carvacrol have 2,2-diphenyl-1-picrylhydrazyl (DPPH)-free scavenging activity, and reducing power of 67.89 and 0.821%, respectively. Additionally, the application of edible coating based on WPNF, glycerol, carvacrol on fresh-cut cheddar cheese can reduce the weight loss (15.23%) with improved textural attributes in comparison with uncoated cheddar cheese [38]. The development of edible coating using silk fibroin, water-based coating, on strawberry and banana helps to improve the shelf life and manage postharvest physiology with reduced respiration rate and water evaporation [39]. The application of edible coating on chilled meat using WPNF and titanium dioxide nanotubes (TNTs) can enhance the shelf life with reduced weight loss, and microbial growth [40]. Additionally, TNTs have better antibacterial property and surface interaction with WPNF in comparison with titanium dioxide NPs. The combined use of WPNF and TNTs provides smooth surface with improved antioxidant, antimicrobial property, and when used as an edible coating on chilled meat provide improved lipid peroxidation.

The use of aqueous dispersion of zein NPs in developing edible water barrier films provide improved properties, where the size of NPs influences the optical, morphology, and other property [41]. Resveratrol has many benefits, however, it has low stability which reduce its bioavailability. However, the encapsulation of resveratrol can increase the bioavailability of stable bioactive compounds. The encapsulated bioactive components can be incorporated in edible packaging system for improved packaging and food properties. In this regard, the loading of Resveratrol within zein nanofibers (Fiber diameter: 404 nm and ribbon like morphology) can be obtained using electrospinning technique having encapsulation efficiency and radical scavenging activity of 96.9 and 66.2%, respectively [42]. The use of nanostructured edible coating on apple slices (experiment time: 6 h) can improve the food properties in terms of controlled moisture loss and maintained color property. The biodegradable films based on encapsulated curcumin loaded caseinate/zein NPs provide tailored packaging properties in terms of tensile strength and water vapor barrier properties [43]. The tensile strength of curcumin loaded

Table 6.3 Protein-based nanostructured materials for edible food packaging

Sl No	Nanoprotein	Other components	Target	Reference
1.	WPNF, Glycerol Trehalose	Edible coating Low cost Biocompatible	Retard product weight loss Browning inhibition Improved film attributes	[36]
2.	WPNF Glycerol Carvacrol	Edible coating of salted duck egg yolks (SDEY)	Reduced weight loss of SDEY Reduced hardness increase rate Provide antibacterial activity	[37]
3.	WPNF Glycerol Carvacrol	Edible coating on fresh-cut cheddar cheese	Provide antioxidant property Provide Antimicrobial property Reduced weight loss (15.23%) Improved texture property	[38]
4.	Silk fibroin	Edible coating on strawberry and banana	Provide enhanced shelf life at room temperature Use of naturally derived materials as a replacement to food preservation technique	[39]
5.	Whey protein nanofibrils Titanium dioxide nanotubes (TNTs)	Edible coating on chilled meat	Improve Antioxidant activity Reduce microbial growth Improved shelf life of chilled meat Improve lipid peroxidation High antimicrobial activity	[40]
6.	Zein Zein NPs	Edible water barrier films	Size of NPs influence the optical property of film Size of NPs influence morphology of film	[41]
7.	Resveratrol loaded electrospun zein nanofibers	Nanocoating on apple slices	Enhanced bioaccessibility of resveratrol Controlled moisture loss of apple slices Color retention in apple slices	[42]

(continued)

Table 6.3 (continued)

Sl No	Nanoprotein	Other components	Target	Reference
8.	Ag and casein NPs incorporated pectin/ sodium alginate/ casein-based bilayer films	Edible films for almond oils	Improved oxidative stability of almond oils Increased peroxide value from 8.02 to 11.5 meq/kg	[44]
9.	Casein Casein-silver conjugated NP	**Edible bilayer pouch** I. NPs (silver) aided casein film (inner layer) II. Sodium alginate and pectin film (outer layer)	Improved water barrier property Improved light barrier Improved mechanical property Improved thermal property Antibacterial property (tested against *E. coli*)	[45]

WPNF Whey protein isolate nanofibrils *WPI* Whey protein isolate *SDEY* Salted duck egg yolks; *NP* Nanoparticle *TNTs*: Titanium dioxide nanotubes)

caseinate/zein NPs films improves from 3.12 to 7.85 MPa, whereas the water vapor permeability of films increases from 0.87 to 3.56×10^{-10} g Pa m^{-1} s^{-1} due to increased proportion of zein in the developed films. The casein NPs (Method of preparation: desolvation technique) and AgNPs (Method of preparation: Mediation of casein protein) are used to prepare pectin-sodium alginate/casein-based bilayer edible films for storage of almond oils [44]. Further, the use of specified edible bilayer films in storing almond oils provides improved oxidative stability during storage. Additionally, the edible bilayer films consisting of two layers (i) Inner layer: Nanocomposite film containing casein-Ag conjugated NPs in casein film, and (ii) Outer layer: Composite films of pectin and sodium alginate developed on the inner layer of the films (casein film) deliver noteworthy film properties in terms of mechanical, light barrier, water vapor permeability, and antibacterial property [45].

6.14 Conclusion

Protein and its nanostructured materials are extensively used in developing edible films and coatings to replace the available synthetic film materials. The protein-based edible packaging provide several beneficial attributes such as eatable in nature, and impart multifunctional properties. In addition, protein-based films also provide impressive gas barrier properties, when nanoemulsion or lipid-based systems are added in comparison with neat protein films. The various features of proteins NPs include body hormonal balance, weight loss, and muscle gain. Furthermore, the use of protein NPs in developing edible films and coatings provides several packaging attributes in terms of antibacterial property, packaging attributes such as barrier property, mechanical property, thermal property, and multifunctional properties. Thus, the packaging attributes of protein NPs can improve functionality as well as material viability of highly perishable food products.

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Chapter 7
Lipid Nanoparticles for Edible Food Packaging

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7.1 Introduction

The inclusion of lipid nanostructured materials in edible food packaging is one of the significant approaches to develop tailored-made packaging properties and further can improve the food properties during storage life. However, the several lipid nanostructured systems such as nanoemulsions, solid–lipid nanoparticles (SLNs), nanolipid carriers (NLCs), nanoliposomes, and others are used in various food systems and pharmaceuticals in a commercial scale for improved features. The various sensitive bioactive components such as antioxidants, antimicrobials, proteins, anticancer agents, probiotics, and others can be delivered to different food system using lipid-based nanostructured materials. In this regard, the use of lipid-based nanostructured materials can improve the functionality, stability, solubility, cellular uptake, bioavailability, controlled release, and other properties of different bioactive compounds in food products and human body when ingested. However, from very early days, the several lipid-based materials for the development of edible food packaging such as edible coating and edible films are waxes, lacs, fatty acids and alcohols, acetylated glycerides, and others. The lipid materials are a good candidate for reducing the water vapor permeability and it influences the property in edible films and coatings. The commonly used lipid materials for improved properties of biopolymeric materials include polysaccharides and proteins in a complex form. However, the characteristics attributes of lipid-based films for developing edible films and coatings depend on various factors including interaction between lipid and other materials, chemical arrangements, physical state, structure, hydrophobicity, and others. In comparison to the lipid materials, the incorporation of lipid nanomaterial-assisted edible food packaging has a better

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effectivity in protecting the food products from environmental damages. The various lipid nanostructures which are used in food systems or edible films or edible coatings in the form of core–shell nanoparticles (NPs) such as NPs filled droplets, NPs filled hydrogels, NP clusters, NP colloidomes, SLNs, nanolaminated NPs, NLCs, and others. The common processing steps in fabricating lipid nanostructure-based edible food packaging as shown in Fig. 7.1 involve different steps such as (i) selection and composition of lipid nanomaterials (nanoemulsions, SLNs, NLCs, nanoliposomes, and others); (ii) characterization of lipid nanostructures; (iii) incorporation in polymer supports; and (iv) used as edible food packaging (edible films/coatings). In this regard, the available techniques to develop lipid-based nanostructured materials are high energy methods and low energy methods. The lipid materials in different aqueous systems are processed to develop nanomaterials to improve the food properties and packaging materials. The nanoemulsion provides transparency, whereas microscale emulsions are opaque in nature, where the use of ultrasound can develop fine emulsions from coarse emulsions. In emulsion-based edible films and coatings, the type of lipid, volume fraction, and drying conditions influence the property of films. In this regard, the current chapter introduces the commonly used different lipid NPs for multifaceted applications. The chapter also focuses to discuss the various crucial aspects of lipid nanomaterials in edible food packaging. The application of lipid nanostructured materials such as nanoemulsions, solid–lipid NPs, nanoliposomes, and others in edible food packaging will also be discussed elaborately.

7.2 Aspects of Lipid-Based Materials in Edible Food Packaging

The lipid and its available nanostructured forms have hydrophobic natures and can limit the moisture migration property in edible films and coatings. The apolar nature of lipids improves the hydrophilicity of biopolymers such as carbohydrate, proteins, and others. The crucial aspects of lipid-based materials in biocomposite-based edible packaging are nature of lipid, structure, hydrophobicity, physical state, chemical arrangement, and interactive properties with the biocomposite materials. The polysaccharides and protein-based edible films and coatings can provide good oxygen barrier properties; however, the hydrophilic nature may increase the water vapor barrier properties. However, the addition of lipids may produce inflexible and opaque films [1]. Further, the addition of lipids has the main significance of delivering good compatibility to other film materials such as matrix materials. The lipid-based hydrophobic edible film or edible coating-based materials include waxes, lacs, fatty acids, alcohols, acetylated glycerides, cocoa-based compounds and their derivatives. The emulsifiers or surface-active agents for developing edible films and coatings include lecithin, monoglycerides, diglycerides, monoglycerides esters, diglycerides esters, fatty sucrose esters, fatty alcohols, fatty acids, etc. The

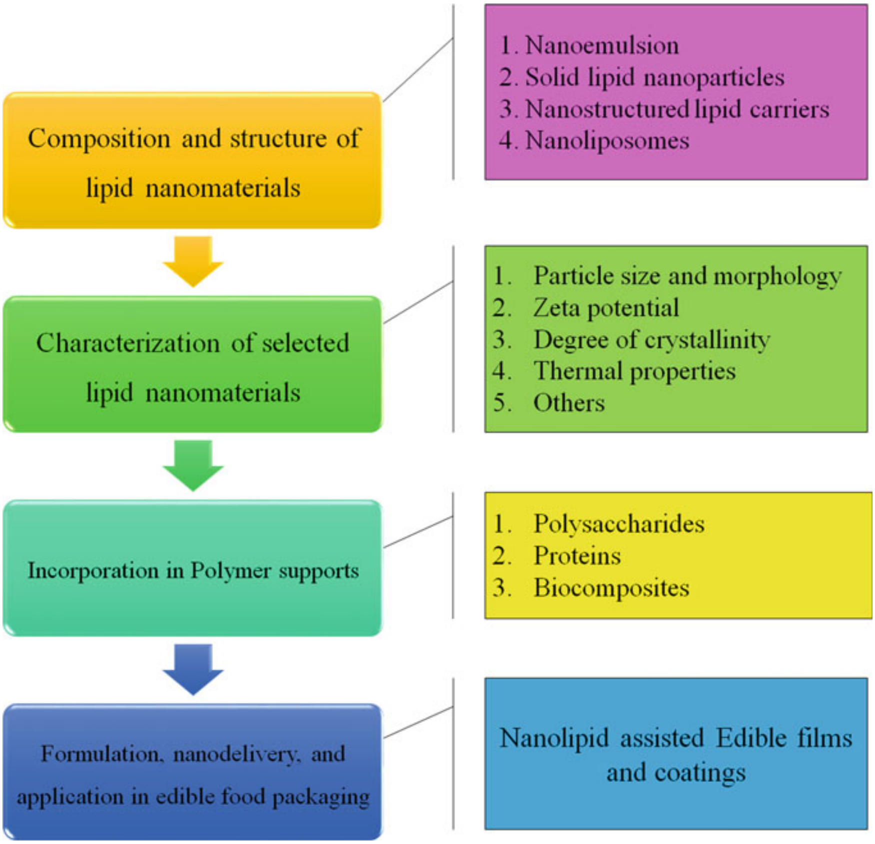

Fig. 7.1 Processing steps in fabricating lipid nanostructure-based edible food packaging

emulsion films consist of lipid droplets in a dispersed phase within hydrophilic phase. However, a detailed discussion on the available lipid and its associated nanomaterials has been discussed later.

Additionally, the structured lipids are considered as a type of functional lipid which is obtained by modifying the fatty acids in the glycerol backbone via chemically, enzymatically, and genetically modification techniques. The various structured lipids are omega-3 lipids, trans-free fats, fat substitutes, phenolipids, salad and frying oils, *trans*-free fats, human milk fat analogues, terpenes, and phytosterol esters, and others. The structural lipids provide the applications in infant formula, nutraceutical lipids, low saturated fats, emulsion and emulsifiers, edible films, and others. In food applications, the structured lipids have the main aspects in (i) processing conditions such as applied shear, crystallization temperature, addition of other components, and other processing technology; (ii) health aspects such as nutritional compositions of structured lipids, and digestion behaviors; (iii) physio-chemical properties: Crystallization behavior, melting behavior, rheological property, interfacial property, oxidative stability, solid fat content, and others; (iv) chemical structures such as fatty acids profiles, acylglycerol composition, and others [2].

Additionally, in edible films and coatings, the various structure of lipids in water are micelle, inverted micelles, bilayer, bilayer vesicle, etc. The lipid structure, chemical properties, surface hydrophobicity, and physical state influence the characteristics features of both edible and non-edible packaging. As mentioned earlier, the inclusion of lipid based nanomaterials as a component of edible food packaging provides an effective barrier against moisture, which in turn helps in maintaining physicochemical and microbial degradation.

7.3 Lipid-Based Materials in Edible Food Packaging

From very early days, the available lipid-based materials for developing edible food packaging are synthetic and natural waxes, fatty acids and alcohols, acetylated glycerides, essential oils (EOs) and extracts, cocoa-based compounds and the derivatives, which has been briefly discussed in this chapter. The hydrophobic components such as antioxidants, antimicrobials, vitamins, and others can be dispersed in developing edible films and coatings which are used to develop lipid in water emulsion. As shown in Fig. 7.2, the various lipid materials in edible food packaging are classified as simple lipids, complex/compound lipids, derived lipids, neutral lipids, miscellaneous lipids. The simple lipids include (i) Fats and oils: Esters of fatty acids and glycerol and (ii) Waxes: Esters of fatty acids and alcohols other than glycerol. The complex/compound lipids include (i) Phospholipids: Consists of fatty acids, alcohol and phosphoric acids; (ii) Glycolipids: Composed of fatty acids, alcohol, and carbohydrate as nitrogenous base; and (iii) Lipoprotein: Composed of lipids and proteins. The derived lipids consist of derivatives of hydrolysis of simple and compound lipids such as lipid soluble vitamins, steroid hormones, hydrocarbon, and others. The natural lipids are mono, di, and triglycerides and cholesterol and cholesterol esters. Further, miscellaneous lipids are carotenoids, squalene, terpenes, and others. The widely used lipid-based materials for targeted edible films and coatings include beeswax, mineral oils, vegetable oils, surfactants, acetylated monoglycerides, carnauba wax, paraffin wax, glycerol esters, and resins. The most used lipids in edible coatings for minimally processed products are stearic acid, palmitic acid, and vegetable oils [3]. The oil-based materials for edible films and coatings are animal and vegetable oils. Emulsifiers and surface-active agents are lecithin, monoglycerides, diglycerides, etc. The sources for fat-based components in developing edible films and coatings are coconut, palm, milk butter, cocoa, etc. The natural waxes are carnauba wax, candelilla wax, rice bran wax, beeswax, whereas the petroleum-based waxes include paraffin and polyethylene wax. The waxes are a category of hydrophobic materials and are extensively used as an edible coating for fresh food products to increase glossiness, quality, and shelf life. The quality and safety of apples during storage life can be maintained using candelilla wax and ellagic acid-based edible coating [4]. Additionally, the edible coating based on carnauba wax can improve the shelf life of eggplant fruits [5]. The use of candelilla wax with fermented extracts of tarbush can improve the shelf life of apple at marketing conditions, where the edible-coating materials are considered as a promising preservative material [6]. Additionally, the lipid-based edible films and coatings are available as monolayer films, bilayer films, emulsion films, and others [7]. The protein and polysaccharide-based emulsion films influence by the lipid type, drying conditions, and others. Further, casein-lipid-based edible films emulsions are used to improve quality of minimally processed carrot such as reduce white blush and others [8].

7.4 Fabrication of Lipid Nanoparticles

As shown in Fig. 7.3, the various forms of lipid NPs are fabricated using high energy and low energy methods. The high energy methods of developing lipid nanomaterials include the use of mechanical devices such as homogenizers, microfluidizers, sonicators, microwave, etc. The use of mechanical devices creates intense forces to disrupt and coalesce the aqueous and oil phases by developing fine lipid NPs. The nanoemulsions are fabricated using high energy (microfluidization, ultrasonic homogenization, high-pressure homogenization) and low energy methods (spontaneous, solvent diffusion, phase inversion temperature, microemulsion, double emulsion).

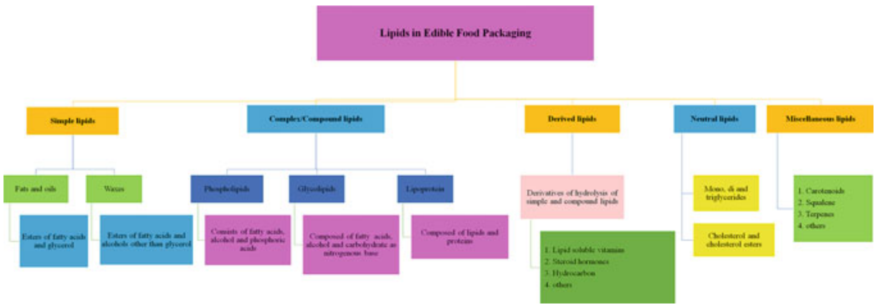

Fig. 7.2 Lipids in edible food packaging

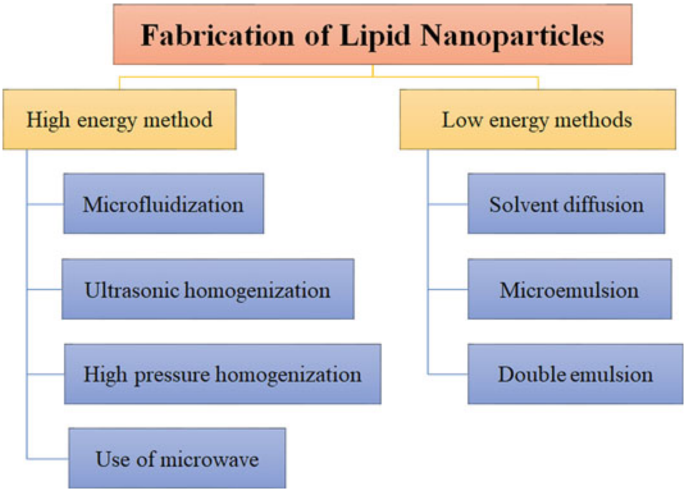

Fig. 7.3 Fabrication of lipid nanoparticles

7.5 Lipid-Based Nanostructured Materials in Edible Food Packaging

Lipid-based nanocarriers are very useful in the delivery of active compounds and provide protection against biological and enzymatic degradation. The different targeted lipid nanostructures in edible food packaging are nanoemulsion, nanoliposomes, solid–lipid NPs, and nanostructured lipid carriers (Fig. 7.4).

7.5.1 Nanoemulsion

The emulsions commonly consist of at least two immiscible liquid phase, where among the two liquid phases, one acts as a dispersed phase and another acts as a continuous phase. The emulsions are available as oil-in-water or water-in-oil

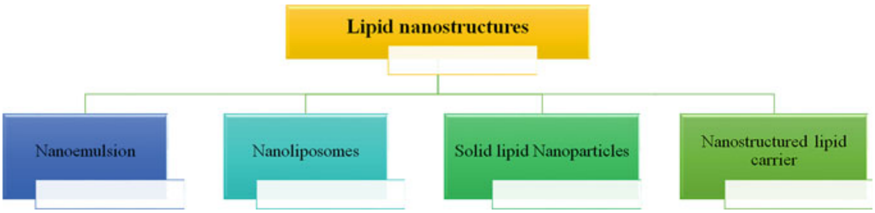

Fig. 7.4 Lipid nanostructures for edible food packaging application

emulsions, and according to the dimensions, emulsions are available as macroemulsion (coarse dispersion, dimension: >500 nm), microemulsions (colloidal dispersion, dimension: >100 nm), and nanoemulsions (colloidal dispersion, dimension: <100 nm). The macroemulsions have some insignificant features such as thermodynamically unstable, opaque, and further require high energy for processing. On the otherhand, microemulsions have the properties of thermodynamically stable, clear, and forms spontaneously. The nanoemulsions have many significant properties such as kinetically/thermodynamically stable, clear in appearance, and require high shear to develop nanoemulsions. The nanoemulsions have further tailored rheology (elastic, slowly relaxing, or viscous), optically transparent, stable against different processing conditions. The nanoemulsions have the application as food, pharmaceuticals, drug delivery, cosmetics, and others. Nanoemulsion is a widely used system targeted for encapsulation of lipophilic active ingredients. The inclusion of nanoemulsion helps to increase the functionality of entrapped compounds and can also increase the transportation of active compounds to food products. The nanoemulsion-based compounds also help in improving the organoleptic properties of food products via increasing the shelf life. The nanoemulsions are attained in two phases such as oil-in-water or water-in-oil phases. The other advantageous properties of nanoemulsions are increased surface area, enhanced solubility, increased bioavailability, etc.

7.5.2 Nanoliposomes

The liposomes (colloidal particles) are composed of lipid bilayer-based membranous system and encapsulate the aqueous spaces [9]. The nanoliposomes or submicron bilayer lipid vesicles are a favorable approach for encapsulation of bioactive compounds with their controlled release and can further used to enrich the food systems [10, 11]. The various components such as nutraceuticals, nutrients, enzymes, food antimicrobials, and food additives can be incorporated in different food systems using nanoliposomes based carrier vehicles. The advantages of nanoliposomes for encapsulating bioactive compounds are targeted delivery, improved bioavailability, reduced toxicity, sustained release, moisture stability, enhanced solubility, prolonged shelf life, reduced side-effects and others.

7.5.3 Solid–Lipid Nanoparticles

The solid–lipid NPs (SLNs) are commonly spherical in shapes and have the dimensions in the range of 50–1000 nm, which is generally consisted of lipid, surfactants, drugs in an appropriate proportion. In SLNs, the SLNs are considered as nanocolloidal carriers, where solid and lipid cores are generally stabilized by the surfactants. The generally used core lipids are acylglycerol, fatty acids, waxes, and others. Further, the commonly used stabilizers are phospholipids, cholesterol, sphingomyelins, and others. The SLNs have been used as a carrier agent for various therapeutic groups such as antioxidants (curcumin, bixin, quercetin, puerarin, etc.), anticancer agents (gambogenic acid, noscapine, docetaxel, paclitaxel, etc.), anti-hypertensive drug, antiviral drugs, ophthalmic drugs, antidiabetic drugs, anti-malarial drugs, anticoagulants, vitamins, and others [12]. Thus, the important factors in developing solid–lipid NPs include selection of lipids; selection of bioactive compounds, selection of surfactants, and selection of cryoprotector. There are several benefits of using SLNs which include (i) use of physiological lipids; (ii) do not involve the use of organic solvents; (iii) use of high-pressure homogenization methods (a well-accepted technique); (iv) protection of sensitive bioactive molecules; (v) provide controlled release of bioactive molecules; (vi) controlled drug release; (vii) possibility of drug codelivery; (viii) incorporation of hydrophobic drug; (ix) longer stability; (x) targeted therapy; (xi) biocompatible and non-toxic; (xii) ease scale up; and others. The general methods of developing SLNs are high-pressure homogenization, microemulsion, emulsification and solvent evaporation; emulsification and solvent diffusion, double emulsion and others [12, 13]. The property analysis of SLNs is done using several techniques such as (i) surface morphology (scanning electron microscopy, field-emission scanning electron microscopy, cryo-FESEM), (ii) thermal properties (differential scanning calorimetry), (iii) particle size (high resolution transmission electron microscopy, laser diffractometry); (iv) surface modification (X-ray photoelectron spectroscopy); (v) viscosity (rheometry); (vi) drug entrapment efficiency and drug loading (High performance liquid chromatography) and others [12]. The application of SLNs in developing edible films and coatings has many advantages such as (i) minimize the senescence of food products; (ii) development of edible nanocoating for whole fruits and vegetables; (iii) reduce the rate of natural maturation process in food products; (iv) may impart antimicrobial effect, antioxidant effect to food products for the components of coating materials, etc. The disadvantages of SLNs are storage at low temperature, drug expulsion, NPs agglomeration, and others.

7.5.4 Nanostructured Lipid Carriers

Lipid nanocarriers act as a nanosystem for the delivery of bioactive substances to protect it from biological and enzymatic degradation. The lipid-based nanocarriers are developed using a mixture of solid and liquid lipids generally in the ratio of 70:30 and this kind of nanocarriers can overcome the problems associated with SLNs [14]. The nanostructured lipid carriers are the second generation SLNs with improved traits used for drug/bioactive compounds delivery [15–18]. The various lipids from natural sources which are used in the fabrication of NLCs are beeswax, carnauba wax, cocoa butter, oleic acid, soybean, grape seed, corn oil, sunflower oil, and others [14]. In edible coatings, the SLNs and NLCs with polymer supports are used in developing edible-coating materials. NLCs are also used as a delivery system for nutraceuticals which are suitable within foods and other beverages. However, the limitations of NLCs in food systems include the optimal selection of food-grade ingredients for prolong use of food products and their chances of developing toxic components. NLCs are generally developed from O/W-based nanoemulsions, where the core materials consist of a bioactive compound solution in a matrix of lipid materials (mixture of solid and liquid lipids) and the external phase are usually made up of a mixture of water and emulsifier. The significance of using NLCs in regards to other nanolipid materials includes (i) better colloidal stability; (ii) no use of organic solvents; (iii) less prone to change in particle shape and others. The NLC-based system has increased used in food industry for encapsulating active compounds such as cardamom oil, vitamin D, pomegranate seed oil, rutin, quercetin, resveratrol, lycopene, and others. The commonly used solid lipids in NLCs are glycerol monostearate, glycerol distearate, lauric acid, stearic acid, tristearin, cocoa butter, and others. On the otherhand, the liquid lipids in NLCs are generally oils such as sunflower oils, oleic acid, olive oil, glyceryl behenate, linseed oil, miglyol oils, capric triglycerides, and others.

7.6 Several Properties of Lipid Nanoparticles

Various characteristic properties of lipid NPs (LNs) influence their stability, biological fate, and release properties inside a food system. Thus, the importance of various properties of LNPs is discussed in the below section.

7.6.1 Structure and Composition

Generally, LNPs have simple core–shell structure where the lyophilic substances such as waxes, mineral oils, EOs, oil-soluble vitamins, mono, di, and triacylglycerols, bioactive compounds, and nutraceuticals are encapsulated (Fig. 7.5). The outer layer shell contains mainly surface-active agents such as surfactants, polysaccharides, proteins, phospholipids, emulsifier, and inorganic particles [19]. However, the biological fate of LNPs in the gastrointestinal tract (GI) depends on the digestibility of core and shell material. Additionally, core and shell materials are not equally digestible in the same region in the human body. As discussed earlier, besides the simple structure of LNPs, the various forms of LNPs are solid–lipid NPs (SLNs), NP colloidomes, clusters, hydrogels, and others (Fig. 7.5). These particles have specific functions including protection of bioactives, control release of nutraceuticals, protection of encapsulated compounds during storage, and others. Therefore, any changes in the structural system of LNPs within the GI tract can lead to alter the biological fate and there may be chances of occurring potential toxicity.

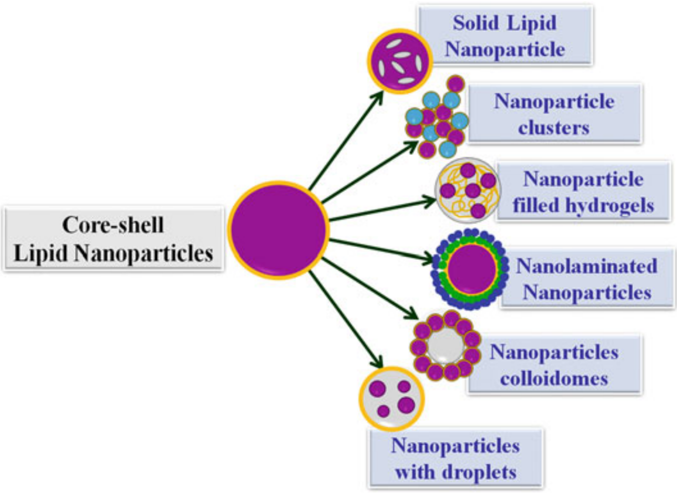

Fig. 7.5 Several core–shell lipid nanoparticles

7.6.2 *Dimensions*

Particle size of LNP has an influencing property on the physicochemical and functional properties as well as on the potential biological fate while using in edible films and coatings in the form of nanoemulsion, nanoliposome, SLNs, and others. Generally, food-grade LNPs have the mean diameter range of 10–100 nm, which provide better stability, viscosity, clarity, and bioavailability [20, 21]. Further, smaller particles have better penetration power in any type of system. However, the particle size depends on the fabrication methods, type of mechanical device used, time of exposure to the device and energy inputs, chemical composition including amount of emulsifier, surfactant, and other factors.

7.6.3 *Interfacial Properties*

The surface of LNPs consists of various molecules and ions which have high impact on their interfacial properties including thickness, polarity, charge, permeability, rheology, digestibility. Therefore, interfacial properties are crucial factor for obtaining specific desirable characteristics which can be controlled via selecting different emulsifiers and supporting materials such as surfactants, phospholipids, proteins, or polysaccharides [22]. In this context, hydrophilic polysaccharide as supporting material can enhance the retention time of LNPs inside the system and reduce the chances of reduced bioavailability in the body after absorption. However, the surface of the LNPs can also be modified via precipitation of inorganic ions on to it, thereby alteration of electrical charge occurs. Further, the electric charge of the LNPs also depends on the pH or ionic strength of the system.

7.6.4 Thickness of Interfacial Coating Material

The thickness of interfacial coating material has great influence on the stability of the nanoemulsion as with increasing the thickness, the emulsion gets more stable and there is a less chance of particle aggregation due to increased steric repulsive force [19, 23]. Therefore, thickness is one of the crucial interfacial factors for LNP-based nanoemulsion system.

7.6.5 Polarity

The surface polarity of LNPs regulates the interaction with other compounds including particles, molecules, and biological substances within the GI tract. In general, LNPs have hydrophilic surface toward the surrounding due to the adsorbed emulsifier molecules. However, there is a chance of presence of non-polar groups if the core is not completely covered by the emulsifier or if the emulsifier consists of some non-polar region which protrudes into the surroundings. Therefore, polarity defines the nature of interaction or bonding with the surrounding or system molecules.

7.6.6 Environmental Effects and Digestibility

The changes occur in the LNPs system due to the surround factors including temperature, pH, ionic interference, and enzymatic activity can be regulated by the interfacial coating of LNPs. In this regard, some emulsified produce highly stable LNPs, whereas others produce sensitive LNPs which can alter the properties by the surrounding factors. Similarly, some interfacial coating materials such as proteins, modified starch, and few surface-active agents are easily digested by the GI tract system; however, others are indigestible. Therefore, the digestibility of coating material also determines the potential biological fate of LNPs.

7.6.7 Physical Properties

The LNPs can be prepared from liquid, solid, or semi-solid form of oil via applying various fabrication methods. In this regard, during hot homogenization method, the oil phase remains in liquid state by maintaining the temperature above its melting point, and subsequently, the crystalline phase occurs via reducing the temperature of the system. The amount and polymorph of crystals can be controlled via fabrication methods and chemical compositions. Moreover, addition of triacyglycerol can control the melting point, where it generally increases the melting point after adding into the system and decreases the unsaturation [19]. SLNs and nanostructured lipids have formed from the fully or partially solidify lipid. Therefore, these two forms of nanolipid are highly crystalline in nature. In this case, the crystalline lipid is dissolved in an organic solvent and then emulsified to form oil-in-water emulsion after that the solvent is removed. In addition, the crystalline nature in nanoform of lipid is more as compared to bulk phase and the crystallization temperature in NPs is less as compared to bulk [24]. Further, the nature of crystals developed in the nanoemulsion is different as compared to bulk due to specific volume for nucleation and crystal growth inside the nanoemulsion, which may influence the biological fate of the LNPs inside the GI tract after consumption [25].

7.7 Application of Lipid Nanoparticles in Edible Food Packaging: Case Studies

With respect to the above discussion, lipid nanostructured materials offer several advantageous properties which can be utilized for tailoring the food packaging properties. The concept of nanolipid is based on the used derived lipids such as various animal and vegetable fats including waxes, fatty acids, and acylglycerol in nanodimension range. In addition, nanolipids and its derivative including nanoemulsions, liposomes, and polymeric NPs are the early developed materials. In this context, targeting to improve the properties and associated difficulties, SLNs and its modified form, namely nanostructured lipid systems, have been developed. In this regard, the effects on various forms of nanolipids in edible food packaging systems need to be addressed for revealing its advantages. The various applications of nanostructured lipid in the edible food packaging system are summarized in Table 7.1.

In this context, edible films developed by incorporating SLNs into xanthan gum can be able to provide improved properties of films [26]. The results indicate that incorporation of SLNs into polymer matrix has improved the color difference, mechanical, thermal, and water vapor barrier properties of the edible films. Besides, the nanosize of particles (<300 nm) provides stability to the film-forming dispersion. In this study, the SLNs have developed from candeuba wax using the hot homogenization method. However, these findings suggest the potential application of developed SLN-based edible films in the preservation of whole fruits and vegetables. In this context, the best developed coating solution using the mentioned system with 65 g/L of SLNs can be able to preserve or reduce the loss of ascorbic acid and total phenol content which contributes the antioxidant activity, control in

Table 7.1 Applications of nanolipids in the prospect of edible food packaging

Types of Nanolipids	Design of packaging material content	Types of packaging	Food products	Properties	Reference
Solid–lipid NPs	Xanthan gum/ candeuba wax SLNs	Edible film	Whole fruits and vegetables	Improved the color difference, mechanical, thermal, and water vapor barrier properties	[26]
Solid–lipid NPs	Xanthan gum/ candeuba wax SLNs	Edible coating	Guava fruits	Reduced weight loss, retarded ripening, respiration rate, controlled change in greenish color, improved quality, and enhance shelf life	[28]
Solid–lipid NPs	Xanthan gum/ beeswax SLNs	Edible coating	Strawberry	Minimized weight reduction, decaying rate, loss of firmness and changes with enhanced refrigerated storage life	[30]
Solid–lipid NPs	β-lactoglobulin/ SLNs	Edible film	Not specified	Improved water vapor barrier and mechanical properties, controlled mass transfer	[31]
Solid–lipid NPs	ZEO SLNs	Edible film	Not specified	Retard fungal growth	[32]
Nanoemulsion	Sodium alginate/ mandarin fiber/ tween 80/ oregano EO	Edible coating	Cut Cheese	Prolong shelf life via preventing microbial growth and improved nutritional quality, safety and appearance during refrigerated storage	[34]
Nanoemulsion	Modified CS/ emulsifier/ lemon EO	Edible coating	Leafy Vegetables	Provides significant antimicrobial activity and extended shelf life of 10–14 days	[35]
Nanoemulsion	Sodium alginate/tween 20/glycerol/ sage EO	Edible film	Not specified	Improved mechanical barrier properties with controlled color change and effective antimicrobial activity against food borne pathogen	[47]

(continued)

Table 7.1 (continued)

Types of Nanolipids	Design of packaging material content	Types of packaging	Food products	Properties	Reference
Nanoemulsion	Sodium alginate/citral/ emulsifier	Edible coating	Fresh-cut pineapples	Improved texture, delayed in respiration, reduced microbial growth, better color retention and extended storage life	[37]
Nanoemulsion	Sodium caseinate/ ginger EO/ tween 80	Edible coating	Chicken breast fillets	Prolonging the storage life of raw poultry meat by preventing the microbial growth with maintained quality and minimization of cooking loss and color changes	[40]
Nanoemulsion	CS/quinoa protein/thymol	Edible coating	Strawberry	The system provides antifungal properties with improved quality and sensorial attributes and consequently extends shelf life	[38]
Nanoemulsion	Sodium alginate/ lemongrass EO/ tween 80	Edible coating	Fresh-cut apple	Delaying maturation rates of cut fruits via reducing respiration rate and ethylene production and slowing down browning effects thereby prolonging shelf life as well as provides antimicrobial activity	[39]
Nanoemulsion	Citrus EO/ CSNPs	Edible coating	Silvery pomfret	Extended shelf life via preventing microbial growth and reduction in protein and lipid oxidation	[42]
Nanoemulsion	Pectin/OEO/ resveratrol	Edible coating	Fresh pork loin	Improved quality with maintained color, pH, and other parameters, prolonged storage life	[41]

(continued)

Table 7.1 (continued)

Types of Nanolipids	Design of packaging material content	Types of packaging	Food products	Properties	Reference
Nanoliposome	Agar/AAO/CS	Edible films	Cherry tomato	The developed system significantly effective against *E. coli* O157:H7 with bacteriostatic effect	[44]
Nanoliposome	gelatine/CS nanofiber/ ZnONPs/ betanin NLP	Edible films	Fresh Beef	Betanin nanoliposome-based system provides noteworthy antioxidant and antimicrobial properties with prevented protein and lipid oxidation and improved quality attributes	[43]
Nanoliposome	CS/SKEO/ soy-lecithin	Edible coating	Lamb meat	Improved quality of the meat by retarding the lipid oxidation, microbial growth and prolonging the shelf life	[46]
Nanoliposome	CS/sodium alginate/ vitamin C	Edible carrier	Mandarin juice	Release of vitamin C into the juice and retarding the lipid oxidation and hydrolysis with higher microbial stability	[45]

SLNs Solid–lipid nanoparticles, *ZEO Zataria multiflora* EO, *EOs* Essential Oils, *CS* Chitosan, *CSNPs* Chitosan nanoparticles, *OEO* Oregano EO, *AAO Artemisia annua* oil, *ZnONPs* Zinc oxide nanoparticles, *NLP* Nanoliposome, *SKEO Satureja khuzestanica* Essential oil

changing color, reduction in ripening rate and maintain the firmness via controlling pectin methylesterase enzymatic activity while applying on guava in refrigerated storage condition (10 °C) during eight weeks [27]. In similar study, Zambrano-Zaragoza et al. [28] have developed SLNs incorporated xanthan gum-based edible coating, targeting to enhance the shelf life of guava fruits. The results provide reduction in weight loss, delay in ripening by reducing respiration rate, minimum or no change in greenish color, best preserved quality and enhance the shelf life of guava while storing under refrigeration condition (10 °C) at 85% relative humidity (RH) for 30 days. Additionally, the candeuba wax SLNs/xanthan gum system is a promising effective edible packaging system for preservation of whole fruits and vegetables via modifying the properties of developed system; thus, the system can also improve the shelf life of tomato under refrigerated storage (12 °C for 26 days) [29]. The system can enhance the mechanical strength by increasing the tensile strength, elongation, and elastic modulus which leads to maintaining the firmness of tomatoes by controlling various parameters such as pH, acidity, soluble solids, color changes, and antioxidant capacity (Lycopene concentration) and subsequently extends the storage life of tomatoes. Another application of nanoedible coating of beeswax-based SLNs incorporated xanthan gum can be useful for prolonging the shelf life of strawberries [30]. The developed coatings is capable to minimize the weight loss, reduction in decaying rate, loss of firmness, and minimum color changes with enhanced shelf life of strawberries during the refrigerated storage at 4 °C for a period of 21 days. Design of SLNs incorporated protein (β-lactoglobulin)-based edible films can provide tailor-made properties of film via improving water vapor barrier and mechanical properties [31]. The designed film is able to reduce water vapor permeability significantly and can be able to control mass transfer.

Beside tunable properties of edible films and coating, the SLNs can also be utilized as suitable carrier for releasing natural antimicrobial agents such as EOs to the specific system. In this context, Nasseri et al. [32] has developed a system of *Zataria multiflora* EO (ZEO) loaded SLNs which is significantly effective against fungal activity. In addition, the ZEO-SLNs system is more efficient in order to inhibit the fungal growth as compared to ZEO, indicates the improvement in antifungal activity. Interestingly, the SLNs can be utilized as drug carrier system via delivering specific and controlled release to the particular site. In this regard, antioxidants, antimicrobial agents, and specific bioactive compounds can be incorporated into the food system via SLN-based edible coating or films system. Besides food packaging, the SLNs can enhance the bioavailability of entrapped drug release in the targeted zone which enlarges the area of SLNs application in the biomedical field. Moreover, the SLNs can provide controlled release of active substances, enhance biocompatibility, act as antimicrobial and topical drug delivery system, release anticancer drug at targeted and to the brain sites, work as gene vector carrier, antitubercular chemotherapy, and also able to put high impact in cosmetics [33].

In early days, the utilization of nanolipid in edible packaging for improving various properties of film and food product is obtained via formulation of nanoemulsion. Nanoemulsion incorporated with oregano EO (OEO) and mandarin fiber can be utilized as edible coating for pronging the shelf life of low-fat cut cheese under refrigerated storage [34]. The nanoemulsion system contains sodium alginate, mandarin fiber, tween 80 as stabilizer and OEO. The design system is significantly effective against *Staphylococcus aureus* by preventing the growth from 6.0 to 4.6 log CFU after 15 days of storage. Besides, this system is also effective against *psychrophilic* bacteria, molds, and yeasts. The OEO incorporated nanoemulsion-based edible coating acts as potential candidate for improving the quality, nutritional properties, and appearance of cut cheese as at the same time, it is able to extend the shelf life. Further, modified chitosan (CS)-based edible coating with nanoemulsions loaded with lemon, mandarin, oregano, and clove EO can be able to provide antimicrobial activity and improve the shelf life of leafy vegetables during refrigerated storage [35]. Among these EOs, lemon oil is significantly effective for extending the storage life and providing the antimicrobial activity. Oleic acid nanoemulsion (OAN) loaded with natural antimicrobials (lactic acid, nisin, and lauric alginate) can be incorporated to develop starch-based edible coating targeting to extend the storage life of fresh food products [36]. The OAN provides stable suspension and high optical clarity and the coating is effective against *Brochothrix thermosphacta*, *Listeria monocytogenes* Scott A and *Micrococcus luteus*. Fresh-cut pineapple can be stored under refrigerated condition (4 °C, 90% RH) for 12 days via citral nanoemulsion-based edible coating [37]. The designed sodium alginate-based coating contains citral nanoemulsion with the average droplet diameters of 66.67–131.08 nm, suppresses the respiration rate, color changes, and prevents the microbial growth, thereby provides prolong shelf life of fresh-cut pineapples. Another developed system of thymol nanoemulsion incorporated quinoa protein/CS-based edible coating can be utilized for strawberry preservation under commercial refrigerated condition [38]. The storage life of strawberries extends by four days in comparison to uncoated sample. This system is capable for providing antifungal activity with improved quality, safety, and controlled sensorial properties. Similarly, Salvia-Trujillo et al. [39] design a lemongrass EO-based nanoemulsion for developing sodium alginate-based antimicrobial edible coating targeting to preserve fresh-cut apples. The system is very significant in preventing the growth of food borne pathogen *Escherichia coli* along with maintained firmness, reduced respiration rate and browning activity with improved quality attributes and shelf life during storage condition.

Besides preserving fruits and vegetable via nanoemulsion-based edible films and coatings, perishable chicken breast fillets can be stored for longer use under refrigeration for 12 days [40]. In order to extend the durability of chicken breast fillet, ginger EO-based nanoemulsion incorporates into the sodium caseinate for developing nanoemulsion-based edible coating system. The design system is more significant for its antimicrobial activity as compared to antioxidant activity which also minimizes cooking loss and color differences. Similarly, fresh pork loin can be preserved with the developed pectin based edible coating incorporated with OEO and resveratrol loaded nanoemulsion during refrigerated storage (4 °C) under modified atmosphere [41]. The fabricated edible coating extends the shelf life of meat reducing the microbial growth, protein and lipid oxidation, controlling pH and color changes during the storage. Moreover, seafood such as silvery pomfret can be preserved via designing citrus EO loaded nanoemulsions-based edible coating along with incorporation of chitosan NPs [42]. The developed system is effective for preventing lipid oxidation and inhibiting microorganisms, thereby extending the shelf life from 12 to 16 days. Generally, nanoemulsions are developed via high mechanical mixture, ultrasonication, ionic gelation, and other methods.

Nanoliposome, another form of nanolipid, has strong imprint on preservation of food packaging via edible packaging. Various fresh produces such as fresh beef [43], cherry tomato [44], mandarin juice [45], lamb meat [46], and others are preserved via nanoliposome incorporated edible films and coating systems which also depicts in Table 7.1. In this context, agar-based edible film incorporated with medicinal plant extract (*Artemisia annua* oil) loaded nanoliposome and CS can be beneficial for preservation of cherry tomato [44]. The system is significant in inhibiting *E coli* O157:H7 due to the presence of nanoliposome with the mean diameter of 191.8 nm and more than 30 mV zeta potential value. In addition, the medicinal plant extract is volatile in nature; therefore, liposome encapsulation has been utilized for protecting the antibacterial natural oil. Similarly, nanoliposome-based nanocomposites have been developed in order to enhance the shelf life and quality of fresh beef [43]. For instance, betanin nanoliposome has been developed due to its high antioxidant activity and betanin is also utilized as natural coloring pigment. Development of nanoliposome not only improves betanin activity, but also protects the active compound from damage. The developed edible nanocomposite film composed of gelatine/CS nanofiber/zinc oxide NPs/betanin nanoliposome. The inclusion of betanin nanoliposome and zinc oxide NPs provides antibacterial and DPPH (2,2-diphenyl-1-picrylhydrazyl) inhibition activity (53.02%) as the indication of improved antioxidant activity to the edible film which further helps to improve the shelf life of fresh beef. Moreover, CS is known for its natural antimicrobial activity and edible nature; however, nanoencapsulation of medicinal plant EO (*Satureja khuzestanica*) in the form of nanoliposome while incorporated into the CS-based edible packaging system provides noteworthy preservation properties during refrigerated storage of lamb meat (4 °C) for 20 days [46]. Soy-lecithin is utilized as emulsifier during development of nanoliposome. The designed system contributes effective antioxidant and antimicrobial properties. The system retards the growth of spoilage causing microorganisms and reduces TBA (2-thiobarbituric acid) value which indicates controlled lipid oxidation thereby enhances the refrigerated storage life of lamb meat. Interestingly, nanoliposome system can be utilized as carrier for vitamin C via CS and sodium alginate edible biopolymer matrices while incorporating into the mandarin juice [45]. Based on the application of nanolipids it can be summarized that SLNs, nanoemulsions and nanoliposomes are effective carrier for bioactive compounds and can be utilized as preservative into the food system.

7.8 Safety of Lipid Nanoparticles

The high-end utilization of lipid NPs in the food and agricultural sector is somehow restricted due to lack of knowledge and unrevealed properties regarding safety and there may be chances of occurring potential toxicity due to long storage time, which may have adverse impact on human health. Moreover, the potential toxicity of these NPs to some extent depends on their particle characteristics such as particle size, bioavailability, biocompatibility, and the quantity of use. In this context, research is carried out to understand the biological fate of lipid NPs. Interestingly, literature reveals that particle size can influence the biological fate of hydrophobic bioactive compounds, and smaller size of encapsulating compound enhances the bioavailability. For instance, decreasing the particle size enhances the solubility of hydrophobic bioactive compounds in the GI fluids indicate adsorption, faster digestion of bioactive carriers such as triacylglycerols allow formation of mixed micelles indicating solubilization and transport, the penetration and diffusion rate in the epithelium cells, and digestibility due to longer retention in smaller intestine. The increase in oral bioavailability is desirable for many bioactives; however, the reverse phenomena associated with potential toxicity is observed due to high level of consumption. Therefore, use of lipid NPs into the food system should maintained the specified limit describes by the standard bodies or agencies. The metal and inorganic NPs (silver, gold, titanium dioxide, silicon dioxide, zinc oxide) are non-digestible and can directly absorb in the epithelial cell where they may accumulate, digest, or transport via blood system. If these particles are transported from the epithelial cell, then they may circulate through the body which leads to metabolic reaction, excretion, and accumulation in certain tissues. In case of lipid NPs, no such incident like direct absorption in GI tract has been found till date [48]. However, there will be chances of occurring similar things if non-digestible oils are used for encapsulation of bioactives during fabrication of lipid NPs. Further, there may be a chance of aggregation of particles based on the size, shape, charge, composition, and interfacial chemistry. The presence of lipid NPs in the mouth, stomach, and small intestine may interfere with the regular functioning of GI tract due to their smaller size, large surface area, and high surface energy which may lead to cause potential toxicity as the behavior is not similar with microlipids [49, 50]. Besides, presence of organic solvents and some components like surfactants may cause potential toxicity as these solvents and components are required during the fabrication of nanolipids in different forms like nanoemulsion, NPs, nanoliposems, solid–lipid NPs, and others. However, the organic solvent mostly evaporates during drying although very trace amount may present in the final product. Therefore, the toxic effects of used chemicals must be known before using in the fabrication of nanolipids. Nevertheless, based on the above discussion, more research on various forms of nanolipids is required to carry out for revealing every aspect of these nanomaterials and their probable effect on human while consuming via food and beverages.

7.9 Conclusion

The idea of incorporation of lipid-based nanomaterials into packaging film paves the importance of using the edible packaging as the preservation technique. The concept of emulsion, encapsulation, and liposome using lipid-based compounds is ancient, and in order to improve their effectiveness, nanotechnology has played a vital role which converts them into nanoforms fulfilled with various significant properties. The lipid-based nanosystems via edible films and coatings not only preserve the food with quality and safety, but also deliver specific bioactives, nutraceuticals which develops value-added products. The function of nanolipids as the nanocarriers will enhance the market demand of nanolipid fortified food and beverages.

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Chapter 8
Inorganic Nanomaterials in Edible Food Packaging

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8.1 Introduction

The non-biodegradable food packaging materials have created enormous environmental pollution and credit most of the plastic-based environmental concerns all across the world. In the present scenario, consumers as well as the complete fraternity of scientists and industrialists associated with packaging materials demand biodegradable materials as a replacement of non-biodegradable materials. Recycling of such non-degradable plastics has not shown much efficacy in terms of both economic and performance aspects. There are several available biopolymers which are used as packaging materials to preserve food products [1]. Certain compostable polymers like poly(lactic acid) (PLA) need more of industrial composting environment to degrade it completely. In that case, the collection and segregation of these biodegradable plastics from non-degradable one, is highly important for safe material processing and is a serious matter of concern for industries as well as composting of these material. Compostable polymers which are frequently termed as biodegradable polymers may or may not shrink environmental pollutions, as these polymers hardly assimilate under non-simulated condition, i.e., under natural conditions. Hence, the biodegradation could be a criterion to boost up the awareness on environmental concern, but cannot completely nullify the environmental pollution. Very recently, scientific research on edible and biodegradable packaging using inorganic nanomaterials has taken tremendous space particularly in food packaging applications. End-of-life of such edible food packaging films is not associated with any separate post-service composting condition or treatment to knock it down to the environment.

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As similar to other synthetic, non-biodegradable food packaging materials, these edible food packaging films based on inorganic nanostructured materials are capable to provide additional activities related to carrying out biodegradation under simulated or non-simulated condition. Though the terms "edible film" and "edible coating" have different meaning in terms of its applicability; herein this book, they are used interchangeably. These edible packaging films are not only able to prevent moisture loss as well as oxygen and carbon dioxide penetration but also effectively retain the aromas and prevent solute transport [2]. As like conventional packaging materials, these inorganic nanomaterials-based edible films are able to retain the quality and stability of packaged food products over extended time period by controlling the exchange of gases, moisture, flavor, aroma, lipids, and other organic compounds between environment and food. Also, these films resist microbial attack on food products. Besides the above-mentioned characteristics, some of these films are capable to provide safety from physical damages. It is obvious that these edible films cannot replace conventional packaging materials completely. The edible films in food packaging applications are more commonly derived from natural sources and are considered as the waste or by-products [3]. The frequently used materials for the production of edible films include plant and animal-based proteins, lipids, polysaccharides, and large possible mixture of these materials. It is also the common trend to incorporate different types of additives into these edible materials in order to enhance various functional properties of resulting films. The inherent film-forming ability of different biomaterials like cellulose, starch, chitosan, pectin, alginate, pullulan, kefiran, etc., makes it more popular for the specified application as edible films in food packaging. Based on the type of food to be protected, the barrier as well as other properties of edible films can further be tailored by the incorporation of multiple food-grade additives. As for example, plasticizers are added to reduce the brittleness of films, inorganic fillers in nanoforms are included for a variety of purposes into the edible films during the processing step.

In this chapter, we are primarily discussing on the effect of various types of inorganic nanofillers as functional additive in edible food packaging materials. The structure–property relationship and functionality of these nanofillers are also discussed in detail. Also, advantages and limitations of different inorganic nanofillers are thoroughly discussed in order to give a broader sense of understanding on its applicability in different edible biopolymer films. Most commonly used inorganic nanoparticles as additive in edible biopolymer films for food packaging application are silver nanoparticles, zinc oxide nanoparticles, silicon oxide nanoparticles, titanium oxide nanoparticles, iron nanoparticles, etc. In the subsequent section of this chapter, we are going to discuss on various functional properties of different inorganic nanoparticles in particular to food packaging applications.

8.2 Inorganic Nanofillers in Edible Films

Edible films are mostly made up of fruits and vegetable extracts that are basically some biopolymers and act as the matrix of the films. Such biopolymers are hydrophilic in nature and possess poor mechanical strength, abrasion resistance, thermal degradation stability, as well as oxygen, carbon dioxide and moisture barrier properties in comparison with conventional polymers. Any material with poor performance in above-mentioned properties is generally not competent enough to be used in food packaging applications. One of the most feasible strategies to tackle this technical limitation of such edible food packaging materials is to inclusion of fillers into the matrix material. Addition of fillers into the biopolymers leads to formation of modified form of material called biocomposites. Further, as the fillers come within the range of nanometer then the resultant material is called bionanocomposites. There are a range of nanofillers available that can add up multiple benefits to the base polymer matrix. In particular, inorganic nanofillers are capable to enhance various physical, mechanical, and thermal properties of the edible polymeric films. Depending upon the factors such as good polymer–fillers, interfacial interactions, uniform nanofiller dispersion within the polymer matrix, certain changes in behavior at molecular level occur, which results in dramatic improvement in thermal stability and mechanical strength of the edible films. High surface area of the added filler available for interaction with polymer matrix is essential to increase the mentioned factors in a polymer biocomposite system. Moreover, a greater surface area of the nanofillers can be achieved through improving the aspect ratio of the fillers. A high aspect ratio of fillers (length/ diameter ratio), fine particle diameter, and presence of holes enhances the surface area of fillers to interact with the polymer matrix. Further, as part of smart packaging, the edible biopolymers are filled with certain inorganic nanofillers that provide functionalities including antimicrobial, antifungal, antioxidant, ultraviolet (UV) resistance property, etc., to the edible films. However, the various available inorganic nanomaterials (zinc, silicon oxide, titanium oxide, magnesium oxide, iron

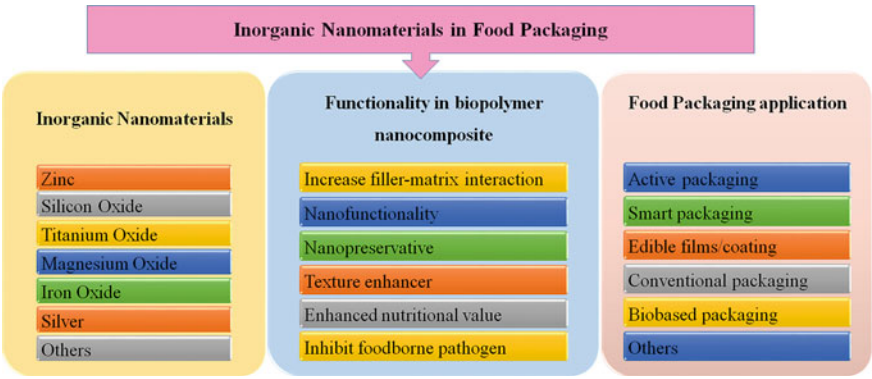

Fig. 8.1 Various aspects in inorganic nanomaterials in food packaging applications

oxide, silver, and others), their functionality in biopolymer nanocomposites (increase filler–matrix interaction, nanofunctionality, nanopreservatives, texture enhancers, enhanced nutritional value, inhibit foodborne pathogens), and targeted food packaging applications (active packaging, smart packaging, edible films/coatings, conventional packaging, biobased packaging, and others) have been mentioned in Fig. 8.1.

8.2.1 Zinc Nanoparticles

Zinc is an essential element for defensive mechanism (immune system) of human body. It also plays important roles in cell division and cell growth in injuries, and helps healing at faster rate. It is the integral element that is found in every cell of human body. Often due to inappropriate diet, deficiency in zinc causes multiple health problems. In those cases, zinc supplement can be fulfilled upon using zinc-based nanoparticles as functional filler in biopolymer of edible films. Other than its nutritional values, zinc oxide nanoparticle (ZN) is multifunctional inorganic nanoparticles that is recognized by the US Food and Drug Administration (USFDA) as safe to human consumption. Hence, instead of making zinc as monofunctional food additive, ZN can be used in edible food packaging for multiple applications such as antimicrobial activity and antifungal activity. The nutritional value including texture, flavor, and storage properties of food products is improved using zinc nanoparticles.

8.2.2 Silicon Oxide Nanoparticles

Silicon oxide nanoparticle in amorphous phase is authorized as food additive by the European Union (EU) and is found to be completely safe for consumption after several long-term experiments in animals. Further, these nanomaterials impart great advantages in edible packaging films like improvement in mechanical strength of the films, antimicrobial properties, enhancing shelf life of packaged food products, generating high gas barrier properties, etc. Silica is most abundant mineral on earth and mostly exists in the form of its oxides in nature. Hence, oxides of silicon are relatively easy to prepare at low cost. Other than improvement in mechanical properties in the edible biopolymer films, these nanoparticles are capable of improving moisture barrier property of films effectively. Further, these nanoparticles have gained tremendous application in commodity food packaging areas, primarily due to its functional properties at cheaper price that impart great advantages in the edible films.

8.2.3 Titanium Oxide Nanoparticles

Titanium dioxide nanoparticles are functional fillers used mostly in variety of food products including medicines. It is the most explored nanomaterial in the area of food and edible food packaging due to its non-toxic, photostable, antibacterial, high thermostability, and inclusion of TiO_2-N resists growth of microorganisms in food products. Other than improving functional properties, TiO_2-N has attained extensive attention owing to its low cost and high antimicrobial activity.

8.2.4 Magnesium Oxide Nanoparticles

Magnesium oxide is a naturally available mineral that is colorless and crystalline material and is produced economically on large scale. It is also used in food and in edible food packaging materials and is also certified as safe in food applications by USFDA. It also exerts great improvement in mechanical strength, opacity that provided safety to food products from UV rays, gas barrier property, etc., to be used as edible biopolymers like chitosan films.

8.2.5 Iron Oxide Nanoparticles

Iron is the most essential and basic requirement of human body as it constitutes the hemoglobin. Hence, inclusion of iron-based compounds into edible films for food packaging will be advantageous as it will be providing nutrition. A hybrid nanostructure of magnetic ferro or ferric oxide nanoparticles coated on graphene oxide nanosheet provides better barrier property even higher than aluminum films [4]. The nanocomposites of Fe_3O_4-NPs with starch, cyclophosphamide, and glycerine show superior property related to its application as edible food packaging materials [5]. Hence, other than nutritional values, iron oxide nanoparticles also provide safeguard to food products from UV rays, thereby increasing the shelf life.

8.3 Effectiveness of Inorganic Nanomaterials of Packaging Properties

8.3.1 Mechanical Property

As referred earlier, mechanical properties of the edible films do not fully comply with the standards required for food packaging applications. Due to this limitation, these edible films may be used as complement to conventional food packaging material. The use of reinforcing agents to the edible polymeric matrix with nanomaterial served the purpose by increasing the mechanical strength of the films. In this respect, silver nanoparticles (AgNPs) have attained maximum attention toward the specific applications [6]. The biopolymer-assisted synthesis of AgNPs gives rise to improve the stability and dispersion of the nanoparticles in the polymer matrix of films that leads to development of final structural stability of films [7]. For in situ technique of preparation of edible biocomposite films, the biopolymer acts as dispersing medium of the already synthesized nanoparticles [8]. In a nanocomposite film of biopolymer with AgNPs, van der Waals force of attraction exists between the hydroxyl group of biopolymer and partial positive charge of the metal nanofiller surfaces. Chitosan-based edible packaging films showed significant improvement in mechanical resistance upon incorporation of AgNPs [9]. Also, the improvement in mechanical strength upon incorporation of the nanoparticle was significant in case of edible film based on konjac glucomannan–chitosan and cassava starch blend system as well as hydroxylmethyl cellulose/bacterial cellulose nanocrystals [10, 11]. Further, the incorporation of AgNPs in pectin film causes slight increment in mechanical strength while overall change in mechanical property was insignificant [21]. This mild increment in the mechanical strength is due to the formation of hydrogen bond between the pectin polymer and the nanoparticle surface as evident from the chemical analysis of nanocomposite samples. In case of bionanocomposite films of carrageenan with minor amount of AgNPs, the increased mechanical strength was attributed to existence of interfacial interaction between the two components [12]. However, a linear decrease in mechanical strength with increasing the nanofiller loading can also be found due to increased aggregation in nanoparticle at higher concentration in base matrix. Further, in case of starch film, a decreasing value in the mechanical strength and elastic modulus was observed, while retaining the percentage elongation at break value of the bionanocomposite films [13]. Similar observation on deteriorated mechanical strength with containment in toughness values was reported for agar-based, corn starch-based, and beeswax-based edible films [14, 15]. However, in case of agar/banana powder-based biopolymers and most other edible films, addition of AgNPs caused significant increment in toughness at the cost of mechanical strength [16, 17]. The hydroxypropyl methylcellulose based edible films containing AgNPs of different sizes provide increase in tensile strength, which is associated with decrease in percentage elongation at break [18]. The effect was found to be more prominent in case of nanoparticles with smaller size.

It can be realized from the above discussion that metal nanoparticles like AgNPs are capable to act as reinforcing inorganic fillers of several biopolymers. In a composite system, reinforcement is a phenomenon that exists only if physical or chemical interaction exists between the polymer matrix and the nanoparticle fillers. Depending upon the nature of biopolymer matrix, different types of inter- and intramolecular interaction occur between the biopolymer chains and the nanoparticle surfaces. Among different interactions, weak van der Waals force of attraction between the polar hydroxyl groups of biopolymer matrix and the partial positive charge of nanoparticle surface exists. In certain cases, the presence of phenolic compounds, different types of proteins, carbohydrates, celluloses, hemicelluloses, etc., may take part in developing bonding between the two phases of composites. The improvement in the interfacial bonding between the nanoparticle and the biopolymer matrix causes effective stress transfer from the biopolymer matrix to filler, which leads to improvement in tensile strength. Uniform dispersion of the nanofillers within the biopolymer matrix is the result of good adhesion between the two components, and this is another factor that contributes in improving mechanical property [19]. Besides the nature of adhesion between the two phases, the concentration of nanoparticle loading also affects the final properties of bionanocomposites. Further, the processing condition has a tremendous impact in determining final properties of biocomposite films. Most commonly, in situ synthesis of AgNPs within the matrix of biopolymers, where the biopolymer reacts with the precursor or reactants and finally produces the nanoparticles, shows better mechanical performance compared to ex situ technique. These inorganic reinforcing nanoparticles may be beneficial in edible food packaging applications considering other advantages, e.g., antimicrobial effect, enhanced shelf life, etc.

8.3.2 Barrier Property

Starch is used for developing edible films, and it can bind with electropositive transition metals like silver (Ag) through electrostatic force of attraction and stabilizes the AgNPs during its synthesis. Inclusion of clay along with AgNPs in starch-based edible films caused significant increment in mechanical as well as gas barrier properties. The clay particles as well as the AgNPs are easily complexed by the huge amount of free hydroxyl groups of the starch [20]. Extensive research indicates significant improvement in moisture barrier property of various edible biopolymer films of celluloses, banana powder, agar, chitosan, gelatine etc., upon incorporation of AgNPs. The reduction in water vapor permeability of several edible films on addition of AgNPs is primarily attributed to the uniform dispersion of nanoparticles in the polymer matrix that increases tortuosity in path of water molecules that passes through the film, which caused decreasing tendency in diffusion of water molecules through the edible films. However, contradictory results can also be expected as the water vapor permeability increases upon addition of AgNPs to carrageenan films, agar, gelatin, and pectin-based edible films [21]. Additionally, nanotitanium dioxide (TiO_2-N) can improve the moisture barrier property in certain edible films. Incorporating TiO_2-N in chitosan–starch blend caused significant decrease in water vapor permeability (WVP), and the effect can be attributed to high degree of hydrophobicity of the infilled nanoparticles. Further, such water-resistant nanoparticle added to the matrix gives rise to tortuosity in the pathway for moistures. As a result, the permeability of moisture decreases sharply.

Nanosilicon dioxide (nano-SiO_2)-based starch edible films show decrease in water vapor transmission rate (WVTR). The morphology indicates highly compact structure of the films upon the addition of the nanoparticles with different sizes.

Hydrogen bond forms between the oxygen atom of nano-SiO_2 and the hydroxyl groups of starch molecules. Other than the physical barrier incurred by the incorporated nano-SiO_2 particles, the increased degree of crystallinity in the starch film caused significant reduction in the WVT rate when nano-SiO_2 (100 nm) incorporated. Smaller the size of the nanoparticle, better is the dispersion, thereby causing remarkable decrease in moisture permeability [22]. However, there are differences in the trend that can be observed based on the nature of the biopolymer matrix. Zinc oxide nanoparticle (ZN) upon addition to kefiran matrix causes significant reduction in the WVP rate, and it is attributed to the fact that kefiran forms hydrogen bond with the oxygen atom of ZN and thereby makes it a strong structure that inhibits diffusion of water molecules through it. Further, the hydrophobicity of ZN and the increased degree of crystallinity resist the moisture permeation through the film. Further, inorganic nanomaterials are also used in non-edible food packaging also to deliver improved packaging property. In this regard, magnesium oxide nanoparticles (MgO-NPs) when incorporated in PLA show improvement in oxygen barrier and tensile strength of the resultant nanocomposite films. Further, it also provides antimicrobial property to the films. Similarly, it also adds functional benefits to different biopolymer-based edible films [23]. Addition of MgO-NPs in chitosan biopolymer films increases mechanical property, film thickness, film opacity, reduced moisture barrier property, and swelling in certain solvents [24].

In other words, the addition of inorganic nanoparticles in an optimized proportion in edible films based on biopolymer matrix can provide certain characteristics including functional properties. There are several factors that are associated with the nanoparticles that dictate the final functional properties like barrier properties of bionanocomposite film. Besides the inherent hydrophobicity of the incorporated inorganic nanofillers, its interaction with the biopolymer matrix, the processing technique for the fabrication of the composite films, nucleation capacity of the nanoparticles within the biopolymer matrix, degree of crystallinity under the influence of the nanoparticles, etc., are some of the essential factors that determined the barrier property of the films. Further, the concentration of the nanoparticles in biopolymer matrix, the size and shape of the nanoparticles, and the uniformity in dispersion of the nanoparticles within biopolymer matrix have great degree of influence upon the barrier property of the bionanocomposite films.

8.3.3 Optical Properties

Optical property of edible films are primarily related to the optical transparency, the aesthetic and gloss of films, and the resistance to UV light, while UV light causes great alteration in the nutritional value of food products through developing certain chemical reaction in food products. Large amount of compounds related to phenolic compounds are present in certain food products that show sensitivity toward UV radiation. Hence, dark color in edible films is beneficial; however, transparency in films along with good aesthetic of food should also be considered.

Bionanocomposites of starch-based edible films with nano-SiO_2 in different sizes ranging from 200 to 800 nm are capable to resist UV light from penetrating and falling onto the food products. Only 30% UV light could pass through the 100-nm films at 600-nm wavelength, which impart great protection to the food products. Further, uniform distribution of these nanoparticles is much required characteristics in the films. Zinc oxide nanoparticles at 1wt% concentration are able to provide high UV stability in kefiran-based edible bionanocomposite films. The concentration of added nanoparticles and size of the nanoparticles have great impact on the final optical property of the films. For example, at higher concentration of ZN, the film opacity increases. Titania nanoparticles at lower concentration cause transparency, whereas at higher concentration of titania nanoparticle leads to increase in the opacity.

8.4 Case Studies on Inorganic Nanomaterials in Edible Food Packaging

As discussed earlier, the inorganic nanofillers used for the fabrication of edible films and coatings include titanium dioxide, silica, nanozinc oxide, silver nanoparticle, etc [25, 26]. As discussed earlier, the properties of edible food packaging can be modified using the mixed organic and inorganic materials for developing nanocomposites [27]. The inorganic nanomaterials have obtained a great interest in fabricating the edible food packaging (edible films/edible coatings). In this regard, the use of various available inorganic nanomaterials in the development of edible food packaging for attaining tailor-made properties has been represented in Table 8.1.

8.4.1 Application of Titanium Dioxide in Edible Food Packaging

As discussed earlier, the nanoparticle titania or titanium dioxide has many beneficial properties to be used in developing edible food packaging such as non-toxicity, cost-effectiveness, photocatalytic disinfection (antimicrobial activity/antibacterial property), and abundant [28–30]. Titanium dioxide is used widely to develop edible films or coatings with antimicrobial property to deliver ready-to-eat food products with controlled food property and quality. The edible films based on sweet potato starch, lemon waste-based pectin, and titanium oxide nanoparticles are considered as potential edible biodegradable packaging materials [31]. The starch, pectin, and TiO_2 (0, 0.5, 1,2,3, and 4% w/w of total solid)-based composite edible films can provide improved properties, where the incorporation of nanoparticle provides rough and uneven surface. Additionally, by increasing the nanofiller

Table 8.1 Application of inorganic nanofiller in the development of edible food packaging materials

Sl. no.	Inorganic nanofillers	Matrix materials	Property
1.	Titanium oxide (concentration: 0.5, 1,2,3, and 4% w/w of total solid) [31]	Sweet potato-based starch	4% w/w TiO_2/starch and pectin-based edible film composite property:
		Lemon waste-based pectin	WVP: $\sim 1.97 \times 10^{-10}$ $gm^{-1}s^{-1}Pa^{-1}$
			Solubility: ~20.92%
			Moisture content: ~15.15%
			Whiteness index: $\sim 75.65 \pm 0.08$
			Glass transition temperature: 79.63 ± 0.42 (°C)
			Melting temperature: 172.33 ± 0.65 (°C)
			Tensile strength: 29.01 MPa
			Remark: Food-grade UV screening biodegradable packaging
2.	Titanium oxide nanoparticle (TiO_2/WPI (w/w, %): 0.10, 0.25, 0.50, 1.0, and 2.0%) [32]	Whey protein isolate	TiO_2/WPI (2.00 w/w%)
			WVP: 2.92 ± 0.06 (10^{-10}) $gm^{-1}s^{-1}Pa^{-1}$
			Moisture content: 33.75 ± 0.89 (%)
			Solubility: 17.15 ± 0.81 (%)
3.	TiO_2 nanoparticle	Whey protein isolate (10% w/v)	Storage of lamb meat:
	Concentration: 1.0% (w/w) [34]	CNFs: 7.5% w/w	Shelf life of lamb meat is extended by 12–15 days
		REO: 2.0% (w/v)	Preserve sensory quality of lamb meats
		Glycerol: 6% w/w	Maintains physicochemical property of lamb meat
		Rosemary essential oil	Improved organoleptic properties of lamb meat
4.	Nanozinc oxide [26]	Chitosan–acetic acid solution	Storage of fresh-cut kiwifruits:
		And ultrasound treatment (40 kHz, 350 W, 10 min)	Delayed senescence
			Enhanced shelf life of fresh-cut kiwifruit
5.	Zinc oxide nanoparticle [36]	Chitosan	Edible films
			Antibacterial action against *E. coli*

(continued)

Table 8.1 (continued)

Sl. no.	Inorganic nanofillers	Matrix materials	Property
6.	Zinc oxide nanoparticle [37]	——	Active packaging
			Can produce ready-to-eat poultry meat packaging
7.	ZnO nanoparticle [38]	Cassava starch	Edible coating for fresh-cut mango cv. Arumanis
		Stearic acid	
8.	Zinc oxide [39]	Chitosan	Cellulosic paper coated with composites of chitosan–zinc oxide containing nisin
		Nisin	
		Cellulosic paper	Packaging of ultrafilter white cheese
			Antibacterial activity
9.	Biosynthesized zinc oxide nanoparticle from spinach leaves [40]	—–	Edible coating on fresh fig fruits
			Provide delayed ripening
			Inhibit microbial growth
10.	Silver nanoparticle [43]	—–	Edible coating materials for sausages
			–Helps to inhibit the growth of lactic acid bacteria for 30 days
			–Provide modified texture of sausages
11.	Nanosilver [45]	Alginate	Edible coating for shiitake mushroom
			– Maintain tissue firmness
			– Inhibit browning
			– Extended shelf life
12.	Silver nanoparticle [47]	Sodium alginate	Edible coating for fruits and vegetables
			Increased shelf life of carrot and pear
			Good antibacterial activity

concentration, the surface of the starch–pectin and titanium dioxide-based composite edible films provide discontinuous phases. The specified edible films have an ability to provide tunable mechanical property, barrier property, physicochemical property by varying the concentration of the nanoparticle. In the starch/pectin/titanium oxide-based edible films, the increased concentration of TiO_2 (0.5–4% wt) has a tendency in decreased moisture content, moisture uptake, and solubility of the film materials. The low conc. of TiO_2 provides improved moisture barrier properties, mechanical properties, etc. In 2011, Li et al. have developed edible films based on the whey protein isolate and TiO_2-based composites, where 0.1% (a low concentration) TiO_2 concentration has many beneficial properties such as (i) improved tensile strength and reduced ability of UVC absorption. On the other hand, a high concentration of TiO_2 provides increased size and crystallization degree, reduced tensile strength, reduced water vapor permeability, etc. However, the UV absorption ability of the composite films is influenced by the presence of the nanoparticle [32]. Thus, the above-mentioned composite films have a tunable transmittance, tensile strength, water vapor permeability properties, etc. The nanoparticle percentage in the composite films (whey protein isolate and TiO_2) is 0, 0.1, 0.25, 0.5, 1.00, and 2%, where the percentage of the nanoparticle in the composite films plays a crucial role in providing the tailor-made properties of the composite-based edible films. The water vapor permeability of the neat WPI films and 2 w% reinforced WPI films are $\sim 3.19 \times 10^{-10}$ g s^{-1} m^{-1} Pa^{-1} and $\sim 2.92 \times 10^{-10}$ g s^{-1} m^{-1} Pa^{-1}, respectively. The moisture contents of the neat WPI films and 2 w% reinforced WPI films are ~32.40% and ~33.75%, respectively. Additionally, the titanium dioxide and clove oil improve the property of chitosan–starch films, where the nanotitanium dioxide improves the tensile strength, physicochemical property, antimicrobial property, and biological property [33]. Further, the edible nanocomposite films for lamb meat based on whey protein isolate, cellulose nanofiber, rosemary essential oil, and TiO_2 nanoparticles have an ability in improving the product life by 12–15 days [34]. The materials can be used to preserve sensory qualities and are very effective in reduced growth of microorganisms such as *Escherichia coli*, *L. monocytogenes*, and *S. aureus*. Rosemary essential oil (REO) is used for food preservation for having antimicrobial, antioxidant, anticancer properties, etc. However, the edible packaging materials based on titanium dioxide nanoparticle-dispersed soy protein isolate can also be used for enhanced shelf life of food products [35].

8.4.2 Application of Zinc Oxide Nanoparticle in Edible Food Packaging

The zinc oxide nanoparticles, being a multifunctional inorganic nanomaterial, have a strong antimicrobial property and are another kind of inorganic nanomaterials which have attained a remarkable interest in developing edible coatings. The zinc oxide nanoparticles are used to fortify food products as zinc is considered as a necessary component for many cellular functions, hormonal and enzyme activities. Further, zinc oxide particles have a GRAS status by USFDA. In this way, zinc oxide nanoparticle is used to develop active edible coating materials. A research reports the study of ultrasound treatment and nanozinc oxide coating on fresh-cut kiwifruits to obtain improved shelf life of the cut fruit [26]. In this study, the fresh kiwifruits are given a combined treatment of nanozinc coating and ultrasound treatment and further stored at 4 °C for 10 days to study the storage life. The sample preparation is done via (i) **control**: dipping in NaClO solution, hand peeling, slicing, and storage study; (ii) **US**: combined treatment of whole kiwifruit is dipping in NaClO solution and ultrasound treatment, hand peeling, slicing, and storage study; (iii) **0.8 NZ**: combined effect of NaClO solution dipped (whole fruit) and cut into slices and coating with nano-ZnO (0.8 g/L) coating solution; (iv) **US + 0.4 NZ**: combined effect of NaClO solution (dipping of whole fruit), ultrasonic treatment, and nano-ZnO coating (0.4 g/L); (v) **US + 0.8 NZ**: combined effect of NaClO solution (dipping of whole fruit), ultrasonic treatment, and nano-ZnO coating (0.8 g/L); and (vi) **US + 1.2 NZ**: combined effect of NaClO solution (dipping of whole fruit), ultrasonic treatment, and nano-ZnO coating (1.2 g/L). The storage condition (vi) provides improved quality of cut kiwifruits with 7.87 N texture, 0.46% water loss, 1.86 $mLkg^{-1}h^{-1}$ of ethylene production at the end of storage life. The effect of the combined treatment on ethylene production and carbon dioxide production during storage of fresh-cut kiwifruits depends on various factors. The development of zinc oxide nanoparticle-reinforced chitosan-based edible films has an antibacterial activity against *E. coli* and provides thermal stability, etc. [36]. Interestingly, zinc oxide nanoparticles are also used to develop ready-to-eat poultry meat, where zinc oxide nanoparticles are effective against foodborne pathogens [37]. The development of edible coating on fresh-cut mango cv. Arumanis utilizing nanocomposite of cassava starch, ZnO nanoparticle, stearic acid helps to maintain postharvest life [38]. A research reported the development of bilayer edible films containing bilayer of cellulose and chitosan–zinc oxide-based composite films containing nisin [39]. The film can be used for the active packaging of ultrafilter white cheese. The use of biosynthesized zinc oxide nanoparticle (from spinach leaves) for coating fig fruit can improve the shelf life by maintaining the keeping qualities such as weight loss, ripening, color changes, and firmness [40].

8.4.3 Application of Silver Nanoparticle in Edible Food Packaging

Silver nanoparticles are very effective against gram-positive bacteria, gram-negative bacteria, protozoa, fungi, etc [41–43]. There are many reports available which reports the use of silver nanoparticle-based edible coating on food products such as sausages, asparagus, tomato, and ladies finger [43–46]. The application of silver nanoparticles as edible coating to sausages has an ability to inhibit the growth of lactic acid bacteria for 30 days, where the silver nanoparticles are fabricated following a green route utilizing starch and glucose [43]. The use of silver nanoparticle-based coating also provides improved texture property, which may be obtained due to the interactions between silver and the components of proteins such as phosphorus and sulfur. Further, application of alginate and nano-Ag-based edible coating on shiitake mushroom (during cold storage) helps in maintaining tissue firmness, reduced the mesophile count, and further provides enhanced shelf life [45]. Further, Mohammed Fayaz and coworkers have developed biogenic silver nanoparticles-reinforced sodium alginate films as an antibacterial edible coating for fruits and vegetables for improved quality which are also suitable for the preservation of fruits and vegetables [47]. Additionally, iron oxides, nanosilicon dioxide, and others are also used for edible food packaging materials in terms of edible coating and films for improved shelf life of food products. However, nanosilicon dioxide is used as an anticaking agent, as a carrier for flavors, to thicken pates, etc. Further, iron-fortified food products can be developed by edible coating the fruits and vegetables [48].

8.5 Conclusion

The chapter provides the readers a basic understanding on the importance of adding various inorganic nanoparticles on edible films for targeted food packaging applications. Some of these nanoparticles not only are capable of synergizing nutritional and antimicrobial properties, but add value to mechanical, thermal stability, water, and other gas barrier properties, which are very essential for the mentioned application. Further, these nanoparticles are non-toxic both to human and to the environment. These nanoparticles are cost-effective and easy to produce via multiple processes making it more convenient to use at a broader sense. In overall aspects, inorganic nanoparticles are capable to enhance multiple properties in edible food packaging.

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Chapter 9
Biopolymer Nanocomposites in Edible Food Packaging: Opportunity and Applications

Tabli Ghosh, Mohammed Modu Aji, Munmi Das, and Vimal Katiyar

9.1 Introduction

The biopolymer nanocomposite is recognized as a green material, consisting of a biopolymeric matrix reinforced with inorganic or organic nanofillers (having one dimension in the size ranges of 10–100 nm), which gives an opportunity to replace the existing packaging materials. However, composite film as a group of hybrid material generally consists of two or more different materials exhibiting distinct attributes in terms of chemical or physical property, where the composite films offer superior characteristic properties in comparison with the individual one. By going with the current trends toward a sustainable future, the fabrication of biopolymeric nanocomposites has been considered as one of the most researched materials to replace synthetic plastic materials in food packaging applications. Interestingly, the use of polymer nanocomposites has gained an extreme enthrallment in the area of edible films and coatings for the improved shelf life of high perishable food products [1, 2]. In this regard, the selection of materials in the form of composites, blends, and individual use should have a biodegradable nature similar to the food materials. Interestingly, biopolymeric composite materials and nanohybrids are an industrially viable candidate with wide availability to be used in developing edible food packaging materials with excellent attributes and can be fortified with nutritional components such as fiber enrichment, mineral enrichment, and vitamin enrichment, and further other nutraceuticals can also be added through nanostructured materials. Additionally, the functionality in edible packaging can be attained using several components such as (1) nanofillers, (2) plasticizers, (3) functional additives, (4) binding agents, (5) purees, juice and extract of fruits, and vegetables, and (6) others. Based on this, the current chapter discusses the several biopolymer

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nanocomposite-based packaging properties in terms of physical property, barrier property, surface property, mechanical property, thermal property, and others. Additionally, the recent advances of biopolymer composite in developing edible films and coatings with the multifunctional attributes have been detailed in the present chapter. The synthesis strategies for attaining improved properties of biopolymer composite including the use of binding agents, cross-linkers, plasticizers, functional additives, compatibilizers, and others have been addressed in the current chapter. Interestingly, the effective properties of biopolymer composite include metal ion releasing property, defensive mechanism, and other benefits.

9.2 Overview of Biopolymer Nanocomposite in Edible Food Packaging

The plastic packaging is easily processable and has received a great attention for the last 50 years for several noteworthy benefits. However, the waste management and utilization are a serious problem from the past few years; thus, the use of synthetic plastic is getting reduced tremendously, and several innovative eco-friendly packaging solutions are researched and developed for greener future. The focused packaging attributes for developing edible food packaging include barrier properties, mechanical properties, thermal properties, antimicrobial properties, functionality, and other health benefits. The incorporation of nanomaterials in packaging systems modifies the packaging attributes to offer noteworthy properties and an improved product life of perishable food products such as fruits and vegetables, meat and meat products, fish and fish products, and dairy products. Interestingly, the nanocomposites have received a great interest in research and developments and for wide commercial packaging applications for being the solution of several socio-environmental issues. In this regard, the polysaccharide and lipid-based composite films are developed with the aim to reduce moisture migration with improved water barrier properties. The edible polymers-based composites are used as the alternatives to the existing synthetic polymers. The polymer composite films of hydrocolloids and lipids provide a barrier against gaseous components. The several polymer composites used in edible films and coatings include hydroxypropyl methylcellulose (HPMC)–lipid composites for *mandarins* cv. fortune [3], gelatin–chitosan composite for fresh-cut melon [4], gum arabic and chitosan composites, etc. However, to combat the current need for developing sustainable packaging materials, the organic and inorganic nanofillers have been mostly utilized to develop biopolymeric nanocomposite-based edible food packaging. As mentioned in the earlier chapters, the most investigated polysaccharide-based bionanomaterial includes nanocellulose, nanochitosan, nanostarch, and others. The inclusion of several categories of nanomaterials influences the composite properties in terms of physicochemical and structural properties and also provides versatility in nature. However, the higher production cost and longer processing time for the fabrication of nanostructured materials and their nanocomposites may put limitations in developing edible food packaging materials for highly perishable food products, minimally processed food products, and others. The polymer nanocomposite can be tailor-made in terms of diverse surface functionality and packaging properties to be used in the edible food packaging sector. However, the noteworthy properties of nanofiller materials such as surface area, surface functionality, and aspect ratio make it a potential candidate in edible films and coatings. The edible food packaging includes use of mineral nanoparticles, metal nanostructures, bio-nanostructures for developing edible food packaging. Additionally, in Fig. 9.1, the several aspects of biopolymer composite in edible food packaging in terms of eco-friendly nature, packaging properties, active functions, delivery system, improved food properties with the focused application have been displayed. The existing versatility of biopolymer composites such as functionality, concentration-dependent properties, nanofiller-dependent, and others provides a boom in industry and edible packaging sector. The biocomposite materials are focused to use as a high-performance edible food packaging materials such as (1) strong films, (2) edible active packaging, (3) drug-loaded edible packages, (4) lightweight materials, (5) ready-to-eat edible packaged food, (6) water-soluble edible packaging, and (7) others. The biopolymers have some disadvantages of brittleness, poor moisture barrier properties, and low processing ability and others [5]. Further, the main aim toward developing nanocomposite-based edible films and coatings is gas impermeable property, mechanical strength, thermal stability, structural property, etc., obtained due to the nanometer size dispersion of reinforced materials in matrix. Further, the biocomposite-based edible films and coatings can be a source to supply nutraceutical rich and fresh, good-quality ready-to-eat fruits for beneficial human health.

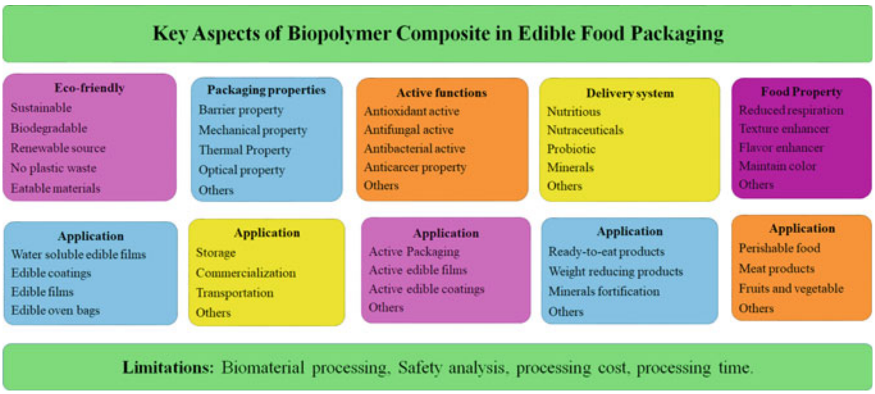

Fig. 9.1 Key aspects of biopolymer composite in edible food packaging

9.3 Application of Biopolymer Composite in Food Sectors and Others

From the last few decades, the use and availability of polymeric composites have increased in several fields including both edible and non-edible food packaging. In food sectors, biopolymer composites are used in (1) nutrition: nutrient delivery system, nutraceuticals, vitamin fortification, mineral fortification, improved sensory of food products, food rheology, etc., (2) protection in food products: antimicrobial delivery, high barrier, edible sensors, UV protection, etc., (3) food processing: nanoencapsulation, nanoemulsions, viscosifying agents, nanoadditives, gelation agents, etc., and (4) packaging sector: edible and non-edible food packaging. However, the multifaceted application of polymer biocomposite includes food packaging, biomedical application, textile industry, construction materials, electronics materials, and other high advanced applications [6–12]. The biomedical application of polymer composite includes scaffolds for tissue engineering, bone repair and fixation device, drug delivery, industrial application, hard tissue replacement, paper coating, textile, green adhesive and structural application, water filtration, etc. The various bioplastics for non-edible packaging are polylactic acid (PLA), polyhydroxybutyrate (PHB), polybutylene succinate (PBS), and their derivatives.

9.4 Biopolymer Nanocomposite Systems in Edible Food Packaging

The biopolymer composites for targeted edible films and coatings are functionalized for improved compatibility and better interaction between matrix and filler materials. The several attributes of nanofiller, matrix, and biocomposites are represented in Fig. 9.2. The multifunctional properties in edible packaging are obtained using versatile nanomaterials such as mechanical property, thermal property, antibacterial property, and barrier property. The effectiveness of nanostructured materials is

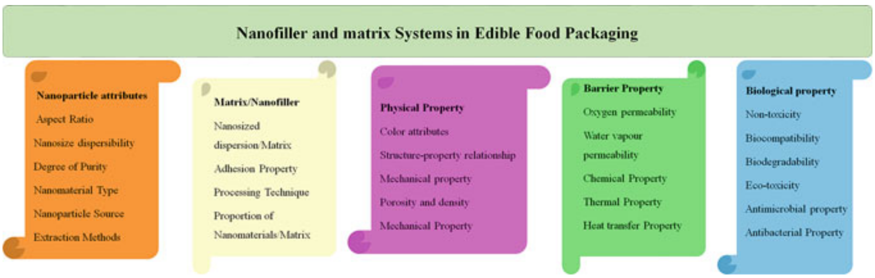

Fig. 9.2 Biopolymer nanocomposite in edible packaging

aspect ratio, nanosized dispersion/matrix adhesion property, degree of purity, moisture content, nanomaterial type, nanoparticle source, dimensions, extraction methods, processing technique, proportion of nanomaterials and matrix, etc. However, the important consideration in designing and application of polymer biocomposite includes concepts, materials, processing, and others. In this regard, the nanomaterials with antimicrobial active property have attracted a great interest and an emerging field of application in edible food packaging sectors. Besides, polymeric nanocomposites in different forms such as films, coatings, and foams are utilized to develop edible packaging materials. The usability of polymeric nanocomposite in the field of edible food packaging acts as a promising candidate to improve the product attributes, reduces the synthetic packaging waste, preserves food products, and also increases the storage life. In this regard, the fabrication of biopolymeric nanocomposites and nanohybrids provides a great consideration for developing edible packaging with high performance in terms of several packaging attributes including barrier property, mechanical property, physical property, antimicrobial property, and others. The several polymorphs of polymeric nanostructured materials also influence the composite properties, such as the crystal structures, morphology, degree of hydrogen bonding, and others. In this regard, cellulose nanocrystal (CNC) polymorphs influence the properties of biocomposite-based polymer properties such as barrier property, mechanical property, thermal property, and structural attributes [13]. The different dimensions of nanofiller materials such as spheres, fibers, particulates, and flakes influence the packaging attributes. The worldwide status of preparing nanosystem-assisted edible food packaging provides a motivating way to develop new polymeric composite materials for targeted edible food packaging materials. The several disadvantages of polymer nanocomposites for improper reinforcement include poor dispersion, non-optimum proportion, weak interface, random structure, and others. The benefits of developing polymer nanocomposites for targeted edible food packaging are aligned structure, well-dispersed composite, better surface interaction, etc. The use of biopolymeric nanofibers, nanocrystals, and nanoparticles has the capability to create a percolation network within the matrix and help to increase the stability of the film. The polymer nanocomposite involves the use of nanometer size dispersion which delivers biofunctional attributes to the composite materials for developing edible food packaging. The use of hydrophobic agents in biopolymeric nanocomposites such as glycerol and other antimicrobial agents improves the edible packaging attributes in terms of physical, mechanical, barrier, and other functional properties. The inorganic nanomaterials in developing biopolymer composites are generally used as additives in biopolymer matrix to improve the shortcomings of biopolymer composites. The other important attributes of biopolymer which are obtained by reinforcing nanofillers are dimensional stability, gas resistance property, tunable surface property, transparency, etc. However, the main aspects of developing bionanocomposites are biodegradability, renewable resources, acts as a carrier of antimicrobial agents, enhanced shelf life, reduced volume, weight and waste of packaging, etc [14].

9.4.1 Physical Properties

The physical properties of polymer nanobiocomposites-based packaging materials include molecular weight, crystallinity, degree of polymerization, density, molar volume, and others. Further, the binary films for developing edible films and coatings are polysaccharide–protein, protein–protein, polysaccharide–polysaccharide, lipid-added polymeric nanocomposite, and others. In this context, polymeric complex-based films have attained a great effectiveness in developing bilayer films to improve the property of perishable food products and others. The use of plasticizers in the fabrication of biopolymer nanocomposite also provides tunable properties in developing edible food packaging. The inclusion of biopolymeric nanofibers in fabricating polymeric nanocomposites provides improved mechanical properties such as tensile strength, thermal properties, optical properties, and others [1]. The gelatin-based edible films have poor mechanical properties, where the incorporation of nanofillers such as cellulose nanocrystal (CNC) and bacterial cellulose nanocrystal (BCNC) to develop biocomposite can improve the mechanical property such as tensile strength and tensile modulus. The improved mechanical property of the gelatin biocomposite films is attributed due to the high crystallinity of cellulose nanomaterials. As discussed in Chap. 3, the noteworthy properties of nanocellulose in terms of dimension, mechanical strength, functionality make it a potential nanofiller to be used in developing biocomposite-based edible films. The tensile strength of BCNC-reinforced gelatin films is $\sim 83.7 \pm 3.2$, $\sim 88.7 \pm 4.8$, $\sim 95.1 \pm 3.9$, $\sim 103 \pm 4.7$, and $\sim 108.6 \pm 5.1$ MPa, respectively, for 0, 1, 2, 3, and 4% BCNC content. Additionally, the tensile modulus of BCNC-reinforced gelatin films is $\sim 2189.5 \pm 50$, $\sim 2225.3 \pm 63$, $\sim 2272.8 \pm 68$, $\sim 2335.1 \pm 59$, and $\sim 2350.4 \pm 65$ MPa, respectively, for 0, 1, 2, 3, and 4% BCNC content [15]. Further, the mechanical performance of some polysaccharide-based films such as pullulan can be tailored using functional agents, essential oils, metal nanoparticles, and others. The physicochemical attributes of biopolymers can be improved using nanoclays (montmorillonite), where the dispersion of this kind of nanofillers can be improved by applying sonication. Further, the application of sonication in some nanofiller dispersion helps to avoid the agglomeration property of nanoreinforcements in matrix for developing edible films and coatings.

9.4.2 Barrier Properties

The water barrier properties of polysaccharides are commonly enhanced by developing composites with hydrophobic compounds such as fatty acids and mixture of lipids. The alginate is used to develop film where the film properties are associated with the formation of strong gel, when multivalent metal cations (such as calcium ions) are present. Interestingly, the interactions between carboxylic group of alginate and calcium ions help to form a cross-linked network. The alginate-based coating materials are found to have low oxygen permeability and can be added to other biopolymers having poor oxygen barrier properties. Further, the water vapor permeability of polysaccharide-based nanocomposites can be tailored using nanomaterials such as cellulose nanomaterials and others. The fruit puree-based films can also provide improved barrier properties when nanofillers are incorporated to develop polymer nanocomposite films. The fish protein has the ability to form networks which provide improved packaging properties such as good oxygen barrier properties, whereas the poor water vapor permeability of fish protein can be tailored with the aid of nanofiller materials.

9.4.3 Functional Attributes

The functional attributes of polymer biocomposite-based edible films and coatings can be improved using metal oxide-based nanofillers. The metal oxides are commonly used to provide antimicrobial, antiradiation, antibacterial, and other properties. The matrix and filler interaction can be tailored via electrostatic interaction, hydrogen bonding, and others. However, the metal oxide should be used in optimum concentration; otherwise, there may be an agglomeration within the matrix, which may result in discontinuity in films and coating properties and film network structure. The bioactivity of some polysaccharide films can be improved using several functional agents. Additionally, the several proteins such as gelatin, lysozyme, casein, and soy proteins are used as a blend or nanocomposite to provide functional packaging. The various biological activities of edible films and coatings are antioxidant, antimicrobial, antibacterial, anti-inflammatory, antihydrolytic activity, antiviral, antitumor, and others.

9.4.4 Antimicrobial Properties

The different types of antimicrobial agents widely used in nanocomposite-based edible films and coating include fatty acid esters, organic acids, polypeptides, bacteriocins, plant essential oils, and sulfites. The antimicrobial properties of polymer nanocomposite for targeted edible food packaging commonly involve the use of inorganic nanofiller materials such as titanium dioxide, zinc oxide, iron oxide, silver nanoparticles, and others. However, the inorganic nanofillers for developing edible packaging should be within permissible limits. The metal oxides are extensively used to deliver antimicrobial attributes to edible films and coatings. Additionally, titanium dioxide is effective against allergens and foodborne microorganisms. On the other hand, the polycationic nature of chitosan and nanochitosan has an ability to make a conjugate with other components, which helps in improving the antimicrobial property of chitosan. However, the antimicrobial activity of nanochitosan is better than chitosan. Additionally, the antimicrobial activity of chitosan nanoparticle is found to be increased when metals are loaded such as silver nanoparticles. Additionally, the small-sized silver nanoparticles provide better antimicrobial property than large-sized silver nanoparticle; thus, silver nanoparticles are widely utilized to develop biopolymer composites to be used as edible films and coatings. Besides, the active components used for developing edible films and coatings are N-acetylcysteine, glutathione, cinnamon oil, rosemary oil, lemongrass essential oils, palmarosa essential oils, ascorbic acid, silver–montmorillonite nanoparticles, citric acid, sodium benzoate, α-tocopherol, potassium sorbate, sodium benzoate, nisin, trans-cinnamaldehyde, etc. The incorporation of antimicrobial agents in biopolymer nanocomposites makes it a remarkable candidate against microbial spoilage to improve the shelf life of food products.

9.4.5 Surface Properties

The application of nanocomposite-based edible coating helps to reduce the oil uptake, reduce water loss, improve sensory property, reduce fat contents, and better nutritive contents in fried food products. The bionanocomposite-based edible coatings are considered as a potential candidate to deliver functional compounds, which reduce the oil uptake ratio [16]. The various coating materials used for reduced oil uptake ratio include egg white, gelatins, sodium caseinate, soy protein isolate, wheat gluten, whey protein isolate, whey protein concentrate, etc.

9.5 Recent Advances of Biopolymer Nanocomposites in Edible Food Packaging

The unique properties of nanostructured materials in terms of mechanical, optical, surface property, nutritional attributes, and others make them a potential candidate in developing bionanocomposite-based edible food packaging with tailor-made properties for highly perishable food products (Table 9.1). The development of edible coatings on acerola fruit and edible films using acerola puree, alginate, cellulose whisker (CW), or montmorillonite (MMT) as a nanofiller provides reduced water vapor permeability of films and coatings on fruit and helps to reduce the weight loss of fruit and ripening rate [17]. The edible nanocoatings based on the nanocomposites of xanthan gum, zinc oxide, and stearic acid have superior antibacterial property and have non-toxic nature [18]. Additionally, the application of this kind of edible nanocoating on apples and tomatoes has negligible weight loss at ambient conditions. The nanocomposite-based edible biodegradable films developed using carboxymethyl CNC and cassava starch have improved mechanical, physicochemical property and provide a cohesive structure to the developed films, where the use of carboxymethyl CNC (0.4 g/100 mL) and cassava starch improves the mechanical property in terms of tensile strength by 554% and water solubility by 123% [19]. The nanocomposite edible films based on HPMC, beeswax, clay, thai essential oils (ginger, finger roots, plai) have a good antimicrobial property and can be used as an active coating for agricultural produces [20]. The HPMC and chitosan/tripolyphosphate (TPP)-based edible films are also preferable due to offering enhanced mechanical, barrier, and thermal stability in comparison with the neat HPMC films [21].

Moreover, the use of fish protein and organo-clay (montmorillonite)-based nanocomposite as an edible coating material on fresh-cut papaya (minimally processed) improves the quality in terms of weight loss, color, microbial count, firmness, lightness during storage (12 days and 5 °C) [22]. Additionally, the edible films based on nanocomposites of *Salvia macrosiphon* and nanoclay are a kind of antimicrobial films and provide improved physical, thermal, and mechanical property [23]. Further, the addition of nanoclay increased the hydrophobicity of *Salvia macrosiphon* seed mucilage films, which can act as a replacement for food packaging materials. The development of edible coating using silver–chitosan-based nanocomposite on fresh-cut melon provides better keeping quality (storage condition: 13 days and 5 °C) such as reduced respiration rate, better sensory property, and reduced mesophilic count, and also enhances the shelf life of fresh-cut melon [2]. The application of coating on cashew nut kernels using **s**tarch, cashew tree gum, and nanoclay helps to reduce the moisture loss, from cashew nut and also a good coating material in extending the stability of cashew nut kernel [24]. On the other hand, the pullulan and lysozyme nanofibers-based nanocomposite films have good mechanical attributes of films (Young's modulus of 1.91–2.50 GPa), good thermal stability till 225 °C, antioxidant activity in terms of 2,2-diphenyl-1-picrylhydrazyl (DPPH) scavenging activity of 77%, antibacterial activity against *Staphylococcus aureus*, and others [25]. Further, the nanocomposite edible films based on whey protein isolate and titanium dioxide have water vapor permeability of $\sim 3.19 \times 10^{-10}$ g s^{-1} m^{-1} Pa^{-1}, 2.89×10^{-10} g s^{-1} m^{-1} Pa^{-1}, and 2.92×10^{-10} g s^{-1} m^{-1} Pa^{-1}, respectively, for 0, 1, and 2 w/w % of titanium dioxide [27]. Similarly, the nanocomposite edible films based on whey protein isolate and titanium dioxide have solubility of $\sim$17% and 15%, respectively, for 0, and 1 w/w % of titanium dioxide [27].

Table 9.1 Biopolymer nanocomposite-based materials for edible food packaging as edible films and coatings

Sl. no	Components	Type of packaging	Property	References
1.	Fish protein	Edible coating on minimally processed cut papaya	Low cost	[22]
	Nanoclay		High shelf life	
			Maintained quality of minimally processed cut papaya	
			Improved appearances	
2.	Silver–chitosan nanocomposite	Edible coating on fresh-cut melon	Reduced shelf life	[2]
			Better sensory quality	
			Reduced mesophilic count	
			A potential coating for fresh-cut fruit products	
3.	Acerola puree	Edible coatings on acerola fruit and edible films	Provide reduced water vapor permeability of films	[17]
	Alginate		Coatings help to reduce the weight loss of fruit	
	CW		Coating helps to reduce ripening rate	
	MMT		Coating also significant in retaining ascorbic acids	
4.	Gelatin	Edible films	Improves mechanical property	[15]
	BCNC		Improve thermal property	
			Reduce moisture affinity of gelatin	
5.	Starch	Edible coating for cashew nut	Use of MMT improves the moisture barrier attributes	[24]
	Cashew tree gum		Improved oxidative stability of kernels	
	MMT		Improve mechanical property	
			Provide reduced moisture content of coated cashew nut (120 days storage)	
			Decrease textural changes of coated cashew nuts	
6.	Whey protein isolate	Edible films	More than 70% visible light can be blocked	[26]
	Titanium oxide		More than 90% UV light can be blocked	
			Tailored tensile properties of the nanocomposite films	
7.	HPMC	Edible films	The nanocomposite films with plai and finger root-based	[20]
	Thai essential oils (ginger,			

(continued)

Table 9.1 (continued)

Sl. no	Components	Type of packaging	Property	References
	finger root, plai)		essential oils increase the oxygen permeability	
	Organically modified clay		A kind of active packaging	
	Beeswax			
8.	Pullulan	Active edible food packaging	High mechanical performance	[25]
	Lysozyme nanofibers		Thermal stability	
			Antimicrobial property	
			Antioxidant property	
9.	Cassava starch	Biodegradable edible films	Improved physicochemical property	[19]
	Modified CNC		Improved barrier property	
			The bionanocomposite film has cohesive structure	
			Can be a potential candidate in developing water-soluble films	
10.	Salvia *macrosiphon*	Edible nanocomposite films	Increased hydrophobicity	[23]
	Nanoclay		Improved mechanical and thermal property	
	Glycerol		Antimicrobial films	
11.	Xanthan gum	Edible nanocoatings	Superior antibacterial property	[18]
	Zinc oxide			
12.	Chitosan/TPP nanoparticles	Edible films	Improved barrier property	[21]
	HPMC		Improved mechanical property	
			Improved thermal property	

CWCW Cellulose whisker; *MMT* montmorillonite; *BCNC* bacterial cellulose nanocrystal; *CNC* cellulose nanocrystal; *TPP* tripolyphosphate; *HPMC* hydroxypropyl methylcellulose

9.6 Application of Nanotechnology in Edible Food Packaging

Considering the environmental concerns associated with the consumption of conventional plastics, biobased polymers have found application in the food packaging industry because of their inherited sustainability and biodegradability [27, 28]. Recent exploration of nanotechnology in edible packaging promises fascinating improvement in the safety and quality of food by improving its mechanical strength, barrier properties, and shelf-life. However, food packaging derived from natural polymers has limitations in terms of barrier properties and mechanical strength; thus, the preparation of a protective edible coating involves blending of the edible polymers with other components, basically bioactive agents and plasticizers and

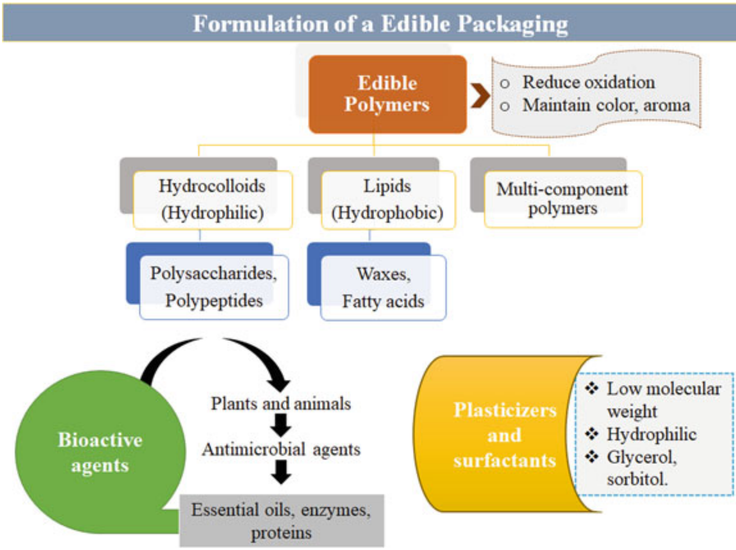

Fig. 9.3 Formulation of a protective edible packaging

surfactants, as shown in Fig. 9.3 [29]. Edible coatings are thin layers applied on the food surface, either by spraying, solvent evaporation, immersing the food into a coating solution or by rubbing the food with the coating material to maintain the features and properties of the food, thereby increasing their shelf life. These edible coatings should be compatible with the food on which they are applied, in an organoleptic manner, and do not always require to be removed from the food surface before consumption [30].

Nanotechnology involving the use and incorporation of the nanomaterials in the preparation of edible coating helps to enhance the barrier properties, antioxidant activity, and mechanical stability which thereby eliminates packaging waste and improves the shelf life [31]. Figure 9.4 discusses the various attributes which are expected to improve with the introduction of nanotechnology in the food packaging industry. With the intention to improve the quality of edible food packaging, nanomaterials play an important role in the preparation of the packaging material. The nanomaterials, often considered as functional materials, possess one of their dimensions in nanometer scale (nm) and are basically categorized as nanoparticles (NPs), nanocrystals (NCs), nanofibers (NFs), nanocolloids, micro- and nanoemulsions [32]. Based on their preparative techniques, distinct physical properties of nanoparticles could be achieved and accordingly they are used in the food industry.

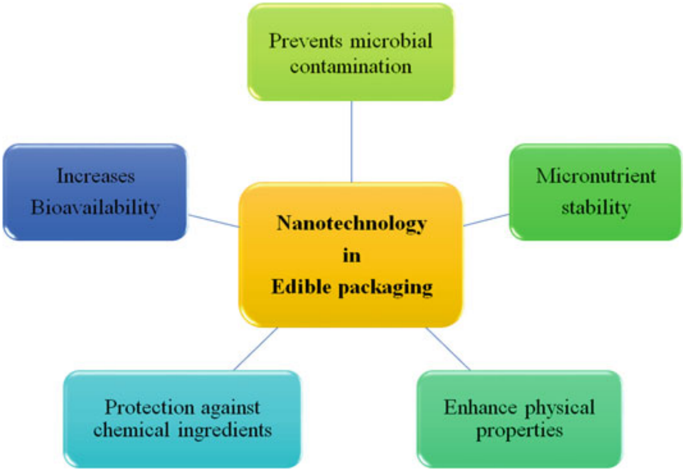

Fig. 9.4 Functionality of nanotechnology in edible packaging

9.6.1 Nanosystems in Edible Packaging

Nanotechnology opens new doors of possibilities in food industry basically in edible packaging by promoting the transport of essential oils and fat-soluble vitamins, incorporated with antimicrobial properties, thus improving the mechanical stability and transparency of edible coatings with the easy transport of gas molecules. The nanometer ranges of materials used in nanosystems make them more stable and do not alter the organoleptic property of the food; also, their higher aspect ratio enables the bioavailability or mobility of the encapsulated nutrients as compared to microsized particles [33].

The nanosystems used in edible packaging basically involve polymer nanoparticles, solid–lipid nanoparticles, nanofibers, nanotubes, nanocrystals, and their nanoemulsions; these nanosystems when embedded into the polymer matrix are commonly termed as polymer nanocomposites and act as a barrier in the controlled release of components to the environment. Polymeric nanosystems with different properties are formulated with the help of nanotechnology/edible coating technique, which encapsulates bioactive molecules and when incorporated in edible packaging acts as preservatives, food enhancers, and nutrient supplements. In this regard, some of the nanostructured materials such as nanocapsules, nanosphere, nanoemulsions, nanofibers, nanoparticles, and organic/inorganic nanofiller have been represented in Fig. 9.5.

Polymeric Nanoparticles. Depending on their structure and morphology, polymeric nanoparticles are categorized as nanospheres and nanocapsules. In nanospheres, bioactive molecules are trapped and dispersed into the polymer matrix, whereas in nanoencapsulation oil core in which the bioactive molecules are retained is surrounded by a polymer membrane. The selection of polymer plays a

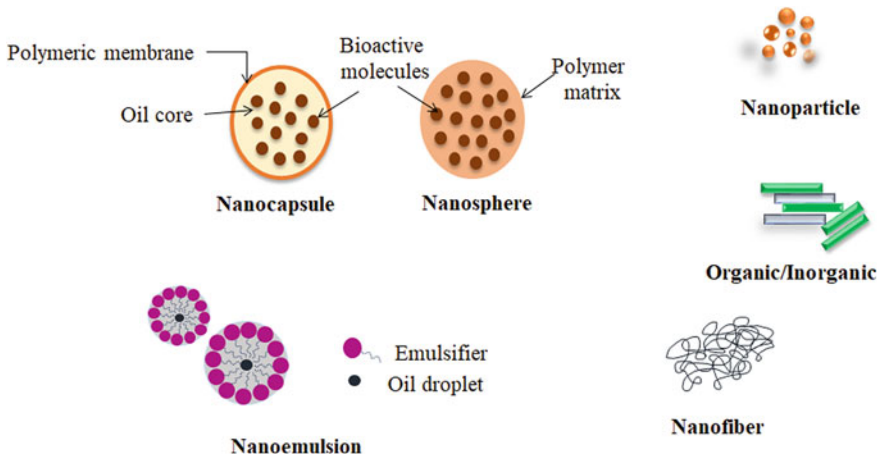

Fig. 9.5 Nanostructured materials for biopolymeric composite-based edible food packaging

critical role in both nanosphere and nanocapsule systems, such as encapsulation efficiency, release of the bioactive component, protection ability, and degradation process. Polymers commonly used in the food industry for the preparation of polymeric nanoparticles are polylactic acid, poly(ε-caprolactone), chitosan, and alginate [30]. Synthesis of chitosan nanoparticles and their application in edible packaging has been widely practiced recently due to the biodegradable, biocompatible, non-toxicity, and natural abundance of chitosan [28]. Because of its solubility in water at lower pH, film-forming ability, and antimicrobial, chitosan is the most widely practiced polymer in edible packaging industry. The edible coating of chitosan can be prepared by dip coating or simply by spraying the chitosan nanoparticle into freshly cut fruit. Curcumin, a common type of polyphenol with its inherent antioxidant and antimicrobial characteristic, acts as an active molecule which can be easily incorporated into nanocapsules in edible packaging. Essential oils are largely used in the preparation of edible coatings because of their antimicrobial activity, and some of the common examples are lemongrass oil, turmeric oil, peppermint oil, etc [30]. The utilization of nanotechnology in the food packaging industry has an exceptionally advantageous impact on the society in terms of reduction of environmental impact, cost-effectiveness, and lower energy consumption. However, depending on the ethical responsibility of using these nanomaterials in edible packaging, a better knowledge of the effects of these materials on the health and environment has to be considered [31].

9.7 Synthesis Strategy for Modification of Polymer Composite

The interest in the utilization of polymer nanocomposite in edible food packaging is tremendously increasing. The demand of supplementing natural and acceptable food packaging, built on economically feasible, effortlessly processed, accessible, and ready-made fresh foodstuffs, has given rise to an increase in customer needs for healthy and high-grade quality food product [32]. To meet up with these consumer requirements and improvements of the quality of edible food packaging, certain strategic modifications are shown in Fig. 9.6. These trends lead to the variations in the normal procedures of food processing. The growing interest to get food products with an extended storage life, with improved properties related to the packaging materials, leads to the preparation of polymer composites blended with active agents, functional additives, plasticizers, cross-linkers, and compatibilizers among other things [1]. This will result in better edible packaging products.

9.7.1 Binding Agents

A binder or a binding agent is a kind of material that tends to hold components of the polymer composites together to improve its mechanical and chemical properties. Commonly, the interfacial linkages in nanocomposites are a critical concern; thus, the interface of the polymer matrix with the filler (nanoparticles) needs to be interacted strongly, which can alter the polymer flexibility and mobility. This indicates that to improve the interaction and reduce the chain, use of a food-grade binder can be effective in edible food packaging. Especially, to maintain the mechanical and barrier properties, the use of binders and compatibilizers serves as a good candidate for edible packaging [33]. In this regard, the surface-active substance provides a route toward linking the chains within the polymer composite; this results in stronger mechanical behavior, increased texture, glossiness, and surface smoothness [34]. These can be obtained by using different types of binding agents such as gluten, gums, agar, gelatin, and starch jellies. Additionally, the

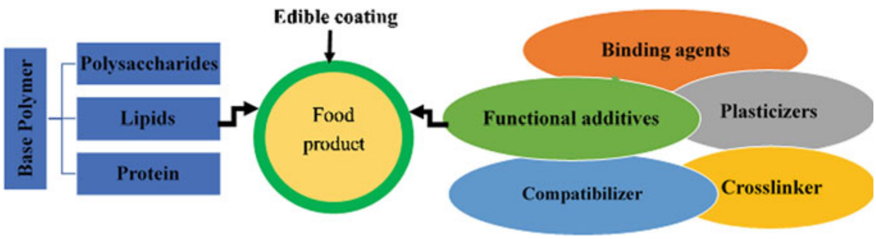

Fig. 9.6 Edible polymer composite constituents

binding agent is essential for the stabilization, thickening, and networking of the polymer composite chains [33]. Thereby, a binding agent serves as an important agent in the modification of edible food packaging to boost the physical, mechanical, chemical, and handling characteristics to deliver desired functionalities of the films and the products.

9.7.2 Plasticizers

Plasticizers are additives used for modification and improvement of the edible food packaging. Many polymeric materials used for packaging and film coating are stiff in nature. To resolve this challenge, plasticizers are blended to tailor the plasticity and pliability of the film [35]. This is achieved basically by reducing the intermolecular forces and intensifying the motion of polymer chains, which in turn tends to enhance the mechanical characteristics of the edible polymer composite. The use of plasticizers in the fabrication of biopolymer nanocomposite also provides tunable properties in developing edible food packaging [1]. Common plasticizers for improving the polymer composites for edible food packaging and layers are comprised of polyols, oligosaccharides, monosaccharides, lipids, and derivatives. Although plasticizers improve the plasticity, mechanical properties and reduce the friction during handling, and it also increases film permeability which affects the barrier properties. Therefore, a suitable selection of plasticizers for a specific material would enhance the performance of the films and also help to achieve the desired effect with the least permeability. Hence, the choice of plasticizers is governed by many factors such as plasticizer's compatibility, effectiveness, stability, and cost, while the performance of the plasticizer depends on the type, concentrations, interactions between the plasticizers and the polymer matrix, etc [36]. Based on these, the suitable selection of plasticizers can deliver effective film properties for targeted application.

9.7.3 Cross-linkers

The chemical networking agent is known as cross-linkers and is beneficial in modifying polymer composites. The reduced polymer movement and effective attributes of polymers can be obtained by the application of cross-linkers via chemical, physical, or biological treatment [35]. The properties of polymer composite film are determined by the chemical and physical nature of the constituent polymers. Most individual polymers have distinct characteristics. To achieve a desired edible food packaging, film, and coating, a blend of polymer composite with different desired properties is required to attain, where the improved functionality is an essential factor and cross-linkers have a remarkable role in this regard. The improved synergetic interaction can be obtained between the different components of the polymer composites using cross-linkers. Thus, cross-linkers are introduced to generate firm networking assembly of the polymer chains for enhancing perpetual characteristics within the polymer composite [37]. The chemical bonding generated by covalently bonded polymer chains generally forms firm network together. Hence, they normally do not split or soften in aqueous solutions. The characteristics of the subsequent polymer composite after the introduction of cross-linkers depend on the degree of the cross-linking pattern. Various networking agents have been utilized as cross-linkers in the processing of edible polymer composite for packaging and coatings such as formaldehyde, glutaraldehyde, carbodiimides, genipin, glyoxal, and transglutaminase. Among all, formaldehyde, the common networking agents, has the widest chain linking specificity. Though formaldehyde encompasses a single functional group, it can link in more than single reaction and consequently cross-link the targeted compound [38].

9.7.4 Functional Additives

Functional additives are groups of organic or inorganic substances incorporated into polymer biocomposite to enhance the functional attributes of edible films and coatings. This can be attained by the application of metal and metal oxide-based nanofillers. The metal oxides are commonly used to provide antimicrobial, antibacterial, antiradiation, and other properties. The polymer matrix and filler interaction can be tuned through different interactions, like hydrogen bonding, and others to achieve the desired outcome. Biopolymer nanocomposite and nanoparticles have advantageous applications in the food and other related manufacturing companies. This is because they have the tendencies to be used as a health-benefitting delivery system, encapsulating candidates, texture agents, and lightening modifiers [39]. The functional additives are also applied to serve as bioactive ingredients. In some instances, specific reactive agents or catalytic agents are imbedded as modifiers. Furthermore, some class of polysaccharides (dietary fibers) has been revealed to possess health-benefitting attributes, like reduction in cholesterol, prevention of cancer, or general improvement in human well-being [40]. Therefore, ingesting of biopolymer nanoparticles loaded with the polysaccharides may benefit human well-being. However, the bioactivity of some polysaccharide films can be improved using several functional agents. Additionally, various proteins such as gelatin, lysozyme, casein, and soy proteins are used as a blend of nanocomposite to provide functional packaging. The various biological activities of edible films and coatings such as antioxidant, antimicrobial, antibacterial, anti-inflammatory, antihydrolytic activity, antiviral, antitumor, and other benefits can be obtained [41]. Consequently, increased attention is generated toward the advancement of numerous types of functional biopolymer additives for viable applications.

9.7.5 Compatibilizers

The edible compatibilizer helps to generate and improve the plasticization of the edible polymer composite film. The incompatibility between the polymer matrix and its disperse phase can lead to poor dispersion or agglomeration of the nanocomposite within the polymer matrix [42], thereby resulting in low moisture resistance, poor performance, and limited polymer composite interactive properties. Therefore, to enhance the interaction between the polymer matrix and its disperse composite interface, suitable compatibilizers can be introduced in the polymer matrix. The several appropriate compatibilizers are comprised of, but not restricted to the following: lactic acid (LA), adipic acid, high molecular weight polyethylene glycol (PEG), and polysorbate [42]. In recent times, due to environmental consciousness researchers are exploring the application of ecologically friendly fillers as compatibilizers in polymer composites. Additionally, an economically viable means to depend on the utilization of natural compatibilizers as a substitute for typical conventional compatibilizers. In this respect, vegetable oils are a fascinating group of modifiers that can deliver a dual benefit of plasticizing and a compatibilizing outcome.

9.8 Effective Properties of Nanoparticles in Edible Packaging

The growing demand for increased fresh food shelf life as well as the need for protection against foodborne diseases urged the development of antimicrobial food packaging. Among the most efficient methods, the combination of organic–inorganic and packaging, i.e., polymer embedded metal nanoparticles, proved to be highly effective. This is normally done by incorporating the active inorganic nanoparticles within the polymer matrix to be used in the proposed application.

9.8.1 Metal Ion Release Property

The importance of inhibiting foodborne diseases and various food-grade agents is applied on food products. Among available, the need of synthesizing the antimicrobial edible polymer film for food packaging is of great interest. The point of concern is the modification of packaging materials with unique nanoparticles that would deliver active biocide constituents to its surrounding [1]. In order to enhance the life span of the food and its packaging, and delaying the decomposition process, the application of either inorganic and/or organic substances is remarkable [43]. The inorganic nanoparticles are primarily silver, iron, or other metals and their oxides, and the organic nanoparticles are mainly carbon-based acid, polymers, and enzymes. The carbon-based antimicrobial substances are unstable at elevated temperatures in comparison with the inorganic counterparts because the nanoparticles of metal and their oxides withstand higher thermal processing situations and handling. This indicates that metal-related nanoparticles are more preferred for the controlled release of the nanoparticles in the polymer composite for the antimicrobial process. Especially, silver nanoparticles have antifungal, antiviral, antimicrobial, and antiyeast activities [43]. Hence, it can be blended with edible polymer composite for beneficial antimicrobial edible food packaging. Additionally, the metal ion release property is also required in an edible food package. The effectiveness of delivering silver, iron, and other metal ions using polymer biocomposites is very crucial. The ability of polymer nanocomposite to release metals is dominant to restrict microbial activities.

9.8.2 Defensive Mechanism

The defensive mechanism of polymer composites in edible packaging is a required attribute. The various food supplements can be delivered with other requirements via including edible food packaging and can further provide improved characteristics. The active additives utilized in polymer composite are edible packaging and coatings, which consist of antiviral, antimicrobials, antioxidants, and biological agents that regulate microbial development, prevent oxidative degradation, and prefer a targeted biocide that preserves food. The nanocomposite and other additives used in edible packaging encompass phenolic and aromatic moieties that are capable of antibacterial activities and antioxidant properties. These compounds react with their surroundings and affect their antimicrobial activities. Chitosan has been established for its antimicrobial activities, and it hinders the development and growth of fungi by triggering a defensive mechanism in fruits against contaminations initiated by some pathogens [44, 45]. The various mechanisms were proposed for the antimicrobial properties of nanoparticles. The interactions and interruption of the microbial cells with the biocides and the transmembranes can have different effects such as destructing the cell cover and oxidizing membrane cell, and produce responsive oxygen species. For instance, the antimicrobial properties of silver nanoparticles in protein-rich products can bind with the cysteine, lysine, arginine, and methionine components [41]. Silver nanoparticles are elucidated by adhesion to the surface of the cell, damaging lipopolysaccharides and degrading the cell, basically increasing absorptivity by penetrating membrane cell, discharging antimicrobial molecules, and destroying the DNA.

9.8.3 Health Benefits

One of the fundamental issues in food processing and packaging is the protection against foodborne diseases which still represent a worldwide issue of general well-being. This is carried out by the delivery of bioactive compounds using biopolymer composite. Polymer nanoparticles and colloids are accumulated from single or multiple biopolymer compounds, which have some possibilities for applications in the food manufacturing process. The composite could be utilized to encapsulate bioactive constituents for the controlled release of health-benefitting ingredients like macro- and micronutrients to the human system [39]. The delivery principle is governed by the concentration of the active constituents encapsulated within the polymer composite. The loading effectiveness depends on the binding sites accessible for phenolic compounds. Further, antidiabetic, antimicrobial, anti-inflammatory, antihydrolytic activity, antiviral, hypolipidemic, antitumor, and antioxidant properties can be linked to polyphenols when consumed at the appropriate level [46]. Biopolymer nanocomposites play a vital role as a delivery system for delivering encapsulated nutritional and nutraceutical health-benefitting compounds manufactured from edible food packaging. The strategic alteration of polymer composite may lead to the advancement of edible polysaccharides and protein-based food packaging with novel bioactive materials that can enhance human well-being and global health.

9.9 Recyclability of the Edible Food Packaging Material

The global yearly consumption of plastic has greatly increased, among which about 40% of the products are used in packaging. This draws attention from environmentalists and policymakers. In this regard, the continuous research and developments are going on to reform the ways of handling waste plastic to create economic and environmental sustainability [47]. The increasing recycling capacity of waste plastic is presently a primary point on the world agenda of global sustainability. Food packaging has a substantial portion, reducing or recycling would yield a significant decrease in solid waste generation, and it may also reduce cost. The present trend in edible food packaging manufacturing is continuously putting efforts that the packaging materials should be natural, edible, functional, economical, and ecologically friendly. Therefore, the mentioned properties when achieved provide biodegradable and eatable film or coatings. This results in substantial interest in edible packaging due to their sustainability and possibilities in reducing conventional non-degradable plastic food packaging. Hence, the food-grade edible biopolymer composite is not only recyclable but also edible, stable, safe, with enhanced storage life. Consequently, this can reduce the nominal values of waste plastic generated via food packaging and will supplement or substitute the conventional plastic with the biodegradable polymer nanocomposite. Biopolymer nanocomposite can be reused and/or valorized in combination with other biological waste in composting amenities, which will generate manure as valuable conditioners and fertilizers for soil amendments [48].

9.10 Application of Biopolymeric Nanocomposite in Edible Food Packaging

The application of edible packaging is gaining momentum in recent times because of its numerous advantages. The issues related to environment, waste generations, food quality, ethics, and product cost are becoming more important to global sustainability and present-day consumers. This leads to the synthesis and strategic modification of polymer composite for edible food packaging to meet the global challenges while satisfying consumers' needs. The use and availability of polymeric composites have increased in several fields including both edible and non-edible food packaging. Researchers and scientists have recognized polymer nanocomposite in preparation of edible film for food packaging in addition to the conventional means of packaging to improve beneficial characteristics, reduce risk and waste, and enhance safety and storage life. This is normally done by incorporating modifiers with targeted applications into the polymer matrix to tune the properties of the polymer composites for the desired application. The fruits, vegetables, meat and meat products, fish and fish products, and dairy products are attracting more attention in edible packaging due to their susceptible nature to microbial attacks. Therefore, one of the foremost applications of edible packaging apart from its environmental benefit is its antimicrobial effect. This is mainly achieved through the leading techniques in the preparation of edible food packaging such as spraying, dipping, and spreading [41]. Among them, spraying is the one with high demand in industrial packaging in comparison with spreading or dipping because of some main issues like the economy and quality of the finished product. The possibility of reducing cost by the application of the spraying method and the superiority of the finished packaging are major factors. Nevertheless, coatings still have challenges in food packaging applications owing to their inadequate barrier to water vapor and poor mechanical characteristics. Hence, the application of polymer composites for edible food packaging via blending with various biopolymers, nanoparticles, incorporation of hydrophobic constituents like oils or other chemical amendments of the polymer composite through structural adjustment has been projected to take care of the challenges in the application of edible food packaging.

9.11 Future Trends and Conclusion

The fabrication of various kinds of edible packaging using biopolymeric composites has been done by several researchers. Its efficacy and usefulness have been identified by the pharmaceutical and food manufacturers as a substitute or supplement to conventional packaging. However, the manufacturing of edible biopolymer films is typically at the research level and the large-scale application of edible biodegradable films at the commercial point is challenging in recent times, due to cost implications, water barrier properties, and other constraints. Thus, studies on large-scale economic feasibility are essential to encourage the full participation and commercialization of edible food packaging. Furthermore, food industries required a long storage life for foodstuffs and the commercial handling, and safety issues are also challenging that need to be studied further. The eatable packaging substances are themselves having bioactive property. Consequently, the defensive mechanism of biopolymer composites is viable for shorter periods than conventional packaging. Hence, the steadiness and handling behavior of edible packaging under specific conditions need to be examined systematically. However, recent advances show that metal and metal oxides are used as an active agent for antimicrobial activities within the edible package. This also draws concern from some quarters, about how much concentration of metal nanoparticles is safe and how best to achieve the controlled release of the nanoparticles for the targeted applications. Another limitation is the lower physical properties compared to the conventional ones. For instance, the sole lipid-based edible coating has better barrier behavior but lacks adequate mechanical properties. On the other hand, protein-based coatings have an organized structural matrix that supports better mechanical strength with low barrier properties. Therefore, there is a need to combine different substances to attain the desired outcome. In order to enhance edible food packaging, various materials and techniques are employed to achieve the desired consumer and manufacturer requirements. Nevertheless, the benefits and potentiality of edible films using eatable biopolymer nanocomposites have been appreciated and have a greater role to play in the future of sustainable packaging.

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Chapter 10
Nanoencapsulation: Prospects in Edible Food Packaging

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10.1 Introduction

The encapsulation is considered as a well-utilized technique to entrap several bioactive compounds (interphase) within carrier materials (external phase). The wall materials are also known as the outer shell, carrier materials, capsules, packing materials, films, etc., whereas, the internal phase is known as core material, actives, coated material, etc. As shown in Fig. 10.1, the arrangements of core (interphase) and wall materials (external phase) in nanoencapsulates of bioactive components provide several categories of nanoencapsultes such as (1) mononuclear containing an outer shell around the interphase, (2) multishell-based nanocapsules consisting more than one shell around the core materials, (3) multicore containing many interphases or core materials inside the shell, (4) matrix consisting uniformly distributed core materials within the carrier materials, etc. Additionally, the internal phase or the bioactive compounds are generally availed from several food extracts or others and inclusion of encapsulation process protects different types of food components from environmental conditions, or helps in masking the unpleasant taste, and odors. The bioactive components obtained from several sources are incorporated into different food systems such as fruit juices and other food beverages. However, the shortcomings for bioactive components to be used in food products and other industrial applications include low oral bioavailability, low solubility, poor stability against light, temperature, oxygen, etc. In this regard, the involvement of nanoencapsulation technique to encapsulate bioactive components can be a solution to overcome the mentioned shortcomings. The nanoencapsulation of bioactive components has further beneficial properties of improved dispersion, protection, convert liquid to powder, site-specific delivery, enhanced solubility/bioavailability, and others.

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The properties of nanoencapsulation depend on particle size, matrix materials, loading efficiency, compartment structure, surface engineering, digestion profile, and others. Additionally, the nanoencapsulation development has received a great interest in comparison with the microencapsulates preparation for delivering enhanced stability of capsules, increased encapsulation efficiency, and further, offer controlled delivery of interphase materials [1]. The various nutrient delivery systems in nanoencapsulation-based techniques are microemulsions, liposomes, microgels, macroemulsions, polymer nanoparticles, nanoemulsions, filled microgels, micro-clusters, multiple emulsions, etc. Among available, the nanoemulsion is a form of stable liquid dispersions, which have received a great interest in the delivery of drugs and lipophilic compounds. Interestingly, O/W-based nanoemulsions have received more interest than microemulsion for providing more benefits. The understanding of the physiochemical properties of nanoemulsions can help to provide a great application in several sectors of food industries; however, the nanoemulsion has thermodynamical instability, where, high energy is required to start emulsification process.

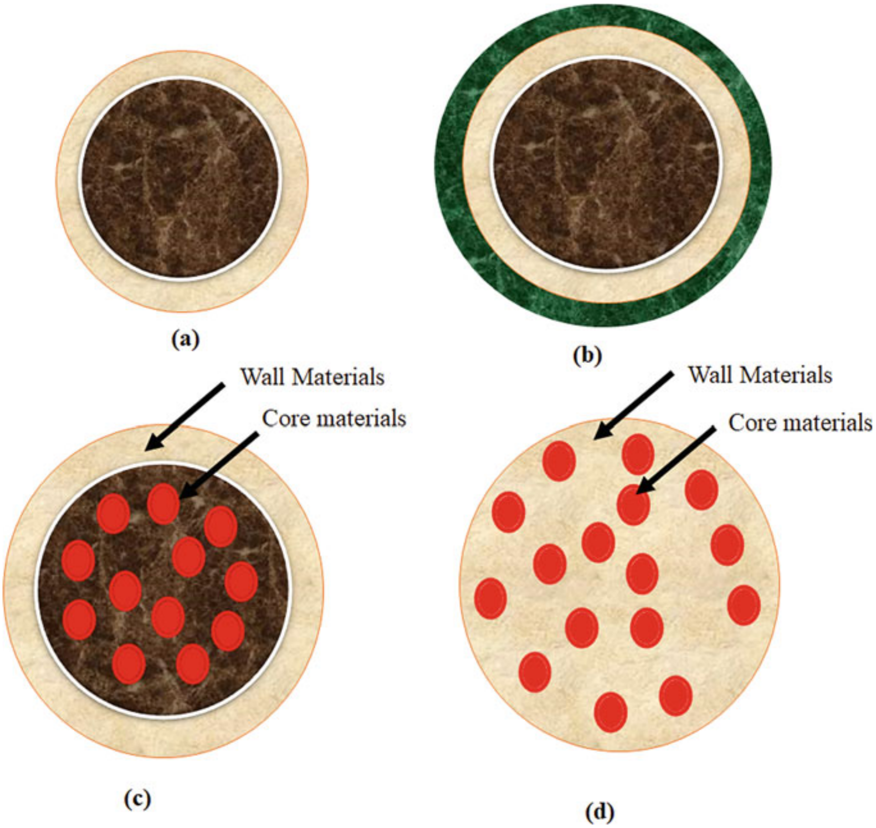

Fig. 10.1 Various structures of nanoencapsulates **a** mononuclear, **b** multishell, **c** multicore, and **d** matrix

Based on the above discussion, the current chapter aims to discuss the various targeted materials for developing nanoencapsulates to protect it from the environment. Further, the chapter also aims to discuss the various available carrier materials used for developing nanoencapsulates along with the targeted techniques. The current research and developments in nanoencapsulation include the characterization techniques and case studies for different active food components. The chapter also focuses to provide a brief overview of the existing techniques for developing nanoencapsulates of compounds.

10.2 Targeted Materials for Nanoencapsulation

As discussed in the earlier section, the nanocapsules consist of core materials (bioactive food compounds) within the wall materials in nanoscale ranges. The core materials which are targeted for developing nanoencapsulates are very sensitive to environmental conditions. The several hydrophobic bioactive compounds (Fig. 10.2) which are targeted for protecting via nanoencapsulation are phenolic compounds, vitamins, flavors, aroma compounds, essential oils, food colorants, etc. [2]. The bioactive compounds are extracted from oils, fruits and vegetables, legumes, and other plant-based sources [3]. The several methods for extraction of bioactive components are solvent extraction (for active compound extraction), microwave, ultrasonication, etc. The phenolic compounds targeted for encapsulation include phenolic acids, flavonoids, lignans, and others. The polyphenolic components are extensively utilized to fortify several food products for preventing the oxidative changes [4]. The natural colorant saffron is widely used in food and pharmaceutical industries for its antioxidant and therapeutic properties. The saffron has the major compounds crocin, picrocrocin, and safranal, which provides the mentioned characteristics attributes; however, the compounds are unstable at several environmental conditions, and the development of nanoencapsulates of this kind of natural colorants can enhance the stability in various environmental conditions. Additionally, the minerals help in building strong bones and are very essential for human body [5]. The main sources of essential minerals are macrominerals (calcium, chloride, magnesium, phosphorus, sodium, etc.), microminerals, and others, which perform several functions in human body. In this regard, the nanoencapsulation of minerals can enhance the bioaccessibility of bioactive ingredients in food systems.

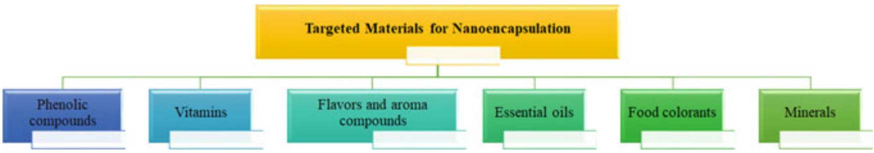

Fig. 10.2 Several targeted materials for nanoencapsulations

The vitamin complexes such as vitamin B complexes (B_1, B_2, B_5, B_6, B_7, B_{12}), vitamin C, vitamin D, vitamin E, and vitamin K are also targeted to preserve using nanoencapsulation and microencapsulation techniques for enhanced protection and controlled release of vitamins in different food products.

10.3 Carrier Materials for Nanoencapsulation of Bioactive Components

The carrier or wall materials used for nanoencapsulation should have some properties such as biodegradable, biocompatible, food grade, generally recognized as safe status (GRAS), and stable in food systems. However, the several required properties of wall or carrier materials for developing nanoencapsulates are represented in Fig. 10.3. As shown in Fig. 10.4, the several wall materials for the delivery of active ingredients include polysaccharides, their modified forms, proteins, lipids, emulsifiers, and others. Among the available wall materials for developing nanoencapsulates, the carbohydrate-based materials have attained a great interest as they can be modified according to the required attributes. The several carbohydrates and their modified form are extensively used to encapsulate bioactive ingredients such as meat flavor, Gallic acid, insulin, doxorubicin, indomethacin, bovine serum albumin, mint oil, human hemoglobin, lysozyme, quinine, and others [6]. The different sources of polysaccharide include plant, animal, algal, and microbial origin. In the earlier section, the details of the several polysaccharides-based materials have already discussed with their beneficial attributes and shortcomings. The several classes of carbohydrates are used for the development of nanoencapsulates of materials such as (1) modified starch: acetylated and succinylated starch; (2) modified cellulose: carboxymethyl cellulose, cellulose acetate butyrate, hydroxypropylmethyl cellulose phthalate; (3) pectin: oxidized pectin, pectin methylesterase modified pectin; (4) modified guar gum; (5) modified chitosan: trimethyl chitosan, linoleic acid-grafted chitosan; (6) modified alginate: alkyl ester alginate; (7) modified dextran; and (8) modified cyclodextrin: acrylic modified cyclodextrin, and others. The starch as a polysaccharide-based carrier materials is utilized for the encapsulation of flax seed, flavors, unsaturated fatty acids, insulin, flavors, and others food. The low cost, easy availability, biodegradability, and biocompatibility pure form makes starch a remarkable agent in developing nanocapsulates of several food components such as bioactive compounds. However, the shortcomings in the use of starch-based materials in the nanocapsulation formation are sensitive against acid attack, amylase hydrolysis, etc. The cellulose-based components can be modified physically, chemically, or biochemically to encapsulate the active compounds. The gum-based polysaccharides have a potential to be used as thickening, emulsifying agents and further used for food flavors and oils nanoencapsulations. Further, gum consists of hydrophilic and hydrophobic groups which make them a good candidate for nanoencapsulation of bioactive components. The cyclodextrins (CDs) consisting of hydrophilic outside surface and lipophilic central cavity, where,

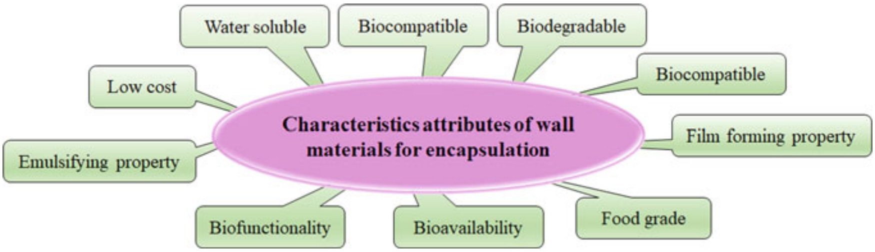

Fig. 10.3 Characteristics attributes of carrier or wall materials for fabrication of nanoencapsulates of bioactive food components

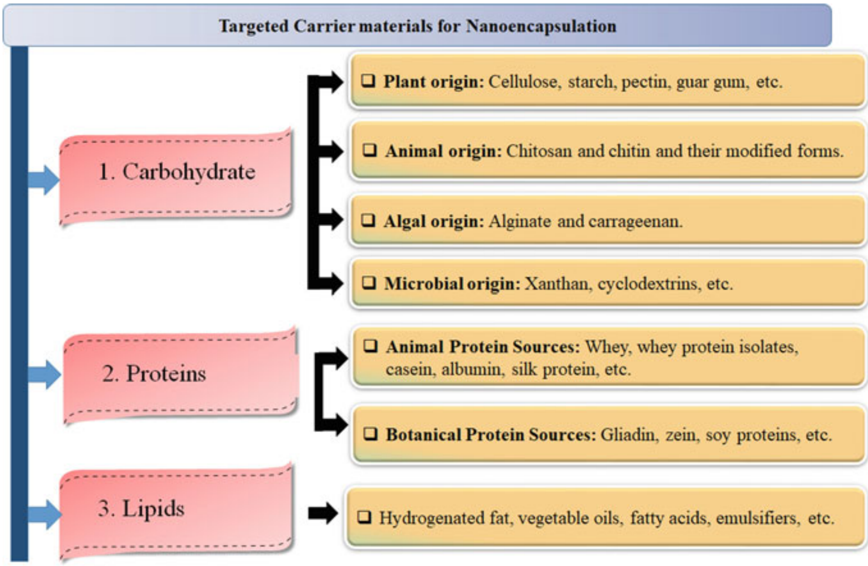

Fig. 10.4 Several targeted carrier or wall materials for nanoencapsulation techniques

hydrophobic compounds can be entrapped in inner cavity. The several types of CDs are α-CD, β-CD, and γ-CD, where the several types consist of different numbers of glucose units. The protein sources for encapsulation of bioactive components are animal protein sources (whey, whey protein isolates, casein, albumin, silk protein, etc.) and botanical protein sources (gliadin, zein, soy proteins, etc.). Moreover, some of the proteins used for the delivery of bioactives are gelatin, collagen, casein, albumin, silk fibroin, zein, soy protein, α-lactalbumin, β-lactoglobulin, bovine serum albumin, pea protein, and others [6]. Interestingly, the biopolymers gelatin, collagen, albumin, and α-lactalbumin are used for the development of nanodelivery systems. The several protein modification techniques for improved functionality of nanocarrier are physical treatments, chemical treatments, and enzymatic treatments

[7]. Additionally, the protein and polysaccharide complexes can also be utilized for the nanoencapsulation of available hydrophobic compounds. The protein components for encapsulation of polyphenols and others include casein, whey protein isolates, sodium caseinate, albumin, silk protein, zein, gliadin, soy protein, etc. The several lipid carrier systems are nanoemulsion, liposomes, solid–lipid nanoparticle, nanostructure lipid carriers, etc. [8]. The advantages of lipid carrier systems include nanodelivery system for water insoluble or poorly soluble ingredients, high encapsulation load and efficiency, flexible in controlled release profile, targetability, etc. The benefits of solid–lipid nanoparticles process in comparison with nanoemulsions and liposomes are high encapsulation efficiency, high flexibility in controlled release profile, slower degradation rate, etc. [8]. The emulsifiers for emulsification of bioactive compounds are tween-20, tween-40, tween-60, tween-8, polyethylene glycol, and other materials.

10.4 Significance of Nanoencapsulation of Several Food Components

The nanoencapsulation of several food components has several benefits such as (1) protect core materials (separate incompatible materials, prevent environmental oxidation); (2) modify properties of bioactive compounds (the carrier materials can mask odor and foul taste); (3) develop new food products/product enrichment (efficient, biocompatible nanoencapsulates in food products to develop new products); (4) triggered release, targeted release, and sustained release of encapsulated materials; (5) convert liquids to free flowing solids; (6) increased shelf life; (7) improved marketing and product aesthetics; and others. The development of nanoencapsulates of proteins has the significance of increased protein bioavailability, sustainable production of protein nanoparticles, and others. Additionally, the use of nanoemulsions has several benefits such as carrier of hydrophobic compounds, toxicologically safe, shelf stable, suitable to use in beverages, and others.

10.5 Research and Development in Nanoencapsulation of Food Components

As represented in Table 10.1, there are several reports available for the nanoencapsulation of several food ingredients such as antioxidants, fish oils, essential fatty acids, vitamins, antimicrobial agents, essential oils, natural food colorants, food flavors, and minerals. Additionally, the several applications of nanoencapsulation have been represented in Fig. 10.5. Some of the bioactive components are D-limonene, flax seed oil, sunflower oil, curcumin, β-carotene, salmon oil, capsaicin, bovine serum albumin, linoleic acid, docosahexaenoic acid, tannins, condensed tannins, hydrolysable tannins, etc. The essential oils are obtained from medicinal

Table 10.1 Application of nanoencapsulation technique for entrapping bioactive components

Sl. No.	Targeted components	Wall materials/ category of nanoencapsulation	Nanoencapsulation technique	Property	References
1.	Essential oils (Terpene mixture from *Melaleuca alternifolia* and D-limonene)	Oil-in-water nanoemulsion	High-pressure homogenizations at 300 MPa And high shear homogenization	Enhanced antimicrobial activity	[10]
2.	Probiotic bitter gourd juice powder	Maltodextrin Gum arabic Mixture of maltodextrin and gum arabic	Spray drying	*L. casei* viability is better in nanocapsules with maltodextrin as wall material	[12]
3.	Fish oil	Nanoliposomes Ingredients: lecithin, sunflower oil	Normal stirring process	Yogurt fortification	[13]
4.	Vitamin D (a lipophilic molecule)	Fish oil	Nanoemulsion by ultrasonication	Higher oral bioavailability Efficient nutrient delivery Encapsulation efficiency: 95.7–98.2%	[14]
5.	EPA/DHA	Sodium caseinate gum arabic complex	High speed homogenizer Ultrasonic homogenizer Freeze drying	Fruit juice enrichment	[15]
6.	Phenolic grape marc extract	Different nanoemulsion formulations using sunflower oil, peanut oil, stearic acid, soy lecithin, ethanol, water, maltodextrin, and others	Nanoemulsion-based nanoencapsulation	Improve lipid solubility Improve antioxidant efficiency Improve shelf life of hazelnut paste	[16]

(continued)

Table 10.1 (continued)

Sl. No.	Targeted components	Wall materials/ category of nanoencapsulation	Nanoencapsulation technique	Property	References
7.	Curcumin	Milk fat (oil medium) and sodium caseinate (Wall material)	Oil-in-water emulsion process (Solvent-free green chemistry approach)	Antioxidant activity enrichment Enhanced water solubility of curcumin	[11]
8.	Saffron extract	Double layer W/ O/W multiple emulsion of pectin and whey protein isolates	Spray drying method	Improved encapsulation efficiency Lowest surface content	[17]

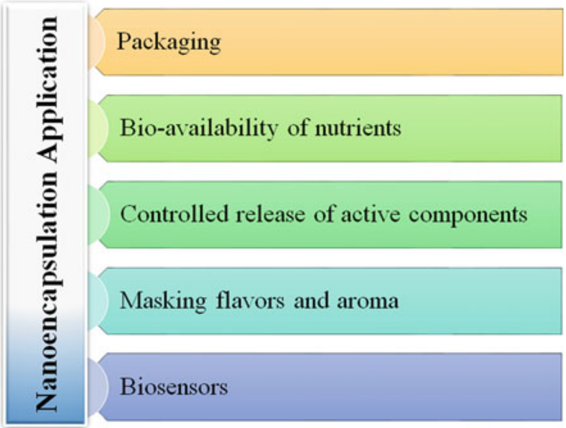

Fig. 10.5 Various applications of nanoencapsulation

and aromatic plants, spices which are a great reservoir of health beneficial components, which have several properties such as hydrophobicity, susceptibility, and high volatility. The essential oils can be nanoencapsulated to protect it from environmental degrading agents and have a potential application in food preservation and in some targeted industries such as producing the solvent-free perfumes [9]. Additionally, the preparation of oil-in-water-based nanoemulsion of essential oils such as terpenes mixtures and D-limonene using high-pressure homogenization and high shear homogenization techniques can provide antimicrobial activity with fruits juices such as pear and orange juice [10]. In this case, the used emulsifiers are soy lecithin, modified gum and a mixture of glycerol monooleate and tween 20, and palm oil or sunflower oil as organic phase. The various tested nanoemuslions are

(1) terpenes(50 g/kg), soy lecithin(10 g/kg), and water(940 g/kg) using high-pressure homogenization technique; (2) terpenes(50 g/kg), soy lecithin(10 g/kg), and water(940 g/kg) using high shear homogenization technique; (3) D-limonene (50 g/kg), clear gum (100 g/kg), and water(850 g/kg) using high-pressure homogenization technique; (4) D-limonene(50 g/kg), palm oil(50 g/kg), soy lecithin(20 g/kg), and water(880 g/kg) using high-pressure homogenization technique; (5) D-limonene(50 g/kg), sunflower oil(50 g/kg), tween(15 g/kg), glycerol monooleate(15 g/kg), and water(870 g/kg) using high-pressure homogenization technique; and (6) D-limonene(50 g/kg), tween 20(7.5 g/kg), glycerol monooleate (7.5 g/kg), and water(935 g/kg) using high-pressure homogenization technique. Interestingly, the application of nanoencapsulates of essential oil in food products including fruit juice enhances the antimicrobial property of food products with enhanced quality. Additionally, a research reports the formulation of nanoencapsulates of curcumin employing a solvent-free green chemistry approach, where milk fat and sodium caseinate as oil and wall materials, respectively [11]. In this method, an encapsulation efficiency of 91% is obtained with a particle size range of 40–250 nm (as analyzed by TEM). The curcumin is one of the most extensively used bioactive compounds for its several health beneficial properties such as antioxidant, antidiabetic, antimicrobial, antibacterial, anticancer, anti-inflammatory, wound healing, and antihepatoma.

The nanoencapsulates of pomegranate bioactive compounds have enhanced anticancer effects in breast cancer cell and can be used for breast cancer chemoprevention [18]. However, the various pomegranates in several forms as extracts, juices, polyphenols provide effective in vitro and in vivo anticancer activity. However, from very early days, pomegranates are used for several medicinal applications such as diabetes, cancer, inflammatory disorder, and cardiovascular disorders. Further, the incorporation of nanoencapsulates of grape marc extract in hazelnut paste helps in improving the shelf life by inhibiting lipid oxidation [16]. Another research reports the nanoencapsulation of probiotic bitter gourd juice powder using maltodextrin, gum arabic, and starch using spray drying techniques, where the *Lactobacillus casei* viability is more in maltodextrin encapsulated powders [12]. In this regard, the encapsulation of antimicrobials is beneficial to increase the availability of bioactive compounds which in turn can be used in food items where microbial growth can be reduced or inhibited. Further, nanoencapsulation can provide protection against environmental agents such as moisture, light, and pH conditions. The development of nanoencapsulated fish oil provides a decreased peroxide value, syneresis, and acidity [13]. The nanoencapsultes of fish oil can be incorporated in yogurt to produce fortified yogurt, where the yogurt with nanoencapsulates of fish oil has a higher *cis*-5,8,11,15,17 -eicosapentaenoic acid (EPA) and *cis*-4,7,10,13,16,19-docosahexaenoic acid (DHA) than yogurt with free fish oils. The encapsulation of fish oil acts as a flavor masking agents and increased the consumer acceptance in the society. Moreover, the use of fish oil for the encapsulation of vitamin D using ultrasonication technique provides efficient nutrient delivery and higher oral bioavailability with an encapsulation efficiency of 95.7–98.2% [14]. Vitamin D is sensitive toward some environmental conditions such as heat, gaseous conditions, and light, thus the nanoencapsulation formation of vitamin D using fish oil can protect it from gastric conditions. The several encapsulation techniques used for drug delivery systems are polymer conjugate, O/W emulsion, liposomes, sol-gel process, organogel-based emulsion, alginate beads synthesis, and others. A report suggests the development of nanoencapsulates of saffron extract via double-layered W/O/W multiple emulsion of pectin and whey protein isolates using spray drying method can provide increased stability of saffron in various environmental conditions. Additionally, the developed nanoencapsulates impart increase encapsulation efficiency and lower surface content [17]. The application of nanoencapsulates of eicosapentanoic acid (EPA) and docosahexanoic acid (DHA) (developed using carrier materials sodium caseinate and gum arabic complex) in fruit juices and other beverages offers enriched food products [15]. Additionally, nanoencapsulation of essential minerals can provide improved release control and can be useful in developing mineral fortified food products with enhanced shelf life and decreased nutritional quality [5].

10.6 Available Techniques for Fabrication of Nanoencapsulates

The several available approaches for nanoencapsulation formulation have been represented in Fig. 10.6. Additionally, the other methods used for increased bioavailability of bioactive compounds are O/W emulsion, sol-gel process, liposomes, organogel-based emulsion, alginate beads synthesis, etc. The approaches in nanoencapsulation techniques include top-down and bottom-up approaches, where the top-down approaches include emulsification, and emulsification solvent evaporation and bottom-up approaches include coacervation, nanoprecipitation, inclusion complexation, and supercritical fluid technique. The top-down approach for nanoencapsulation includes the size reduction and structure shaping of developed nanoparticles, whereas the bottom-up approach includes the development of nanoencapsulates via self-assembly and self-organization of molecules and is effected by several factors temperature, concentration, ionic strength, etc. The available approaches for the preparation of lipid carrier systems include several techniques such as (1) mechanical methods: homogenization (hot homogenization, cold homogenization, high-pressure homogenization, etc.), extrusion, colloid mill, microfluidization, ultrasonication; (2) non-mechanical method: solvent diffusion, reversed phase evaporation, heat treatment, ultrasonic solvent evaporation, etc. Interestingly, electrospinning is considered as a remarkable approach employed for developing nanoencapsulates of bioactive compounds, where the process has several benefits including cost-effective method, easy incorporation of bioactives, flexibility in size requirement for bioactives, better for heat sensitive components, etc. [19]. The physical techniques for nanoencapsulation of bioactives include spray drying, freeze drying, fluidized bed coating, centrifugal extrusion, supercritical fluid method, and others. The fabrication of microencapsulation and nanoencapsulation

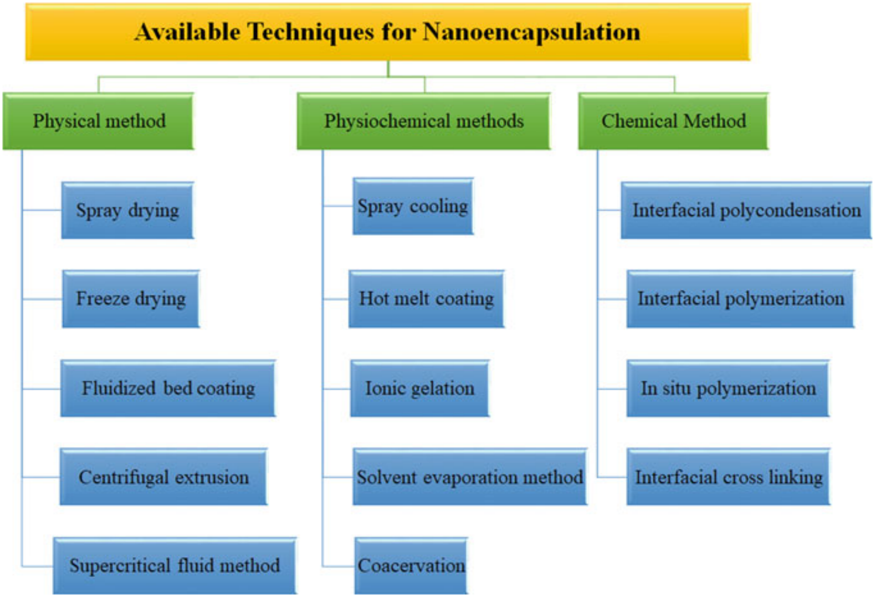

Fig. 10.6 Several techniques for developing nanoencapsulates of bioactive components

using supercritical fluid techniques provides enormous benefits in pharmaceutical applications [20]. In this technique, carbon dioxide is used widely, where CO_2 at supercritical conditions provides several attributes such as gas like viscosity, liquid like density, low viscosity, high diffusivity, and enhanced solubility. The physiochemical methods of nanoencapsulation of bioactive are spray cooling, hot-melt coating, ionic gelation, solvent evaporation method, coacervation, and others. The targeted chemical methods for developing nanoencapsulates of bioactive components are interfacial polycondensation, interfacial polymerization, in situ polymerization, interfacial cross-linking, and others. Thus, various processes are employed for developing nanoencapsualtion of food components.

10.7 Characterization Techniques for Nanoencapsulates

The characterization of nanoencapsulated powders is commonly evaluated for the appearance, morphology, size distributions, surface charge, surface composition, physicochemical property, thermal property, stability, and others. In this regard, a list of the use of various characterization techniques that are analyzed for nanoencapsulated materials has been made in this section. As shown in Fig. 10.7, the several characterization techniques used in the analysis of nanoencapsulated particles are represented [21, 22]. The morphology of nanocapsules is determined

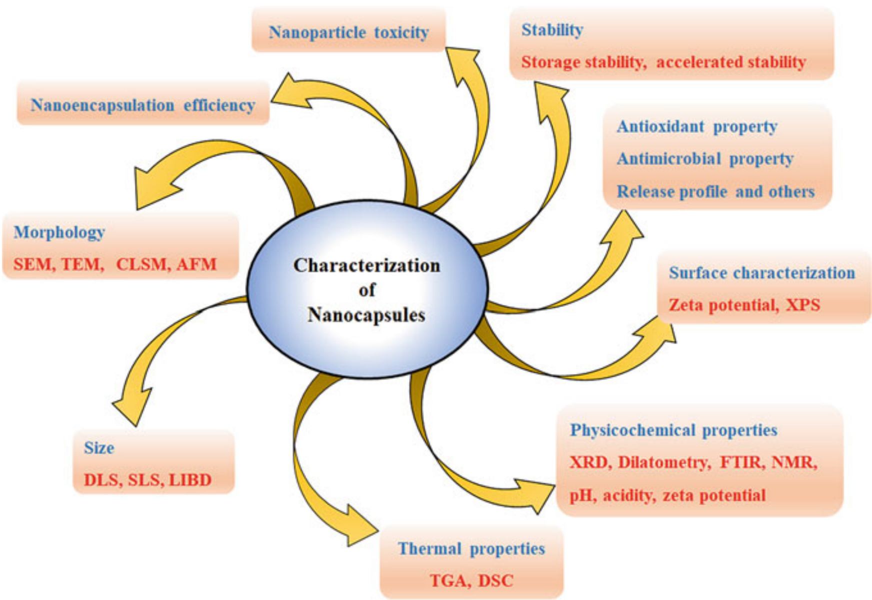

Fig. 10.7 Characterization techniques of nanoencapsulated powders (SEM: scanning electron microscopy; TEM: transmission electron microscopy, CLSM: confocal laser scanning microscopy; AFM: atomic force microscopy; DLS: dynamic light scattering, SLS: static light scattering; LIBD: laser-induced breakdown detection; XPS: X-ray photoelectron spectroscopy; XRD: X-ray diffraction; FTIR: Fourier transform infrared spectroscopy; TGA: thermal gravimetric analysis; DSC: differential scanning calorimetry)

using scanning electron microscopy (SEM), transmission electron microscopy (TEM), confocal laser scanning microscopy (CLSM), atomic force microscopy (AFM), and others. The size distribution of nanocapsules is measured using dynamic light scattering (DLS), static light scattering (SLS), laser-induced breakdown detection (LIBD), and others. The surface characterization can be analyzed using zeta potential (surface charge), and the surface composition of nanocapsules can be characterized using X-ray photoelectron spectroscopy (XPS). The physicochemical properties of nanocapsules are measured using X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), etc. Further, the various properties of active components can be measured by determining antioxidant, antimicrobial, medicinal properties, and other properties. The thermal properties of nanocapsules can be determined using thermal gravimetric analysis (TGA) and differential scanning calorimetry (DSC). The encapsulation efficiency, toxicity, and stability studies in terms of storage stability and accelerated stability are determined for maintaining food quality and safety. The other properties such as color, rheology, and active components specific properties are also measured. However, the details related to various equipment and analysis will be done in the later chapters.

10.8 Conclusion

The nanoencapsulation has attained a great interest in protecting health beneficial compounds from unfavorable environmental conditions during material processing, storage life, and transportation of the food components. Further, the nanoencapsulation is extensively considered as a valuable technique to increase the functionality and bioavailability of bioactive compounds. Thus, the chapter provides a brief overview of various aspects of nanoencapsulation, a multiphase-based edible food packaging for improved bioactivity of active compounds.

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Chapter 11
Nanodelivery System of Bioactive Compounds in Edible Food Packaging

Chethana Mudenur, Tabli Ghosh, and Vimal Katiyar

11.1 Bioactive Compounds

Food is an essential part of our life that provides the nutrients required for the proper functioning and maintenance of the body. Some of the ancient methods of food preservation are conventional drying, fermentation, salting, and canning to remove the majority of the water content from the food to protect from spoilage. Recently, researchers are focusing more on the biopolymer packaging materials and the edible packaging materials in food packaging due to their eco-friendly nature, edibility, and non-toxic behavior. There is a wide range of bioactive compounds used in the food packaging, where aroma compounds are one of such efficient bioactive agents delivering wide applications in antioxidant, antimicrobial, and insect repellent packaging. These compounds are highly preferred in extending the shelf life of packaged food. However, the bioactive compounds may have bitter flavor, foul smelling (such as bioactive compounds from fish has fishy odor) and are volatile in nature at ambient temperature, which leads to the restricted application in polymer packaging. The polymer-based packaging undergoes processing at high temperature, and the temperature sensitivity of bioactive compounds leads to the loss or transformation of bioactive compounds during the processing. However, the bioactive compounds need to satisfy some requirements for effective use in the packaging, which includes (i) ability to retain in the polymer matrix, and (ii) their release in the polymer matrix in response to the triggering effects. In contrast, proteins and polysaccharides are aroma carriers, which favor the release of specific active compounds, by their structural modification in response to the temperature and relative humidity. The active compound can be used in different ways; direct coating on the surface of the product, self-supporting films, or as coated papers [1].

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In the context of the growing demand for preservative-free food with minimal processing, the use of synthetic colorants and the aroma compounds are of least importance. Thus the natural products are gaining importance in the food sector, due to its non-toxic nature. In the current situation, conventional petroleum-based polymer is hard to replace due to its versatility, lightweight, inexpensiveness, and excellent mechanical properties. The increasing plastic waste is a major concern though it has applications in many fields. Thus, there is a need for the edible food packaging in the current scenario to replace the existing packaging materials. Meanwhile, the edible nature of the wrapper along with food products increases the concern about hygiene, safety, associated health benefits, and consumer acceptance. Thus, to tackle such problems, the advancements in edible food packaging are necessary [2]. The main idea behind bioactive compounds aided edible packaging is the consumption of packaging material along with food having additional health benefits and improved packaging quality. These kinds of packaging materials are likely to gain momentum in the coming years. In this chapter, the emphasis is made on the demand for the bioactive compounds, their types, methods of extraction, various medicinal properties, and use of nanosystem in delivering the bioactive compounds to the edible packaging materials.

11.1.1 State of the Global Market and Research Insights on Bioactive Compounds

The market development in the field of edible packaging is showing improved growth in recent years. Such growth is due to the increase in waste disposal and poor waste management of non-biodegradable plastics. In the case of edible packaging, there is no waste generation after the consumption of the product by the end users. Further, packaging industries are focused on the development of advanced methods to initiate the standards of packaging. According to the Transparency Market Research (TMR), the total revenue of the edible packing is expected to increase worldwide in the future. Since past decades, several changes are taking place to replace the synthetic packaging for increased concern on packaging waste, global warming, and environmental pollution. Several public entities have joined their hands to ensure the eco-friendly pollution-free world. This evolving factor is responsible for several new trends in the packaging sector. The retail outlets are focusing more on the edible packing to generate market demand. The global packaging market has witnessed some of the small-scale edible packaging vendors, though such marginal vendors cannot challenge large-scale producers [3]. As already mentioned in Chap. 1, the global vendors of the edible packaging market are JRF Technology LLC [4], Tate Lyle Plc [5], MonoSol, and WikiCell Designs Inc [6]. Further, the market value of global edible packaging valued USD 0.77 Billion and expected to increase to USD 1.3 Billion by 2024 [3]. Based on the geography, material used, and the end user applications, the global edible packaging sector can be divided into different types as represented in Fig. 11.1 according to the global industry analysis and forecast 2016–2024.

The market demand for edible packaging and the active compound leads to the increased interest in the scientific community. In recent years, the research is growing to improve the performance and effectiveness of the food packaging material using the active compounds. Major fractions of the publications are focused on the quality improvement and biodegradability of the packaging material. According to the SciFinder® data, the number of articles published on "active compounds and food packaging" has increased four times during the year 2000–2020, which is represented in Fig. 11.2.

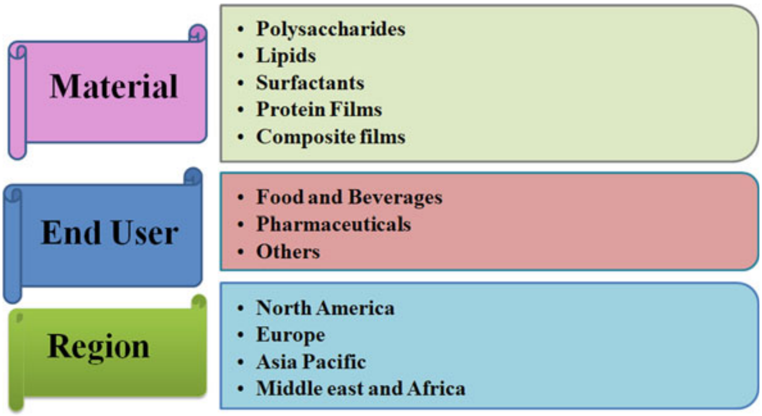
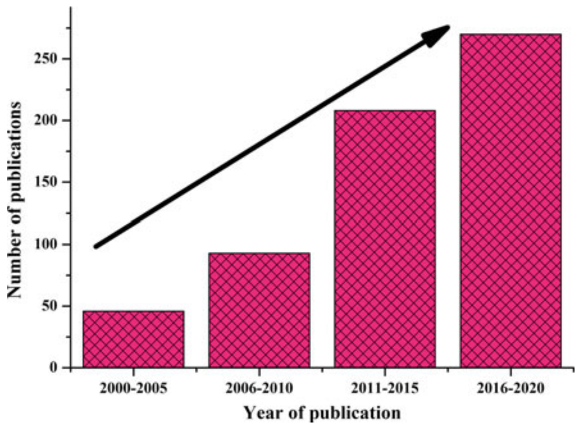


Fig. 11.1 Segmentation of global edible packaging market

Fig. 11.2 Number of articles published in the field of food packaging. The data is based on the search results obtained using key words "active compounds" and "food packaging" search on SciFinder®



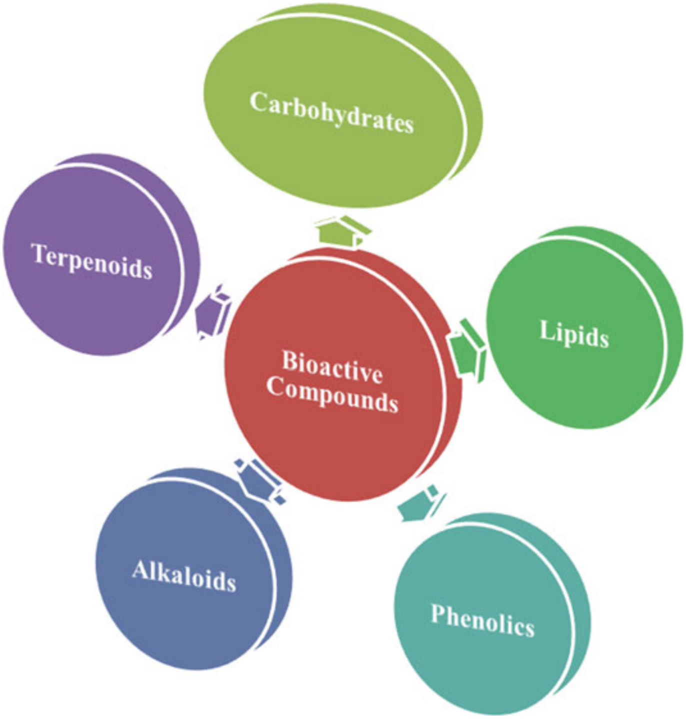


Fig. 11.3 Major classification of bioactive compounds based on the chemical properties

11.2 Types of Bioactive Compounds

There are various types of bioactive compounds in nature. These are classified as carbohydrates, lipids, phenolics, alkaloids, terpenoids, and nitrogen-containing metabolites based on their chemical properties [7] (Fig. 11.3). The major classification of the bioactive compounds and associated method of extraction for some of the bioactive compounds are listed in Table 11.1.

Table 11.1 Classification of bioactive compounds and methods of extraction

Sl no	Active compounds	Types	Method of extraction	Examples	References
1	Carbohydrates	Monosaccharides	Aqueous extraction	Glucose, Fructose, Galactose	[8]
		Disaccharides		Sucrose, Lactose, Maltose	
		Polysaccharides	Enzymatic, Microwave, Ultrasonic-assisted extraction	Starch, Glycogen, Cellulose	[9]
2	Lipids	Triglycerides	Supercritical carbon dioxide and ethanol	Tristearic acid	[10–12]
		Phospholipids		Phosphatidylcholine, Phosphatidylserine	
		Sterols	Supercritical fluid extraction	Campesterol, Sitosterol, Stigmasterol, Cholesterol	[13]
3	Phenolics	Phenolic acid	Liquid–Liquid, Ultrasound, Microwave, Supercritical fluid extraction	Cinnamic acid, Benzoic acid	[14, 15]
		Tannins	Aqueous acetone extraction	Gallic acid esters, Proanthocyanidins	[16]
		Lignans	Pressurized low polarity water extraction	Enterolignans, Enterodiol, Enterolactone	[17]
		Flavonoids	Solvent-based ultrasound-assisted extraction, High hydrostatic pressure-assisted extraction, Polarity dependent extraction	Myricetin, Morin, Rutin	[18–20]
		Stilbenes	Fluidized bed extraction, Soxhlet extraction, Accelerated solvent extraction	Trans-resveratrol, Trans-piceid, Cis-piceid	[21]
		Coumarins	Extraction with polar solvents	Warfarin, Phenprocoumon, Acenocoumarol	[22]
		Curcuminoids	Batch extraction, Three phase partitioning, Crossflow ultrasound-assisted extraction	Curcumin, Demethoxycurcumin	[23, 24]

(continued)

Table 11.1 (continued)

Sl no	Active compounds	Types	Method of extraction	Examples	References
4	Alkaloids	True alkaloids	Surfactant-assisted extraction, Solvent extraction	Atropine, Nicotine, Morphine	[25]
		Protoalkaloids		Mescaline, Adrenaline, Ephedrine	[26]
		Polyamine alkaloids		Putrescine, Spermidine, Spermine	[27]
		Peptide and cyclopeptide alkaloids		Discarine C, Discarine D, Myrianthine A	[28]
		Pseudoalkaloids		Caffeine, Cathinone	[29]
5	Terpenoids	Monoterpenes	Methylene chloride extraction, Rectification, Freezing	Pinene, Citrol, Nerol, Menthol, Camphor, Limonene	[30, 31]
		Sesquiterpenes		Nerolidol, Farnesol	
		Diterpenes		Phytol, Vitamin A	
		Triterpenes		Squalene	
		Tetraterpenes		Carotene	
		Polyterpenes		Gutta-percha, Natural rubber	

11.2.1 Carbohydrates

These are polyhydroxy aldehydes and ketones. There are three classes of carbohydrates, monosaccharides (glucose and fructose), disaccharides (lactose and sucrose), and polysaccharides (cellulose and starch) [32]. Monosaccharides and disaccharides are simple forms, provide a rapid source of energy, while polysaccharides are complex forms that take a longer time for digestion. Foods having complex carbohydrates are more nutritious than simple forms [33]. Carbohydrates are widely studied elements in the development of edible food coatings and films. Since the last decade, starch, chitosan, derivatives of cellulose, and pectin are being evaluated for their applications in the packaging sector [34]. Biopolymers such as xanthan gum, alginate, and carrageenan are used in the edible coatings to reduce the conventional packaging. These are widely available and non-toxic in nature. These have selective permeability to oxygen and carbon dioxide for an extend shelf life of fruits [35].

11.2.2 Lipids

The roles of lipids in the edible packaging are gaining importance due to its hydrophobic nature and moisture barrier properties. Lipids are gaining importance in the edible packaging, and their efficiency completely depends on the structure, chemical arrangement, lipid interaction with food, and hydrophobic nature. The lipids can be classified into two groups based on polarity, such as polar and non-polar lipids. The non-polar lipids such as paraffin oil, waxes are not used in the edible packaging due to their insoluble nature, but the polar lipids such as diglycerides, triglycerides, cholesterol, vitamin A, D, E, K, phospholipids, monoglycerides are used in the edible packaging sector [11].

11.2.3 Phenolics

Phenolics are one of the most abundant antioxidants of the human diet. They can be characterized by hydroxyl groups and aromatic rings with considerable structural diversity. Based on chemical property, phenolics are classified into simple phenols and polyphenols. Simple phenols are further subdivided into phenolic acids and coumarins. Polyphenols are subcategorized into flavonoids and tannins [36]. The main mechanisms of action of phenolics are on the scavenging reactive species of nitrogen, oxygen, and chlorine [37].

11.2.4 Alkaloids

These are organic compounds present in nature, consisting of basic nitrogen atoms and are produced in a variety of plants, bacteria, fungi, and animals [38]. Alkaloids are divided into five groups such as true alkaloids, protoalkaloids, polyamine alkaloids, pseudoalkaloids, peptide, and cyclopeptide alkaloids. They possess structural diversity, due to which there is no single method of extraction from their respective raw materials. They are insoluble in water, but most of them are soluble in organic solvents [39].

11.2.5 Terpenoids

The terpenoids are natural organic chemicals also known as isoprenoids. These are derived from isoprene and are produced by various plants and animals. These are phytoalexins of defense mechanisms in plants [40]. There are different types of terpenes, such as hemiterpenes, monoterpenes, sesquiterpenes, diterpenes, sesterterpenes, triterpenes, and polyterpenes. Terpenes are mostly applicable as food flavoring agents for food also to enhance the fragrance in the perfume industry [41].

11.3 Bioactive Compounds in Edible Packaging: Current Trends and Challenges

The social interest and demand for natural, renewable, and biodegradable materials have been increasing in recent years. In addition to this, consumers and producers are keen on choosing the quality food products with improved shelf life and retained nutritional properties [42]. Active packing technologies are of greater importance in the current scenario, which satisfies the long-term storage of the processed food products in addition to antioxidant, antimicrobial, anticancer, and other medicinal properties in the packaging materials. The rising demand encourages the use of plant-based bioactive compounds in edible packaging. Herbs and spices are also the potential agents to be used in the edible packaging with antioxidant and antimicrobial properties. There are huge numbers of start-ups working on the development of a variety of edible packaging solutions. Various innovations and advancements are taking place in the field of the edible packaging industry, where the inclusion of nanotechnology can increase the nutritional value of food. The encapsulated active composite materials (nanoencapsulation) with edible films help to preserve the food from various environmental factors such as moisture, heat, dust particles, and other contaminants entering the packaged food. Some of such advanced products developed by many start-ups will be explained in later sections.

11.3.1 Disappearing Package

The Skipping Rocks Lab Ltd. is a London-based start-up company that is keen on the development of the disappearing active packaging to tackle plastic pollution. This packaging is an ideal candidate for food products. It is made up of flexible and sustainable plant extracts such as corn, seaweed, sugar, and agave. Ooho is such a flexible packaging suitable for packaging of beverages and sauces, which degrades within 4–6 weeks. This packaging can also be eaten to make it ideal for on-the-go consumption [43].

11.3.2 Seaweed-Based Packaging

Evo & Co. is an Indonesian social enterprise that provides the plastic-free alternatives to promote sustainable packaging. It produces the biodegradable single-use plastic films using seaweed as an eco-friendly raw material. These are biodegradable, compostable, and even safe to consume. The product Ello Jello is an edible cup having variations in flavor; is safe for consumption and is biodegradable in nature. Another product is seaweed-based packaging which is printable and heat stable, dissolves in warm water, with a shelf life of 2 years. This also consists of high fiber, minerals, and vitamins. The key applications include the packaging of sugar, cereals, salt, seasonings in instant noodles, burgers, and sandwich [44].

11.3.3 Potato Waste Based Packaging

Do Eat is a Belgium-based company, exclusively develops containers made of potato residues and draft beer. In addition to its biodegradability and edible nature, it also enhances the food preservation in comparison with traditional plastic packaging. It produces various types of edible bowls, trays, and plates to serve salads, ready meals, cakes, etc. [45].

11.3.4 Casein-Based Packaging

Lactips is a French start-up, produces, and markets plastic-free polymer packaging materials made of casein (milk protein). In combination with the alkali, casein film provides the oxygen barrier properties better than conventional plastic wrappings. It has good resistance to humidity, moisture, and temperature [46].

11.3.5 Sugarcane-Based Packaging

Candy Cutlery is a Canadian company aimed to develop edible utensils made of 100% natural sugar to reduce the use of plastics. It is a producer of dessert spoons and glasses consisting of natural cane sugar with coffee, vanilla, and peppermint flavors. These products are free of artificial sweeteners and can be used as shot glasses or as an ice-cream dishes [47].

11.3.6 Challenges in Use of Bioactive Compounds in Edible Food Packaging

Cost. The manufacturing cost of the edible packaging is quite high in comparison with conventional packaging, due to high sanitary requirements during manufacturing and transportation. Moreover, the edible films are prone to catch the dust particles, which lead to food contamination. Therefore, to protect such edible packaging films, the manufacturer has to spend a lot of money to make it safe and contaminant-free [48].

Regulatory Requirements. The active and edible packaging films must meet the regulatory requirements. Such materials should be approved as generally recognized as safe (GRAS) by the U.S. Food and Drug Administration. Also, the edible packaging material should consist of water-soluble polymers and must be manufactured using machines without any human interventions. Such products are considered to be suitable for edible packaging. The additives used must be of general purpose ingredients. If the added additives are coloring agents, then they must be strictly regulated, studied, and monitored to determine their safety [49].

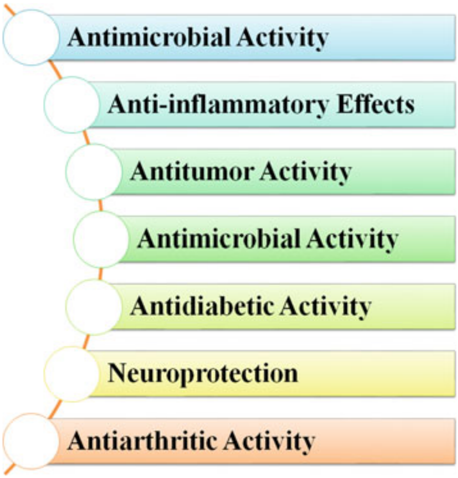


Fig. 11.4 Medicinal properties of the bioactive compounds

11.4 Medicinal Properties of Bioactive Compounds

As mentioned in Fig. 11.4, the bioactive compounds have various medicinal properties based on their effect and mode of action and are explained in detail in this section.

11.4.1 Antioxidant Activity

The free radicals in the biological systems are derived from sulfur, oxygen, and nitrogen. Free radicals have an unpaired electron, due to which they are highly reactive with other molecules. These are produced during cellular metabolism and play a key role in ion transport. The human body has the natural antioxidant defense mechanism that includes the metal chelators, enzymes, and free radical scavenging activities. This neutralizes the reactive oxygen species that are generated during the process. Besides, the intake of the dietary antioxidants helps to maintain the required quantity of antioxidant status in the body [50]. Phenolic compounds (phenols and polyphenols), flavonoids, thiols, steroids, and carotenoids have an exceptional antioxidant activity, which can reduce the production of free radicals, by which it lowers the oxidative stress and the risk of chronic diseases. These are present as microconstituents and are responsible for a wide array of biological activities. In this regard, some of the bioactive compounds which play a key role as cellular antioxidants are vitamin E, and vitamin C. Additionally, ginsenosides obtained from ginseng have antioxidant activities against free radical to protect vascular endothelium [51]. Catechin and epicatechin are the flavonoids present in the seeds of grape and green tea also reported to exhibit antioxidant activities [52].

11.4.2 Anti-inflammatory Effects

The biological response of the immune system to an injury, infection, or irritation is referred to as inflammation. The anti-inflammatory effect is regulated by inflammatory cytokines, which include interferon gamma-γ, interleukins, and nitric oxides. The several food products such as fruits, vegetables, and legumes contain more quantity of phytochemicals that exhibits anti-inflammatory properties [53]. Cyclooxygenase-2 (COX-2) and inducible nitric oxide synthase (iNOS) are the important enzymes that are the mediators of the inflammatory process. Ingested curcumin in the body is induced by Tumor Necrosis Factor-α (TNF-α) and inhibits the COX-2 expression in human colon epithelial cells [54]. However, complete understandings of the mechanism of such anti-inflammatory compounds are not clear. These also have some drawbacks such as high dosage and low bioavailability [55]. Some of the food consists of anti-inflammatory properties are food legumes, peptides, and lectins.

11.4.3 Antitumor Activity

The tumor is a non-specific term used for neoplasm, which is defined as the rapid and abnormal growth of new cells. These can be benign (harmless) or malignant (cancerous) cells. Benign tumors have localized growth with no spreading to other parts of the body, whereas the malignant tumors are not localized, and they are often resistant to treatments, and there is a possibility to recur once they are removed [56]. The target in the carcinogenesis is the abnormal cell proliferation, and the characteristics of the cell embodies allow the survival of the cells beyond the normal limits. The most general therapy includes cytotoxic drugs and radiations. However, such drugs could affect the normal cells in the body, including blood forming cells and hair follicles [57]. However, physicians try to treat with the levels with the minimum side effects. Some of the natural active compounds that play important roles in such treatments include didemnin and depsipeptide from tunicates, which are potential inhibitors of the leukemia cells (L1210) and are also active against melanoma cells (B16) [58]. The polysaccharides derived from the fungal cell walls are playing significant effects recently due to their immunomodulatory effect, resulting in antitumor effects [59]. Some wild mushrooms produce low molecular weight catechol, steroids, and high molecular weight glycans, glycoproteins with exceptional antitumor activities [60]. Some plant-based bioactive compounds such as myricetin, geraniin, tocotrienols, and curcumin exert anticancer activities [61].

11.4.4 Antimicrobial Activity

Antimicrobial property is a process of inhibiting or killing of microbes responsible for certain diseases. These could be antibacterial, antifungal, and antiviral, with a unique mode of actions. The phenolic extracts of *Lactarius deliciosus* (wild edible mushroom) are effective against *Bacillus cereus*, *Bacillus subtilis*, *Candida albicans,* and *Cryptococcus neoformans*. The plant extract from *Arum maculatum* is effective against *Staphylococcus aureus*, *Listeria monocytogenes*, *Escherichia coli*, and *Pseudomonas aeruginosa* [62]. The methanolic leaf extracts of *Jatropha tanjorensis* consisting of friedelin, stigmasterol, β-amyrin exhibit antimicrobial activity against *Aspergillus fumigatus* and *Trichophyton rubrum*.

11.4.5 Antidiabetic Activity

Metabolic disorder characterized by a high level of blood glucose is referred to as diabetes mellitus. The most common in adults is type 2 diabetes, which can be seen when the pancreas is incapable to make enough insulin [63]. Insulin therapy is popular in recent years. However, obesity management is an important factor, and further, hypercholesterolemia is also associated with it. The several treatments for human health should also address hypertension, cardiovascular risk factors, and dyslipidemia to minimize cardiovascular diseases. The antidiabetic activities of bioactive compounds act through a mechanism of α-glucosidase inhibition thereby increased secretion of insulin. In this regard, Apigenin, coixol, glutinol, and scoparic acid A, D are antidiabetic bioactive compounds [64]. The crude plant extract such as *Stevia rebaudiana* shows antidiabetic effect in induced diabetic rats [65]. The treatment with glibenclamide could lower the glucose levels by 51%, and phytochemical analysis of the glibenclamide confirms the contribution of phenolic compounds, flavonoids, steroids, and saponins in reduced blood glucose level [66].

11.4.6 Neuroprotection

Neurodegeneration is a process of losing the structure or function of nerve cells [67]. It represents a group of neurological disorders with pathological expressions affecting the neuron subsets. Alzheimer's disease, Huntington disease, and Parkinson's disease are few among hundreds of neurodegenerative diseases. In the last few decades, bioactive compounds have been rigorously explored for their therapeutic potential for neurodegenerative diseases. Honey is a product of beehive, enriched with antioxidants and polyphenols, and helps in neuroprotection by reducing oxidative stress and improving learning, memory, cognitive function [68]. The rosemary extract, *Uncaria rhynchophylla* and *Withania somnifera* (ashwagandha) consisting of flavonoids, tannins, alkaloids, tannins have been reported as neuroprotective agents.

11.4.7 Antiarthritic Activity

Synovial inflammation and irreversible joint destruction are referred to as rheumatoid arthritis. The old age people are most affected by this disease, especially women. The flavonoids extracted from *Centipeda minima* reported to show antiarthritic activity [69]. Methanolic extract of the plant *Vitellaria paradoxa* and glocosides, phytosterols, phenolic acids, and xanthazones of *Xanthium strumarium* exhibits a significant effect on Freund's adjuvant-induced rheumatoid arthritis [70].

11.5 Nanocarrier System for Delivery of Bioactive Compounds to Food

As discussed in the earlier sections, the several bioactive food components for developing bioactive rich food systems are anthocyanin, carotenoids, polyphenols, probiotics, terpenoids, etc. The bioactive compounds are sensitive toward environmental factors such as light, gaseous composition, heat, and others, where the use of nanocarrier system for bioactive compounds can increase the bioavailability and stability of bioactive compounds against environment degrading factors. In this regard, as represented in Fig. 11.5, the targeted nanodelivery systems for bioactive compounds are polymeric nanostructured materials, nanoencapsulation, nanoemulsion, solid–lipid nanoparticles (NPs), nanostructured lipid carrier, nanoliposome, polymer dendrimers, and others. Interestingly, the nanovehicles systems for delivery of bioactive compounds to various food systems include solid and liquid systems, which provide controlled stability, solubility, bioavailability, and controlled release of bioactive components [71]. The biopolymer complex such as lipid–protein and lipid–polysaccharides has attained a great deal of interest to act as a nanoscale delivery system for polyphenols and bioactive compounds such as gingerols (from ginger), curcumin (from turmeric), lycopene (from tomato), anthocyanin (from strawberry, blueberry), essential oils (EOs), allicin (from garlic), capsaicin, cinnamaldehyde (from cinnamon), and others for developing bioactive rich food components. Besides, there are several approaches to produce edible nanoscale delivery systems including (1) Preparation of protein-based edible nanoscale delivery system: Natural nanostructures, liquid–liquid dispersion, self-assembly, cold gelation and desolvation; (2) Preparation of polysaccharide-based edible nanoscale delivery system: Natural nanostructures, polyelectrolytes (using ionic gelation), non-polyelectrolyte; and (3) Preparation of lipid-based edible nanoscale delivery system: Homogenization, sonication, solvent diffusion [72]. The delivery of quercetin nanocrystal can also be done using maltodextrin-based fast dissolving film [73]. Additionally, the delivery of bioactive components can also be obtained using biopolymer complex NPs (protein–protein and protein–polysaccharides) and hybrid NPs of lipid-biopolymer (lipid–protein and lipid–protein–polysaccharide).

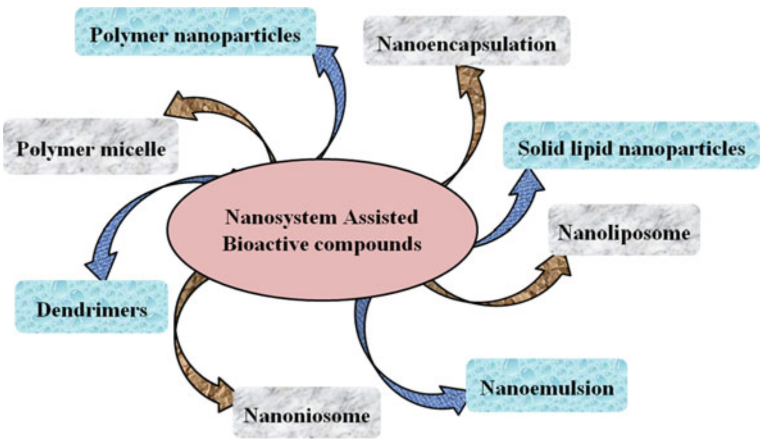


Fig. 11.5 Nanodelivery system of bioactive compounds in edible food packaging

11.5.1 Delivery of Bioactive Compounds Using Nanoparticles

The delivery of several bioactive compounds such as quercetin, tannins, curcumin, lignin, lycopene, naringenin can be done using green nanotechnology such as green nanomaterials [74]. The nanostructured materials are widely used for delivery of antioxidant, anticancer, antibacterial, antifungal, and other materials. In food systems, polymeric NPs including starch NPs, amylopectin NPs, chitosan (CS) NPs, cellulose NPs, and others used as a delivery system for improved bioavailability, improved antioxidant property, and others. The curcumin can be loaded with starch NPs (nanovehicles) and further used to improve the attributes of aloe vera and banana starch-based films in terms of barrier, mechanical, and thermal property [75]. Additionally, curcumin has different properties such as antimicrobial, antioxidant, anti-inflammatory, antibacterial, antiviral, hypocholestremic, anti-rheumatic, antihepatotoxic, and other properties. Amylopectin NPs having diameters of 20–50 nm are used as a delivery system for anthocyanin (Source: *Aronia melanocarpa*) for increased bioavailability and stability. Additionally, the diameter of anthocyanin and amylopectin NPs complex is 100 nm, and amylopectin protects anthocyanin with better antioxidant and antidegradation ability [76]. Interestingly, water-soluble CSNPs (Method of preparation: Ionic gelation using pentasodium tripolyphosphate) are used as a vehicle for loading and delivery of protein such as bovine serum albumin [77]. Quercetin is another type of flavonoid obtained from food plants having various biological activities, which is widely used to develop functional food products, where polymeric NPs are used as a delivery agent. Quercetin loaded CSNPs are developed using ionic gelation methods, which may be good in enhanced bioavailability of quercetin [78]. The quercetin loaded lecithin and CSNPs developed using electrostatic self-assembly technique are used in developing functional food products [79]. The encapsulation of quercetin in lecithin and CSNPs has an encapsulation efficiency of $96.13 \pm 044\%$ and improved antioxidant properties in comparison with free quercetin. Further, other polymeric nanomaterials such as nanocellulose, nanostarch, nanochitosan, nanoproteins, and nanolipids are extensively used in edible films and coatings as a delivery agent for bioactive compounds with enhanced shelf life of food products.

11.5.2 Lipid Nanocarrier System

As discussed in Chap. 7, lipid-based nanostructured materials in terms of lipid nanocarriers and solid–lipid nanostructured carriers are used for the delivery of bioactive compounds. The extensively used lipid-based nanodelivery systems used in edible food packaging are nanoemulsion, nanostructured lipid carrier, solid–lipid NPs, nanoemulsion, nanoliposomes, and others. The nanolipid carriers are solid–lipid NPs, nanostructured lipid carriers, and smart lipid nanocarriers. Additionally, nanostructured phospholipid carriers are nanoliposomes (monolayer or multilayer), nanophytosomes, structural nanoliposomes, and others.

Nanoemulsion. The nanoemulsion system is one of the most used approaches in food system for controlled delivery of bioactive compounds [80]. The nanoemulsion is a type of nanosized emulsion and colloidal dispersion system, which is extensively utilized for delivery of bioactive compounds and is prepared by emulsification approach using water, oil, and surfactants. The enhanced quality of nanoemulsions is obtained by using different kinds of EOs such as cinnamon EOs, garlic EOs, and sunflower EOs. The curcumin-based nanoemulsions systems are developed using several systems such as curcumin-cinnamon EOs (CCN), curcumin-garlic EOs (CGN), curcumin-sunflower oil EOs (CSN) with the mean droplets sizes of 9, 40, and 130 nm, respectively [81]. The nanoemulsion systems are used to deliver various bioactive compounds including fish oil, corn oil, β-carotene, lemongrass EOs, soy oil, oleic palm oil, polyphenols, fatty acids, vitamins, flavonoids, and others to food system and edible food packaging. Additionally, nanoemulsifiers are also used for the delivery of bioactive components, which has several benefits including modified texture, enhanced antioxidant property, improved emulsion stability, increased bioavailability, and other attributes. The nanoemulsions are available as single nanoemulsion, double nanoemulsion, pickering nanoemulsion, structural nanoemulsion (single or double interface layer) [82].

11.5.3 Nanoencapsulation

As discussed in Chap. 9, bioactive compounds are extensively used in several food systems for providing significant health benefits such as anticancerous, antioxidant, antidiabetic, cardiovascular disease preventing activity, antimicrobial, antiproliferative, antiobesity, and others. However, the bioactive compounds such as curcumin, eugenol, nimbin, ginseng root, menthol, carotenoids, flavonoids, vitamins, tryptophan, eicosapentanoic acid, decosahexanoic acid, and others are generally encapsulated using different carrier agents to protect against environmental conditions. Nanoencapsulation is defined as a delivery system for bioactive compounds to food products with improved bioavailability, where the active or core materials are entrapped within matrix or shell materials or secondary material to develop nanocapsules. The core materials are the bioactive materials such as lipids, vitamins, minerals, antioxidants (limonene, black pepper, terpene), probiotics (bifidobacteria, lactobacillus), enzymes, and others. The shell materials are cellulose, CS, gum (gum arabic, sum acacia), lipids, and others. The nanoencapsulation-based delivery system includes the use of several components such as aqueous phase, oil phase, emulsifiers, cosolvents, and carrier materials. The nanoencapsulation of active ingredients is extensively done using modified polysaccharides, where the several fabrication approaches include spray drying, electrospray, reverse micelle, supercritical fluid, electrospinning, coacervation, etc. [83]. Besides, the nanoencapsulation of food bioactives and various nutraceuticals has been attained using various nanocarrier system.

11.6 Nanodelivery System of Bioactive Compounds in Edible Food Packaging

The several aspects of nanosystem-assisted bioactive compounds in edible food packaging have been represented in Fig. 11.6, which includes protection of bioactive compounds and food products; enhanced health benefits and consumer acceptance for improved food texture. The bioactive compounds including antioxidants, vitamins, and lipids are protected using nanoderived assemblies such as nanoemulsions, nanoliposomes, NPs with increased bioactivity and functionality [84]. The edible coating based on sodium caseinate consisting nanoemulsion of ginger EOs provides improved antimicrobial and antioxidant activity and quality attributes of chicken breast fillets [85]. The phenolic components are alkaloids, phytosterols, vitamins, oils, nutrients, minerals, dietary fibers, and others are transferred to edible films and coatings using nanodelivery system. The development of edible coating on chilled chicken fillets (storage conditions: 4 °C and 12 days) using curcumin loaded nanoemulsion systems (CCN, CGN and CSN) and pectin influences the attributes in terms of chemical property, microbiological quality, sensory properties, and others [81]. In this regard, the use of specified edible-coating system on chilled chicken fillets provides influenced microbiological attributes such as reduced yeast and mold growth, reduced total plate count, reduced

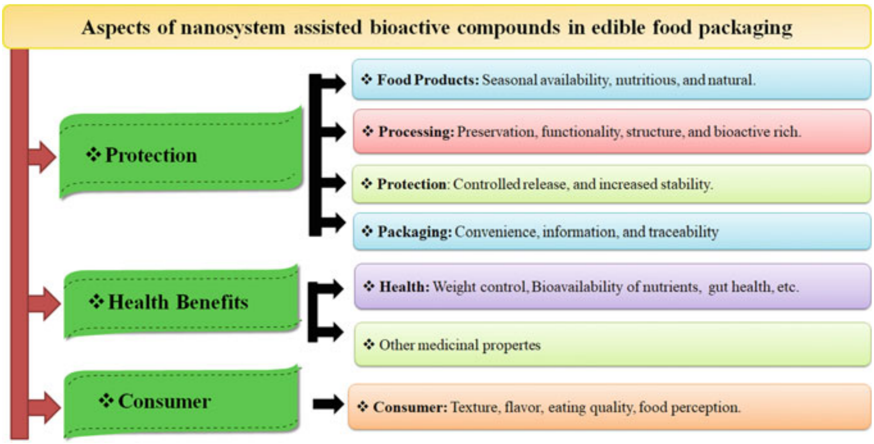


Fig. 11.6 Aspect of nanosystem-assisted bioactive compounds in edible food packaging

psychrophilic bacteria, and others. However, the use of pectin and CCN nanoemulsion-based edible-coating system for chilled chicken fillets provides better microbiological property in comparison with the other systems. The edible coating is better for chemical property such as thiobarbituric total volatile nitrogen and water holding capacity and texture property,. Additionally, the quality attributes and ripening of benitaka table grapes (BTG) can be controlled using edible coating based on gelatin hydrogel (prepared via enzymatic cross-linking using transglutaminase), bioactive NPs (nanoencapsulated curcumin in zein spherical NPs) or antifungal agents (calcium propionate) during a storage period of 7 days, at 25 °C and 50% relative humidity [86]. The use of gelatin hydrogel and nanoencapsulated curcumin-based edible coating on BTG can improve the color intensity and luminosity of the food product. Additionally, the use of edible coating based on gelatin hydrogel and calcium propionate can better control the ripening of BTG. On the other hand, the development of bioactive preservative coating on chicken fillets during a storage period of 12 days using starch nanocrystals (SNC) (fabrication process: sulfuric acid hydrolysis of potato starch granules) and sour lemon peel extract (ultrasonic extraction technique) offers improved properties in terms of physicochemical property, sensory property, and textural property [87]. The edible-coated strawberry using apple pectin, CNC, glycerol, and EOs of lemongrass has enhanced shelf life with improved quality (weight loss, titratable acidity, pH, total soluble solids, anthocyanin content) in comparison with uncoated strawberry products [88]. The edible packaging material based on gelatin and tea polyphenols (TP) encapsulated CSNPs with encapsulation efficiency of 66.7 ± 9.9% (developed by electrospray technique) helps to provide improved stability of oxidizable oil over 14 days of storage conditions. Further, use of 30% TP encapsulated CSNPs in gelatin provides enhanced barrier, mechanical, and improved free radical scavenging property [89]. The tea-based bioactive compounds such as catechin and epicatechin polyphenols provide improved properties such as neuroprotective property, antioxidative, antitumorogenic, antipathogenic and are used as a component in food systems [90]. The application of edible nanocoating based on nanoemulsions of α-tocopherol (<200 nm) and nopal mucilage on fresh-cut apple provides controlled enzyme activity against pectin methylesterase and polyphenol oxidase and controlled browning [91]. The edible multilayer nanocoating based on κ-carrageenan/CS can deliver controlled release of a model bioactive compound with improved functionality [92]. The edible nanocoating based on resveratrol loaded zein nanofibers (electrospinning) on cut apple slices provides controlled moisture loss and color retention property [93]. The encapsulated resveratrol (using zein nanofiber) provides enhanced bioaccessibility up to 67.6%, whereas the bioaccessibility of native resveratrol is about 48.1%. The development of edible nanocoating based on fresh-cut apple using nanoemulsion of vitamin E, potassium sorbate (antimicrobial agent), calcium chloride (antioxidant agents), fresh lemon juice delivers better properties, enhanced nutritional value, and shelf life [94] (Table 11.2).

Table 11.2 Delivery of bioactive components using nanosystem-assisted edible films and coatings

Sl. No.	Components	Nanosystem	Attributes	References
1	Curcumin EOs (Cinnamon, garlic, sunflower) Pectin	Curcumin loaded nanoemulsion (Edible coating)	Increased shelf life of chilled chicken fillets Microbiological property Improved sensory attributes	[81]
2	Gelatin hydrogel (Enzymatic cross-linking using transglutaminase) Bioactive NPs: NC in zein spherical NPs Antifungal agents: Calcium propionate	Bioactive NPs (Nanoencapsulated curcumin) (Edible coating)	Preserved quality of BTG Addition of NC to edible coating provides increased color intensity and luminosity of BTG Addition of calcium propionate offers controlled ripening of BTG	[86]
3	SNC Lemon peel extract	SNC (Bioactive preservative coating)	Application of edible coating on chicken fillets Improved quality in terms of physicochemical, texture, and sensory attributes	[87]
4	Apple pectin CNC Glycerol EOs of lemongrass	CNC (Edible coating)	Enhanced shelf life of strawberries Improved quality of strawberry Minimization of weight loss of strawberry	[88]
5	Gelatin TP encapsulated CSNPs (CS-TP)	TP encapsulated CSNPs developed by electrospray technique (Edible packaging material)	Improved stability of oxidizable oil for 14 days Use of 30% CS-TP in gelatin provides enhanced barrier, mechanical and improved free radical scavenging property	[89]
6	α-tocopherol emulsion Nopal mucilage	Nanoemulsion of α-tocopherol	Application of edible nanocoating on fresh-cut apple Controlled enzyme activity against pectin methylesterase and polyphenol oxidase Controlled browning in fresh-cut apple	[91]

(continued)

Table 11.2 (continued)

Sl. No.	Components	Nanosystem	Attributes	References
7	κ-carrageenan/ CS nanolayer Methylene blue	κ-carrageenan/CS nanolayer (Edible nanocoating with improved functionality)	Multilayer nanocoating for controlled release of bioactive compounds Edible nanocoating with improved functionality	[92]
8	Resveratrol loaded zein nanofiber	Resveratrol loaded zein nanofibers (Edible coating)	Edible nanocoating on apple slices Enhanced bioaccessibility up to 67.6% of encapsulated zein nanofiber Controlled moisture loss and color retention property of apple slices	[93]
9	Nanoemulsion of Vitamin E Potassium sorbate (antimicrobial agent) Calcium chloride (antioxidant agents) Fresh lemon juice	Vitamin E nanoemulsion (Organic phase is Orange oil (source: dried orange peel))	Edible nanocoating on fresh-cut apple Enhanced nutritional value Inclusion of fresh lemon juice provides better properties of fresh-cut apple	[94]

EOs Essential oils; *BTG* Benitaka table grapes; *SNC* Starch nanocrystal; *CNC* Cellulose nanocrystals; *CS* Chitosan; *CS-TP* TP encapsulated CSNPs; *TP* Tea polyphenols; *NC* Nanoencapsulated curcumin

11.7 Safety Issues of Nanosystem-Assisted Bioactive Compounds

The several safety issues for using nanosystem-assisted bioactive compounds in edible food packaging include high dosage/above permissible limits, chemical reactivity, nanoscale reaction, size and shape, increased oxidative stress, free radical productions and others as shown in Fig. 11.7. Further, in complex biological systems, the severe problems such as increased free radical production and reactive oxygen species production occur, which can create adverse health effects such as increased inflammation, oxidative stress. The health degrading factors may occur due to polymer toxicity and solvent residues. Additionally, the intake of bioactive compounds above permissible limits creates detrimental effect on kidney, liver and

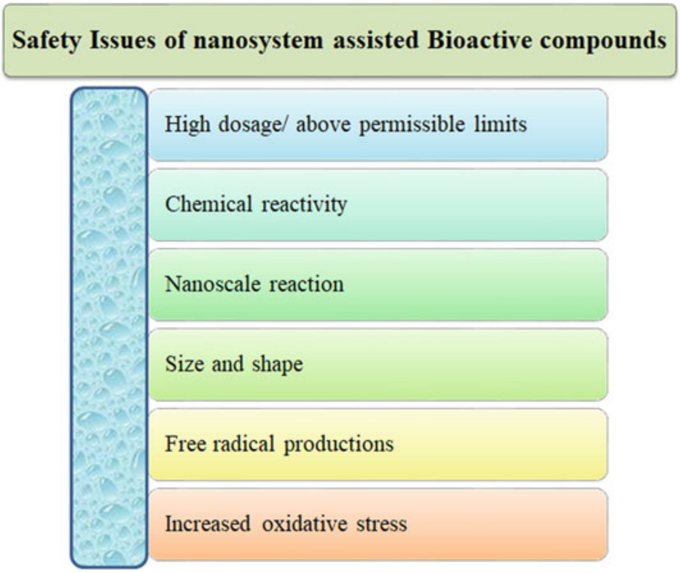


Fig. 11.7 Safety aspect of bioactive compounds in edible food packaging

skin, immunetoxicity. However, the toxicity effect of nanocarrier delivery system includes physicochemical, physiological, molecular consideration, interactions with cells, and others.

11.8 Conclusion

In this chapter, a discussion on bioactive compounds for edible films and coating with the aid of nanostructured materials of biopolymers such as CS, pectin and dextran with enormous potential has been made. Further, development of polymers with bioactive coating is an important area of research, with a target for solving the problem of contamination and bioactive rich food products. A detail discussion on the use of bioactive component within the coating and film-based matrix to make nutritious has been made. Generally, herbal drugs and EOs are incorporated into either natural polymers like CS, pectin and dextran by blending or composite approach. A wide range of natural bioactive agents such as aloe vera, curcumin, sandal wood oil, clove oil, and honey are available to develop excellent materials for edible packaging.

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Chapter 12
Nanotechnology in Antimicrobial Edible Packaging: A Candidate for Prolong Shelf Life of Food Commodities

Kona Mondal, Tabli Ghosh, and Vimal Katiyar

12.1 Introduction

In recent past years, there is a growing consumer's demand for minimally processed food products for being the fresh and convenient food products with maintained quality and safety, which has increased the inclusion of new packaging techniques to combat the modernized lifestyle. Further, the additional advantage of accessing digital media makes consumers for demanding fresh, minimally processed, nutritious, safe, ready-to-eat, and extended product life. Food packaging is one of the best ways in order to fulfill the demands for maintaining quality, authenticity as well as safety of food products throughout all segments of food. The concept of next-generation packaging has come into the current scenario based on the market needs where active and smart packaging systems are considered as a novel packaging system. This type of packaging technology is flourished with some important factors such as enhancement of shelf life, improved quality, and safety of food with a reduced food waste which supports the food security. Antimicrobial packaging (AP) is the subset of active packaging which is capable of suppressing the microbial growth and reduces the risk of food spoilages from pathogens and spoilage-causing microbial degradation followed by enhancing the product life [1]. The development of AP is executed in two different ways such as the incorporation of active ingredients or compounds into the packaging material and/or using active polymers with inherent antimicrobial properties [2]. This packaging system can act as bactericidal or bacteriostatic material thereby eliminate the spoilage causing pathogens. Additionally, the growing concern of global warming due to excessive waste food packages is one of the prime reasons for fabricating biodegradable and edible food packaging material which reduces the generation of packaging waste to some

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extent. For instance, biodegradable polymers are used mainly for developing biodegradable films, divided into bio- and non-biobased polymers based on the origin. Some commonly used semi-synthetic biodegradable polymers are poly (lactic acid) (PLA), poly(ε-caprolactone) (PCL), polyhydroxyalkanoates (PHA), polyhydroxyvalerate (PHV), polybutylene succinate (PBS), etc. Besides, starch, cellulose, chitosan (CS), agar are biobased polymers used for developing biodegradable packaging material. In addition, the mentioned sources are also utilized for the development of biodegradable thermoplastics-based packaging. Interestingly, nanotechnology has an immense impact in the food packaging sector and also in the development of AP. Using nanotechnology, the active substances such as antimicrobial agents can be delivered into the food system via packaging material. In modern times, more researches are carried out for a better outcome in this field. Based on the discussion, the current chapter addresses the fabrication, characterization, and application of nanotechnology in the development of antimicrobial edible packaging. The impact of nanotechnology in developing edible and non-edible AP is also being discussed. Additionally, the chapter also details the various types of antimicrobial agents based on their source and impacts against foodborne diseases, and others. This chapter also addresses the influence of AP material for an extended shelf life of food products and discusses the commercial and regulatory bodies for the safety concern and global market scenario of nanotechnology in the area of antimicrobial food packaging system.

12.1.1 Significance and Motivation of Antimicrobial Packaging

The contaminated food products are inedible as can cause foodborne diseases when consumed. The growth of foodborne pathogens and spoilage-causing microorganisms are the main sources of contamination which further causes loss of color, flavor, and altered texture and nutritive values (Fig. 12.1). The occurrence of food contamination can happen at different processing stages of food manufacturing, where foods are directly exposed to the environment at the time of harvesting, slaughtering, food processing, and finally, during packaging. In addition, conventional food preservation techniques are being used to avoid contamination through heating, drying, freezing, salting, fermentation, which helps to increase the shelf life.

However, recontamination has been observed in various stored food products, which can make food products unpalatable and leads to the generation of food waste that can indirectly corelate to the food economy. Therefore, the idea of minimizing the microbial activity at the final stage and development of new preservation techniques as an outcome of AP has been established. Besides, the increasing concern of improved shelf life along with the maintained quality of food products has raised a need for more effective regulation system for maintaining food protection, preservation, and transportation to consumers [3]. Nowadays,

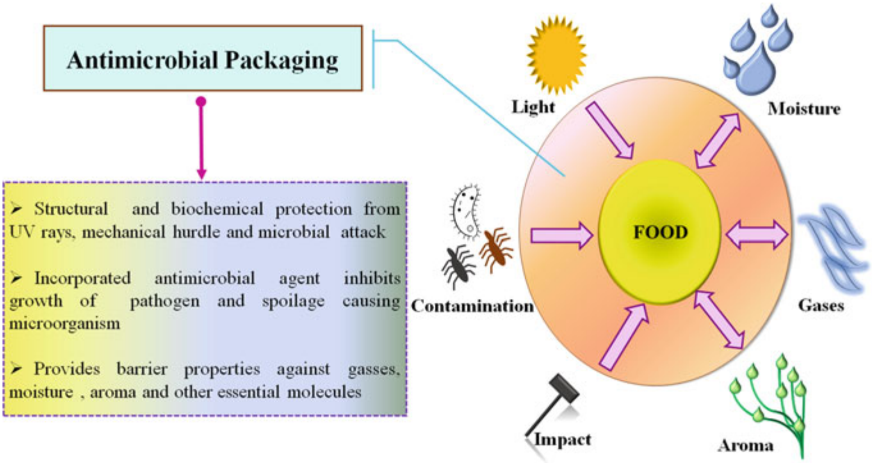

Fig. 12.1 Schematic diagram of antimicrobial packaging showing protection of food components from various factors

consumers are interested in consuming organic products as they possess less added chemicals and provide healthy molecules [3, 4]. Therefore, the necessities of efficient natural food preservatives as a replacement of chemical preservatives have occurred in the food industry. Based on the requirements, various antimicrobials and antioxidants have been extracted from grapefruit and other natural sources which have shown their effectiveness in the laboratory scale [2, 5–8]. However, the commercialization of food products incorporated with natural active compounds requires further research to observe the occurrence of any characteristic change in food material.

AP has been developed by the addition of antimicrobial agent into the polymeric films or by using inherent antimicrobial polymers for slowing down the activities of specific microorganisms. These targeted microorganisms are selected based on the type of food and chances of growth of particular microorganisms into that specific food material [9]. The goals of AP generally follow the reverse order of traditional packaging systems. In this context, AP first concerns about the assurance of food safety followed by maintenance of food quality and shelf life extension of food [2]. Moreover, the aim to minimize the waste plastic generation from food packages and several biobased multifunctional antimicrobial agents-incorporated packaging materials have started developing with ensured food safety as well as deliver several beneficial attributes [9]. The achievement of antimicrobial effects has been obtained through the prevention of microbial growth by using antimicrobial agents-incorporated packaging materials. Usually, the antimicrobial agents extend the lag period and reduce the exponential growth or log phase of the microbial growth cycle where antimicrobial agents slow down the microbial activity [2]. However, food security is one of the biggest challenges and, therefore, this specifically designed AP system has been developed to provide long shelf life and safe food. In this regard, dairy, meat, bakery, fruits, and vegetable products are more prone to the microbial attack due to the easy availability of nutrition for the growth of microbes. AP will be the most effective preventive measures in this case for enhancing the storage life of perishable foods [10]. In this context, the periphery of AP has been started creating a major interest in extensive scientific research.

12.1.2 Role of Nanotechnology in Antimicrobial Packaging

The rising concern of fresh food with retained quality and safety has started pressurizing on food industries worldwide. Therefore, the development of new technology is essential for the fabrication of new packaging material solutions which will provide cost-effective, biodegradable packaging material with maintained food quality. In this context, the concept of nanotechnology has gained wide acceptance toward introducing diverse applications in the area of food packaging [10]. Nanotechnology predominantly consists of fabrication of nanodimensional molecules (<100 nm) which are further characterized for analyzing the actual nature of nanomaterials and required modifications of nanomolecules where the concept and particular term were invented by Richard Feynman in 1959 and Norio Taniguchi in 1974, respectively. The three different possible ways of introducing nanotechnology into the packaging system are the addition of nanomaterials straight into the food, direct addition to the packaging material, and can be added during the processing of food products.

From the past few years, the use of nanostructured materials as food packaging components has gained wide acceptance by the consumers due to its significant functional properties. However, direct incorporation of nanomaterials into agro-food products is less acceptable [10]. From the aspect of food packaging, nanomaterials have been utilized for developing food nanopackaging materials with characteristic features of containment, convenient, protection, and preservation of food with proper marketing and communication to meet the consumer's need [11, 12]. Additionally, food nanopackaging is the most active area for nanoscience research and development. The release of antimicrobials, antioxidants, flavors, nutraceuticals, and many other significant beneficial components can be attained through nanopackaging technology, which helps in maintaining the food quality and further improves the shelf life of food products [8]. In order to design and meet the effective requirements of food packaging, polymeric materials are filled with nanoparticles and nanodevices [13–15] to develop new nanopackaging materials. Aiming to provide fresh and quality packaged food along with suppressing the food packaging waste, AP has earned massive attention. Figure 12.2 depicts the various aspects of nanotechnology for development of AP. Many commercial pristine polymers fail to provide desirable properties including antimicrobial activity. Thus, composites are developed by incorporating filler materials or blending with other polymers for obtaining desirable properties. The nanomaterials having antimicrobial characteristics are introduced into the packaging systems to develop

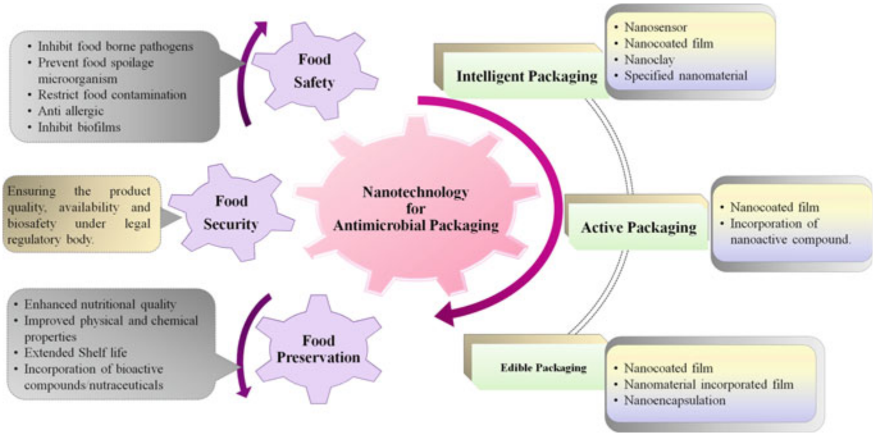

Fig. 12.2 Utilization of nanotechnology for development of antimicrobial packaging

nanosystems-aided AP. However, based on the origin, different types of nanoparticles/nanomaterials are available as antimicrobial agents; among them, two broad categories are inorganic and organic nanoparticles. AP material can be developed in different ways, mainly through preparation of nanocomposites which are mostly non-edible, whereas nanocoating, nanolaminate, antimicrobial film is used for edible packaging [16, 17]. Polymer nanocomposites become the most popular novel food packaging solution, which are the mixture of polymer as a continuous phase or as matrix material with filler material (inorganic or organic nanoparticles) as a discontinuous phase [18, 19]. The nanodimensional materials exist in various forms such as nanoparticles, nanorods, nanowhiskers, nanosphere, nanotubes, nanofibers, nanosheets, and nanoplatelets based on their morphological property [19]. The introduction of specific nanomaterials into the polymer matrix also provides enhanced mechanical strength and restricted gas and moisture barrier with additional antimicrobial properties [20]. In addition, the aspect ratio is defined as the ratio of length to the diameter of nanoparticles and can influence the properties of nanocomposites where highest aspect ratio provides better reinforcing ability. The chitosan nanoparticles (CSNPs) and its various derivatives are the biopolymer-based antimicrobial nanoparticles, whereas silver (Ag), copper (Cu), zinc (Zn) nanoparticles and the oxide of titanium, Zn, silicon, magnesium are used as inorganic nanofillers in the development of food packaging material due to their antimicrobial activity. Edible nanocoating of food products is developed using edible components in combination with other edible nanomaterials such as active agents having antimicrobial activity, where food products are either dipped coated or spray coated. In this context, a US company developed nanocoating for bakery products [21]. Similarly, nanolaminates are developed via multiple layer depositions of polymers with an added antimicrobial agent for the generation of edible coating material. Furthermore, edible and non-edible antimicrobial films are developed by including agro-based extract into the edible polymer matrix such as protein, carbohydrate, and lipids, whereas non-edible films can be developed by incorporating some available inorganic nanoparticles. The carbon nanotubes are found to have antimicrobial activity; however, the cytotoxic nature limits its application in food packaging [22, 23]. Moreover, nanotechnology through various antibacterial nanofilms can able to reduce the processed food waste and preserve the fresh produce after harvesting by covering the product with nanocoating, which keeps the food fresh, thereby extending the shelf life. The nano-antimicrobials can keep the processed food safe and fresh for longer storage time before consumption.

12.1.3 Scenario of Antimicrobial Packaging in Laboratory Scale and Commercial Market

The laboratory scale research on AP has gained significant interest due to the growing concern of safe food. The laboratory scale study on antimicrobial activity and their release mechanism from packaging film to the stimulant are less complex in comparison with the real food system. It is well known that food is a complex system with higher water activity, nutritious components enriched with protein, carbohydrate, fats, and other micronutrients, less salinity which provides an enriched environment for the easy growing of microorganisms [24]. Besides, the storage and transportation condition of food products have a significant influence on the activity of antimicrobials with food microbes which are different from laboratory scale stimulants. The release of antimicrobials is greatly influenced by two important parameters such as moisture content and temperature [25]. In addition, the migration rate of antimicrobials is important which defines how many antimicrobials are able to release through diffusion over the concentration gradient and saturation of released antimicrobials at the food surface and in the headspace [2]. However, the releasing phenomenon also depends on swelling and water uptake of films other than diffusion. The analytical and chemical tests can determine the quantity of antimicrobials released from the film; however, for real food achieving the same result is difficult and challenging [26, 27]. The literature says the effectiveness of antimicrobials is less in the real food system as compared to laboratory scale experiments [7, 28]. It is found that a high quantity of antimicrobial loading is required for achieving better activity than laboratory scale, probably due to higher organic acids, trace metal availability of nutrients, and interaction with compounds that may inactivate the working capacity of active substances [3]. For example, Kim et al. [7] reported the lesser affectivity of nisin released from commercial non-edible low-density polyethylene (LDPE) film in the extension of shelf life of oyster and ground beef packaging in comparison with laboratory scale. This kind of outcome reveals the lack of experimental essential data such as pH, released and retained substance, growth kinetics of targeted microorganism for monitoring, and thus, more research is required for revealing the actual scenario.

12.2 Composition of Antimicrobial Packaging Systems

Food packaging is one of the most reliable conventional techniques of food preservation and protection, which has been continuously investigated to fabricate an advanced packaging system via incorporating antimicrobial agents. The addition of antimicrobial agents can further enhance the product storage life by preventing the growth of spoilage and pathogens causing food degradations. Therefore, the design of AP consists of two principle parameters such as AP material and antimicrobial agents. In this regard, this section discusses the different types of packaging material and several available antimicrobial agents which are allowed for food packaging.

12.2.1 Antimicrobial Packaging Material

The biodegradable and non-biodegradable packaging materials are the two principle categories under AP. In addition, microorganisms present in nature are capable to produce low molecular weight fragments from complex polymeric structures (high molecular weight) including biodegradation of packaging material via microbial attack, which are known as biodegradable packaging. The edible films and coatings are developed by utilizing mostly carbohydrates (mostly starch, CS), protein, lipids, whereas non-edible packaging materials are produced from chemically synthesized thermoplastic polymer, namely PLA, PCL, several categories of PHA, and others which are biodegradable in nature. In the case of non-biodegradable packaging, microorganisms are not able to degrade the high molecular weight polymeric substances including conventional polymers such as high-density polyethylene (HDPE), LDPE, polystyrene (PS), nylon, polyethylene terephthalate (PET), and others. The conventional polymers are majorly chosen as food packaging material over others due to its various significant properties including cost-effectiveness, excellent gas and moisture barrier properties, great mechanical strength, transparency, low density, inertness, heat sealable, and easy printable [29]. In contrast, at the same time, these synthetic materials are the cause of occurring global pollution. Thus, the focus has been shifted toward biodegradable and biobased packaging as a desirable solution for minimizing the environmental pollution in recent days. Interestingly, CS is one such biopolymer derived from chitin of crab shell and prawn, which possess advantageous characteristics of biodegradability, biocompatibility, non-toxicity, wide spectrum of antimicrobial activity, and edible nature [30]. Besides, the most popular biodegradable thermoplastic, PLA, is synthesized from renewable resources through the process of fermentation, condensation, and ring opening polymerization. Further, PLA has shown better antimicrobial activity as a polymeric matrix after incorporation of active compounds. Moreover, poly (vinyl alcohol) (PVA) is another synthetic biodegradable polymer, which has been blended with other natural and synthetic polymers due to its biocompatibility, hydrophilicity, non-carcinogenicity, and stable nature. Tripathi et al. have reported about the blends of CS/PVA with improved biological characteristics [31]. Additionally, edible AP films and coatings are one of the emerging candidates for extension of product shelf life, where edible polymers are used as packaging material with added food additives such as antimicrobial agents, plasticizers, colorant, flavors, emulsifiers, and stabilizers.

12.2.2 Antimicrobial Agents

As mentioned earlier, the AP system has been formulated by the addition of antimicrobial agents into the polymeric matrix materials. Since food products are more affected by the microorganism, therefore making the food free from contamination is essential for preservation and enhancement of the shelf life of products, where the AP system is a potential candidate. Various antimicrobial agents are incorporated into the packaging material for obtaining the desirable antimicrobial property. Moreover, for a better understanding of different types of antimicrobial agents, it can be categorized into two broad zones, namely organic and inorganic antimicrobial agents. However, few researchers also classified antimicrobial agents into three groups, namely chemical agents, natural agents, and probiotics [2]. Further, these are the parts of organic and inorganic antimicrobial agents. In this regard, Table 12.1 represents the various antimicrobial agents with their effectiveness on food products.

Table 12.1 Several antimicrobial organic and inorganic agents with their beneficial effects on food products

Antimicrobial agent	Origin	Nature of packaging material	Targeted microorganism	Effectiveness	References
Oregano oil/ pimento oil	Biobased (EO)	Edible	*Escherichia coli, Pseudomonas* spp.	• Enhanced shelf life of beef muscle meat till 7 days • Suppressed growth of targeted microbes by 1.12 and 0.95 log reduction	[32]
Clove oil	Biobased (EO)	Edible	Gram-negative bacteria	• Showed highest inhibitory efficiency against spoilage and pathogen causing bacteria and	[33]

(continued)

Table 12.1 (continued)

Antimicrobial agent	Origin	Nature of packaging material	Targeted microorganism	Effectiveness	References
				natural microbiota in chilled storage fish • This study predicts the antimicrobial agent can extend the shelf life of stored chilled fish	
Rosemary oil	Biobased (EO)	Non-edible	*Coliform, Pseudomonas* spp.	• Suppressed growth of *Coliform* effectively in refrigerated chicken breast cuts	[34]
AgNP/ modified clay	Non-biobased (inorganic)	Non-edible	*Staphylococcus aureus, Escherichia coli, Candida albicans*	• Improved barrier and mechanical properties of film along with antimicrobial property of film	[35]
CS oligomer	Biobased (biodegradable polymer)	Non-edible/ edible	*Yeast, Mold*	• Significant reduction of molds and yeast growth, while storage of perishable food products such as strawberries, ricotta, and flavored bread in sachets for 7 days	[36]
GSE	Biobased (fruit seed extract)	Edible	*Listeria monocytogenes, Escherichia coli*	• The log reduction of 0.50 and 0.53 CFU/g of targeted microbes has achieved during 15 days chilled storage of salmon	[37]

(continued)

Table 12.1 (continued)

Antimicrobial agent	Origin	Nature of packaging material	Targeted microorganism	Effectiveness	References
				• GSE has shown antioxidant activity by reducing the PV value and TBA value by 23.0 and 23.4%	
GSE	Biobased (fruit seed extract)	Edible	*Escherichia coli, Listeria monocytogenes, Staphylococcus aureus, Pseudomonas aeruginosa, TMAB*	• Enhanced shelf life of vacuum packaged chilled storage chicken breast fillets • The log reduction of 1.50–2.33 in TMAB and *Coliform* count during storage period	[38]
Nisin	Biobased (enzyme)	Edible	*Listeria monocytogenes*	• Improved shelf life of refrigerated ricotta cheese during the storage of 28 days • Improved antimicrobial activity • Significant bacteriostatic activity	[39]

AgNP Silver Nanoparticle, *CS* Chitosan, *EO* Essential Oils, *GSE* Grapefruit Seed Extract, *PV* Peroxide Value, *TBA* Thiobarbituric Acid, *TMAB* Total Mesophilic Aerobic Bacteria

12.2.2.1 Organic Antimicrobial Agents

Essential Oils

Essential oils (EOs) are recognized as generally recognized as safe (GRAS) substances, which can be used as natural food additives [40]. In addition, EOs are the agro-based organic extract associated with strong antimicrobial activity. Being a GRAS element, the use of EOs in the development of biobased films and coating has been studied. Researchers also have found potential interest in EOs due to its antimicrobial property for applying into active packaging. However, many studies are ongoing in a laboratory scale, which needs to be implemented in a commercial level. Some of the widely used EOs with their characteristic attributes have been demonstrated in this section.

Among available, carvacrol is an organic antimicrobial compound, obtained from oregano EO, which has been used as active agents. Interestingly, the addition of more than one EO into a film material has been found to provide synergistic antimicrobial activity. However, the use has been found in both edible and non-edible packaging materials. In this regard, the addition of carvacrol and thymol together in the conventional non-edible HDPE-based nanocomposite film with modified montmorillonite (MMT) nanoparticles has displayed improved antimicrobial activity [41]. Another similar synergistic antimicrobial effect has observed in the direct application of film for strawberries, storage against *Botrytis cinerea*. The synergistic effect has been found effective by determining the half maximal inhibitory concentration (IC_{50}). The reduced value of IC_{50} (13.2 mg/g) has confirmed the synergistic effect oregano EO while added into the carvacrol-incorporated film (IC_{50} = 40.4 mg/g). However, oregano EO itself has shown the antimicrobial activity against *Alternaria alternate* on tomatoes [42].

Cinnamon EO is another most effective antimicrobial agent, wherein cinnamaldehyde is the major compound responsible for showing the highest antimicrobial activity. It has been reported that multilayer coating material incorporated with cinnamaldehyde showed affectivity against *Escherichia coli* and *Saccharomyces cerevisiae* by 3 log CFU/mL reduction. These two microorganisms are the major spoilage-causing microbes in a food system. Furthermore, obtaining better performance of cinnamon EO, β-cyclodextrin can also be added together into the PLA matrix and the developed PLA nanofibers (via electrospinning) have a tendency to reduce the loss of volatile substances and further enhance the shelf life of pork storage at 25 °C for 8 days as compared to control (storage of pork in normal film without an added antibacterial agent) [43]. Additionally, Higueras et al. reported the controlled release of cinnamaldehyde of cinnamon EO via reversibly anchoring to CS polymeric film through an imino-covalent bond [44], where there can be obtained a reduction in the growth of *Listeria monocytogenes* (an easily available pathogen in milk under refrigerated condition).

Thyme, an herb of genus *Thymus*, has been used to offer improved shelf life and safety of cooked cured ham due to its antimicrobial property [37]. The major compound in thyme EO is carvacrol. The thyme EO has shown its effectiveness during storage period of sliced cooked pork (ready-to-eat) under refrigerated condition. The thyme EO is also able to suppress the growth of aerobic mesophilic, lactic acid bacteria, and yeast counts in food products for 21 days [45]. Basil leaf essential oil (BEO), an antimicrobial agent, showed extended storage life of sea bass slices wrapping with BEO-incorporated fish protein composite films, under refrigerated for 12 days [46]. Further, vanillin has exhibited antimicrobial activity against *L. monocytogenes* on crab sticks packed food [47], whereas *Allium* spp. extract inhibits the mold growth in lettuce during the storage of 7 days. However, research is still going continuously for commercialization of various EOs with antimicrobial property, extracted from clove, tea, coriander, laurel, rosemary, sage, and others [48]. The mentioned EOs along with other available EOs have the potentiality to work as an active agent, though more research needs to be done for real-time use or commercialization. Moreover, the focus must be given in analyzing the impact of EOs on the food physicochemical property, the interaction between food, packaging material and EOs, the organoleptic changes (flavor, taste, and odor) on food, and the manufacturing of such packages under real-time condition. However, the inappropriate consumption of EOs may have health risk based on the current research [49]. It has been reported that natural antioxidants can act as pro-oxidants, able to produce free radicals, and can damage deoxyribonucleic acid (DNA) and mutate the gene [50]. Therefore, more research is necessary for optimizing the dose level and future effects of natural antimicrobials onto the food component.

Enzymes and Bacteriocin

Available proteins specifically enzymes and bacteriocins deliver antimicrobial activity into packaging material by inhibiting the food spoilage and pathogenic microorganism. The affectivity of enzymes as antimicrobials can be obtained through chemical bonding, physical interaction with the packaging material. In this section, lysozyme is mainly focused as an antimicrobial agent which is approved by the US Food and Drug Administration (USFDA 2001) [51] and further, it is accepted as a food additive (E1105) in Europe (European Union 1995) [52]. Lysozyme, one of the most potential antimicrobials, is effective against gram-positive bacteria. The working principle of lysozyme is obtained by breaking down the glycoside linkage and peptidoglycan layer of microorganisms. Min et al. reported the enhanced shelf life of smoked salmon due to the application of AP, where lysozyme has been incorporated into whey protein film [53]. This study shows the reduced activity of *L. monocytogenes*. Furthermore, lysozyme along with another antimicrobial agent, lactoferrin, shows inhibitory effect against microbes while incorporated in the carboxymethyl cellulose (CMC)-based paper sheet upon thin meat slice [54]. However, this study proved that lysozyme is more effective than lactoferrin. In addition, other natural proteins such as whey, zein, and casein are also available which has been used as antimicrobial agents in the development of AP.

Bacteriocins are produced through a group of lactic acid bacteria which enable to inhibit the growth of food spoilage-causing bacteria. Moreover, bacteriocins are composed of peptides or smaller chains of proteins, which are toxic against microbes especially for gram-positive bacteria. Nisin, one of the promising bacteriocins, has shown restriction of bacterial growth over food products such as hot dogs, beef, milk, orange juice, cold-smoked salmon, and others. The coating of nisin-incorporated (10,000 and 7500 IU/mL) cellulose derivatives when used as a packaging film for storage of hot dogs (refrigerated storage till 60 days) has found to offer a significant reduction of *L. monocytogenes* on the surface of hot dogs [55]. Besides, nisin-coated commercial LDPE films showed a high impact on milk during the refrigerated storage of raw and pasteurized milk at 4 °C for 7 days [25], where there has been shown a log reduction of microbial count by 0.9 and 1.3 CFU/ml, respectively.

It has been reported that nisin along with other antimicrobial agents showed an inhibitory effect on the growth of *Brochothrix thermosphacta*, *coliform;* as a result, the shelf life of beef and fresh oyster mushroom got has improved [56, 57]. Another study has revealed that the immobilization of nisin and ethylenediamine tetraacetic acid (EDTA) in the presence of a cross-linking agent genipin, over the surface of cellulose nanocrystals (CNCs)/CS films, has shown the restricted growth of psychrotrophs, mesophiles, *Lactobacillus* sp., *L. monocytogenes*, and *E. coli* in fresh pork loin meat and further provides extended shelf life of about 35 days [58]. Similarly, the storage life of refrigerated drumsticks was found to improve by the coating of a mixture of components including nisin, citric acid, EDTA, polyethylene glycol (PEG), and sorbitan monooleate over commercial polymers such as polyvinylchloride (PVC), nylon, or linear LDPE [59]. The growth of *Salmonella* species on cooked ham has found to be inhibited by nisin with enterocins, sakacin, and potassium lactate [60]. Besides, other bacteriocins such as natamycin, pediocin, enterocins, and lactocins are incorporated into bio-polymer based films and coatings, have been found to possess restricted bacterial and fungal growth in cooked ham, wieners, gorgonzola cheese, sliced ham, and others. In addition, nisin (E234) as food preservative has been approved by European authority under the Directive 95/2/EC on food additives (European Union 1995) [52]. However, more study needs to be carried out for the controlled release of bacteriocin and enzymes along with their safety and synergistic effects in the presence of other antimicrobials.

Polymers with Antimicrobial Activity

Chitosan and its nanoparticles

CS is a heteropolysaccharide-based biopolymer with inherent characteristics of antimicrobial and antifungal activity, which has been proven to be beneficial for food packaging applications. The presence of polycation in nanochitosan (NCS) biomolecules is responsible for delivering the antimicrobial property [61]. Besides, CS becomes an attractive polymer due to its other properties such as non-toxic nature, film-forming ability, and biodegradability. It was reported that CS solution restricts the growth of *E. coli* and *Staphylococcus aureus* by 1 log reduction [59]. In addition, the coating of CS solution on polymeric films also provides mechanical support. Further, CS has been used as an antimicrobial food additive in non-biodegradable polymer LDPE [62] and biobased polymer CMC [63] for developing coated and biocomposite films. CSNP is a derivative of CS, formed by ionic gelation, wherein amino groups with positive charged ions of CS interact with polyanions by electrostatic interaction [64, 65]. It has been found that addition of CSNP-based nanocomposites provides antimicrobial, mechanical, and barrier properties in food packaging film [66]. Recently, edible films and coatings are developed by utilizing CS as matrix material along with other edible biopolymer, which have shown antimicrobial activity with enhanced shelf life of freshly cut fruits and vegetables [67, 68]. The storage life of tomato has been extended by the application of CS-based antimicrobial film which is able to suppress microbes such as *E. coli*, *S. aureus*, and *Bacillus subtilis* [31]. Similarly, antimicrobial bags have been developed using CS and non-edible polyethylene (PE), which provides inhibitory action against molds, yeast, *coliforms*, and total mesophilic aerobic bacteria (TMAB) for chicken drumsticks, maintaining other properties (pH, color, and hardness) of the product [69]. The increased shelf life of refrigerated soft white cheese is found to be around 30 days at 7 °C using CS-based bionanocomposites, where the film is active against total bacteria, yeast and mold counts, and *coliforms* [63]. CS along with other antimicrobial agents has shown wide affectivity against a broad range of microbes including *Aspergillus niger*, *Candida albicans, Pseudomonas aeruginosa* along with previously mentioned microbes [70, 71]. Similarly, CS coatings with other added antimicrobials such as nisin, sodium lactate, sodium acetate, potassium sorbate, and sodium benzoate can inhibit *L. monocytogenes* on ham sticks [72], and cold-smoked salmon (for 42 days) [73]. Besides, the biodegradable non-edible polymer, PLA coated with a mixture of CS (5%), lauric arginate ester (5%), sodium lactate (2%), and sorbic acid (0.3%), has shown to provide inhibition of *S. typhimurium* and *L. innocua* within 48 h on surface-contaminated turkey slices with 3 log reduction of microbial count [74]. Higueras et al. have displayed antimicrobial activity of CS films with added lauric arginate ester against lactic acid bacteria, mesophiles, psychrophiles, coliforms, yeast, fungi, *Pseudomonas* species, and hydrogen-sulfide-producing bacteria on chicken breast fillets [75]. It must be mentioned that CS has been approved as a GRAS substance by USFDA. Furthermore, a detailed research needs to be carried out for observing the changes in the sensory and organoleptic property of foods under real-time experiments.

Starch Nanocrystals

Starch, another explored biopolymer, has been studied as a food packaging material due to its biodegradability, non-toxicity, biocompatibility, low cost, easily available properties, and others [76]. The starch nanocrystals are developed by hydrolyzing native starch granules at a particular temperature below its gelatinization temperature, wherein hydrolysis occurs at the amorphous regions keeping the crystalline lamellae intact which are resistant to hydrolysis and finally removes the amorphous domain. In addition, platelet-type morphology of starch nanocrystals has been observed with a thickness of 6–8 nm [18, 77]. Moreover, starch nanocrystal has an ability to improve the tensile strength and modulus of pullulan films whereas elongation has reduced. In addition, the occurrence of antimicrobial activity of any antimicrobial agent is due to the presence of positive charges. In this context, Arora et al. have proposed the addition of metals onto polysaccharide surface, which can provide or enhance antimicrobial spectrum [61].

Organic Acids and Other Compounds

Some of the organic acids and organic compounds have inherent antimicrobial nature and, therefore, can be utilized by incorporating into the packaging films.

Citric Acid

Incorporation of citric acid into an extruded film of corn starch/LLDPE has shown a reduction of total bacterial count by approximately 1 log CFU/g of minced beef in comparison with control sample [78]. In this study, 30% citric acid is used which ultimately enhanced the storage life of minced beef. Further, various salts of citric acid have been utilized as a potential source of antimicrobial agents. However, further investigations are required for a wide spectrum application.

Sorbic Acid and Potassium Sorbate

Sorbic acid is composed of a straight chain of α, β-unsaturated monocarboxylic acid. Different salts and esters of sorbic acid are also formed during the interaction of the carboxyl group with sodium, calcium, potassium, and other esters. The development of non-edible poly(vinylidene chloride) films with the addition of 1.5 and 3% w/v sorbic acid has been used to wrap beef bologna and cheddar cheese which are contaminated by *L. monocytogenes* with a loading of 10^3 and 10^5 CFU/g, each for beef and cheese, respectively [79].The restricted microbial count of *L. monocytogenes* is observed after 28 days of refrigerated storage (4 °C) of beef, which is inoculated with 10^5 CFU/g. In comparison with control, around 4.4 log reduction is obtained through sorbic acid-incorporated film. However, no significant restriction of microbial growth is found in cheese samples for *L. monocytogenes* and mesophiles after 35 days of storage period under the same refrigerated condition, whereas beef samples with 10^3 CFU/g inoculation load are found to be lower as compared to control against *L. monocytogenes*. Besides, increasing the sorbic acid concentration has an ability to reduce the count around 6.5 and 7.2 log for 1.5 and 3% loading of sorbic acid, respectively. In another study, sorbic acid is mixed (0.5% and 1%) with algal extract (*Fucus spiralis*) and incorporated into PLA films, which shows restricted microbial growth on megrim, a type of flatfish [80]. This study reveals a reduction in 0.9 log CFU/g of psychrotrophs in comparison with control after 7 days of storage period with a lower value of aerobes and Enterobacteriaceae count. However, no significant antimicrobial effect is found in 11 days of storage life of fish. Sorbic acid-based antimicrobial film shows improved sensory properties. Under the Commission of European Regulation, the use of sorbic acid as a food additive in meat products has been restricted (European Commission 2011) [81]. Sorbic acid is only allowed for specific applications in some meat products including jelly coating, collagen-based casings, dried meat products, and others. Further, the scaling up of sorbic acid-incorporated packaging materials at an industrial level requires further study.

Potassium sorbate is commonly used salt of sorbic acid which works best at lower pH. The antimicrobial activity of potassium sorbate has been found to be effective against food pathogens like *E. coli, Serratia liquefaciens,* and others. Cestari et al. have investigated the effect of potassium sorbate in starch/ polybutylene adipate terephthalate-blended non-edible biodegradable films on chicken steaks under frozen storage [82]. The study has shown no detection of *E. coli* count after 30 days of storage, wherein control sample has a microbial growth. Similarly, the effectivity of potassium sorbate as antimicrobial agent is found in brine solution on smoked rainbow trout fillets stored under refrigerated condition (~6 °C) for 4 weeks [83]. In this study, sodium lactate is separately mixed with potassium sorbate in brine solution and is experimented to obtain the better performance. The fillets are submerged into the sodium chloride (8%) solution before processing to be smoked fillet. The highest inhibited mesophilic count is observed by around 3 log reduction due to potassium sorbate among other brine solution. This study has also found the effectivity against *S. liquefaciens,* which is most common pathogen in smoked fish.

Potassium Metabisulfite

The fresh produces such as fruits and vegetables become unacceptable when got degraded due to several enzymatic reactions along with microbial attacks. These fresh produces are highly liable to the enzymatic browning. In this context, potassium metabisulfite (PMBS) has been used as food additives, working as antibrowning, antimicrobial, and antioxidant agent for extending the product life of the fresh produces. The effectiveness of PMBS has been experimented on gala apple cut, where the cut apples are wrapped within the active film containing PMBS-incorporated non-edible PVC film with varying loading concentration of 1% and 2% for longer storage at different temperature ranges (4, 8, 12, 16, and 20 °C) with 30% relative humidity (RH) [84]. The observation states an extended shelf life and lowered browning in apple cut, confirming the affectivity of added food additive. In comparison with control, shelf life of apple increases from 4 to 8 days at a storage temperature of 8 °C. Further, apples wrapped with 2% PMBS are shown to provide microbial stability and toxicological stability for 20 days of storage. This study suggests the best consumption of cut apple before 12 days of storage period at 8 and 12 °C.

Oxidized Regenerated Cellulose

Another bio-based food additive has been reported by Sezer et al. where oxidized regenerated cellulose microparticles had been utilized for providing antimicrobial activity [85]. The antimicrobial activity is obtained from the active non-edible film of PCL incorporated with oxidized regenerated cellulose (4% w/w) on sliced salami contaminated with *L. monocytogenes*. The counts of inoculated microbe are reduced by around 50%, after the storage period of 14 days at 4 °C. Besides, the active PCL film has been found to decrease the activity of *E. coli* and *S. Aureus* as well as lowers the permeability of water and oxygen by 70% and 93%, respectively.

Allyl Isothiocyanate

Another GRAS-approved antibacterial food additive is allyl isothiocyanate. This additive has been used in commercial packaging in various forms of sheets, label, and films. In addition, these films are available in the Japanese market under the trademark Wasaouro™. Additionally, the antimicrobial activity is confirmed by Pang et al. on fresh catfish fillets against *P. aeruginosa* with extending the product life up to 4 to 5 days, 11 days, and 23 days at 8 °C based on the amount of used allyl isothiocyanate of 11 μg/L, 18 μg/L, and further combined with modified atmosphere packaging (MAP), respectively [86]. Similarly, Kim et al. have reported the antimicrobial activity against a wide range of bacteria. However, the pungent and strong smell of this additive often restricts its acceptability [87]. Besides, allyl isothiocyanate has been approved for using as a food additive for a wide range of food products based on their safe chemical characteristics with food. The focus must be given on the development of AP for commercialization and fulfilling the specific legislation by the standard authority.

12.2.2.2 Inorganic Antimicrobial Agents

Several metals predominantly Ag, Cu, gold (Au), Zn, and metal oxides such as titanium dioxide (TiO_2), zinc oxide (ZnO), magnesium oxide (MgO), and silicon oxide (SiO_2) have been reported in the literature because of their significant antimicrobial activity. In recent days, nanoparticles derived from these metal and metal oxides have gained immense interest due to their advanced and rapid mechanism of antimicrobial activity in the area of active packaging [88]. Moreover, these inorganic nanoparticles can show their activity when in a direct contact with the food surfaces as well as can migrate from packaging material into the food substances and have a tendency to react with organic matters. The improved antimicrobial activity might be due to their different mechanisms of action toward microbes. The inorganic antimicrobial agents can interact directly through penetrating the cell envelope, which disrupts the cell wall, and alters the transmembrane electron transfer, and oxidize the cell component by changing their redox potential and produces secondary metabolites such as reactive oxygen species (ROS), which are responsible for damaging the microbial cell (Fig. 12.3).

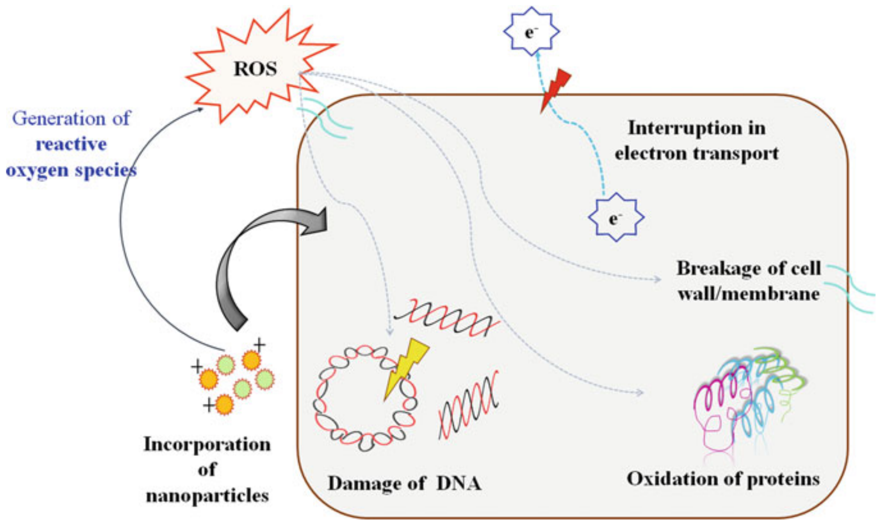

Fig. 12.3 Schematic of the mechanism of action of nanoparticles and inorganic compound upon microbial cell

Silver Nanoparticle

One of the most common potential nanomaterials, widely used as antimicrobial agents in the food packaging system, is silver nanoparticles (AgNPs). From the ancient era, Ag has been used as an antimicrobial agent. The reported literature reveals the effectivity of AgNP against food pathogen and spoilage-causing bacteria, fungi, yeast, and viruses [89, 90]. The bacteriostatic effects of Ag have been observed, where it interferes with the bacterial metabolism through binding to cellular substances such as proteins, DNA, and enzymes [91] (Fig. 12.3). In addition, AgNP has shown the antimicrobial activity by disrupting the cellular membranes, which produces ROS and inhibits the function of respiratory enzyme [92]. Various edible and non-edible AgNP-based nanocomposites have been developed, which are stable and able to release silver ions slowly from packaging or coating material into the food organic system, resulting in potential antimicrobial activity and extension of shelf life. The ability of AgNP against the growth of microorganisms depends on their nanodimension. The antimicrobial efficacy of AgNPs in hydroxypropyl methylcellulose (HPMC) with a diameter of 41–100 nm inhibits the growth of *E. coli* and *S. aureus,*, whereas a smaller particle of AgNPs with less than 41 nm shows greater activity as compared to bigger size particles. Nowadays, many studies have been done on AgNPs to showcase the antimicrobial activity in food packaging material. Azlin-Hasim et al. report the enhanced shelf life of chicken breast fillets by developing AgNPs-incorporated non-edible PVC nanocomposites [93]. Lin et al. have compared the antimicrobial effect between cellulose/CS/Ag and cellulose/CS nanocomposite films, wherein Ag-incorporated film has shown better activity against microbes [94]. Similar studies reported about the remarkable antibacterial activity against *E. coli* and *S. aureus* through AgNP-incorporated sodium alginate films [95].

Besides popular non-edible packages, exploring the antimicrobial activities on real food, edible films of AgNP-incorporated alginate have been developed and applied on sterilized carrots and pear [95]. Similarly, fresh asparagus spears coated with AgNP/polyvinyl pyrrolidone edible film provide an extended shelf life of asparagus by 25 days under refrigerated condition [96]. Furthermore, AgNPs provide the prolong shelf life and antimicrobial activity in the presence of other incorporated nanoparticles and antimicrobial food additive. In this context, rice stored under commercial LDPE packaging shows a serious mildew condition with an increasing trend of total plate counts (TPC) by 2.3 log CFU/g after one month. On the other hand, there has been found to obtain lesser TPC count in rice stored with Ag/TiO_2-incorporated LDPE for the same time period [97]. Another study has reported the enhanced bread storage life through minimizing the degradation rate of major nutritional components by using Ag/TiO_2-based packaging material as compared to common plastic packaging [98]. This Ag-based packaging also showed effectivity against yeast and mold growth, namely *B. cereus* and *B. Subtilis*. Furthermore, Li et al. have documented that AgNP along with TiO_2-incorporated non-edible PE nanocomposite bag delayed the ripening as well as lowered the decaying rate of Chinese jujube fruit for a period of 12 days [99]. Emamifar et al.

displayed non-eatable LDPE incorporated with TiO_2 and AgNPs (10 nm) has restricted the growth of *Lactobacillus plantarum* in orange juice under refrigerated condition (4 °C) for the storage period of 112 days [100]. The blended nanocomposite of agar and banana powder with added AgNPs showed immense antimicrobial activity against *E. coli* and *L. monocytogenes.* The incorporation of banana powder is responsible for displaying the improved characteristics of ultraviolet (UV) light absorption, water vapor barrier (WVB), and antioxidant property of the blends; however, mechanical properties have decreased. In recent studies, cellulose pads incorporated with AgNPs deliver reduced antimicrobial growth in fresh foods with lowering the ripening rates and extended the shelf life [101, 102]. This nanocomposite further reduces the microbial exudates in meat products together with MAP and restricts the microbial growth in beef coating [103]. Besides, non-edible LDPE incorporated with TiO_2, nano-Ag, and ZnO delivers the antimicrobial activity with expanded shelf life of orange juice up to 28 days. In addition, Ag–zeolites are another antimicrobial agent used for developing polymer nanocomposites. It has been observed that the use of Ag–zeolites in packaging material provides better immediate antimicrobial effect in comparison with AgNP-incorporated active films.

Other Antimicrobial Nanoparticles

The TiO_2 nanoparticle (TiO_2NP) is another promising candidate for contributing antimicrobial activity among other inorganic nanoparticles. Basically, TiO_2 is a metal oxide which exists naturally in three different forms, namely anatase, rutile, and brookite with varying crystallite size [104]. Further, TiO_2 has been explored in various forms of nanomaterial such as nanoparticles, nanorods, nanowires, nanotubes, mesoporous, and nanoporous [104]. The nanoscale TiO_2 provides surface activity through which metabolic molecules such as proteins and DNA of microorganisms get attached and inactivated [105]. The antimicrobial property of TiO_2 is well known; however, the nanodimensional TiO_2 possesses better activity and can provide a barrier against the exposure of UV radiation [106]. However, the bactericidal activity of TiO_2 is still unclear. Kubacka et al. have proposed the possible mechanism of function of TiO_2, where the nanoparticle attacks the cell membrane of the microbe followed by altering the activity of enzyme and further produces the ROS which allows damaging of DNA [107]. The reduced microbial count of *E. coli* is observed on freshly cut lettuce, coated with TiO_2NP-incorporated oriented non-edible PP film after 3 h of illumination as compared with uncoated sample under same condition. Similarly, TiO_2NP-incorporated ethylene vinyl alcohol (EVOH) films have shown antimicrobial activity against major pathogenic and spoilage-causing bacteria of nine species [108]. Moreover, TiO_2 nanoparticle along with other food additive (Ag) has been reported showing significant improvement in the antimicrobial property.

Silica nanoparticles (SiNPs) are able to deliver the improvement in mechanical and barrier properties to the food packaging material. The SiNP-enriched films hinder the passage of gases and moisture, thereby preventing food from the occurrence of spoilage which increases the shelf life of food products. In food packaging, nanosilica is kept in contact with food surface materials. The nanocomposite with 5% SiNP improves the mechanical and physical properties of the film reported by Salami-Kalajahi et al. [109].

Besides, ZnO has been explored in food packaging material due to its enriched antimicrobial activity. Further, decreasing the particle size of ZnO enhances its antimicrobial activity [110, 111]. Sirelkhatim et al. reported the possible antimicrobial mechanism of ZnO, wherein it reacts with the cell wall of microbes and resulted in disrupting the cell integrity while releasing the antimicrobial ions (Zn^{+}) and generation of ROS during direct contact with food surface [112]. ZnO is found to be effective against *S.aureus* as compared to MgO and calcium oxide (CaO) additives and is also effective for *Streptococcus sobrinus* against dental biofilm formation [113, 114]. Nano-ZnO in various forms including powder, film, and coating has antimicrobial effects against *L. monocytogenes* and *Salmonella enteritidis* in liquid egg white [115]. It has been reported from a comparisonal study between ZnO nanoparticle and ZnO powder that ZnO nanoparticle displayed more effectivity against tested food pathogens [99]. In addition, ZnO has found to be a great antimicrobial agent in comparison with essential oil. Petchwattana et al. have reported a greater antimicrobial effect of non-edible PBS/ZnO films as compared to thymol-added PBS film against *E. coli* and *S. aureus* [116]. Similarly, carvacrol-incorporated PBS film has shown significant antimicrobial efficiency; however, the film is unacceptable due to its unpleasant odor [117]. In this context, ZnO-incorporated HDPE, polylactide, and polypropylene (PP) film has reported significant improvement in antimicrobial activity without generation of odor [118–120].

12.3 Construction and Function of Antimicrobial Packaging System

AP is an attractive innovation of active packaging. Besides, AP provides improved food safety and food security through restricting the growth of pathogenic and spoilage-causing microbes. Moreover, the attributes of antimicrobial activity are achieved by the addition of antimicrobial or active agents and using active functional polymers. For instance, the pictorial view indicates the working mechanism of active compounds (Fig. 12.4). This system kills or inhibits the growth of microbes by arresting their nutrients. The perishable and fresh produces (meat, fish, fruits, and vegetables along with bakery products) are easily attacked by the microorganisms, which can be protected by developing the AP with targeted inhibition of specific microorganisms. The antimicrobial materials prevent the microbial growth via extending the lag period of their growth cycle subsequently decreasing the growth rates of microbes. This active packaging system is fabricated by adding microbial agents in sachets/pads and in packaging materials. Further, the mechanism of each antimicrobial agent is different on targeted microbes depending on the characteristics of antimicrobials and physiological condition of microbes. It

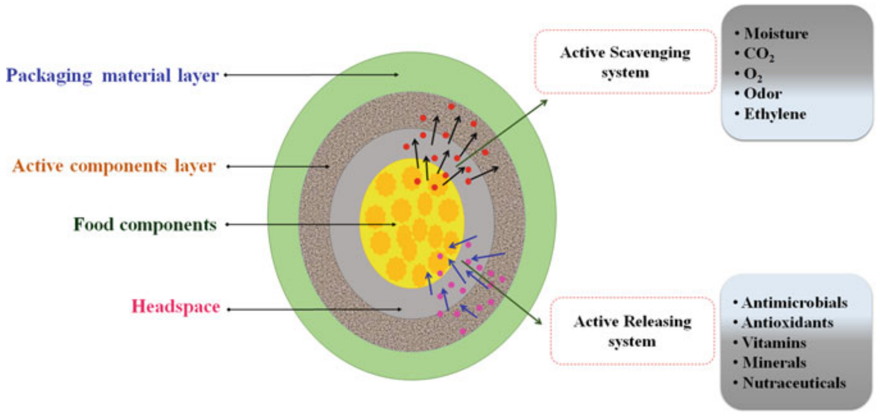

Fig. 12.4 Schematic representation of mechanism of active components in food packaging

is necessary to understand the characteristic of antimicrobial functions and their limitations. Some antimicrobials attack the metabolic pathway of microbes by altering regular enzyme function reproductive system, while others target the cell wall or membrane to inhibit or kill the microorganisms (Fig. 12.3). In addition, hurdle technology is the key factor in AP. The hurdle technology includes water activity, pH, redox potential, heat treatment, and others to obtain optimum lethality of microorganisms through the combined approaches. The working principle of hurdles should be in such a way that must cause minimum damage to food nutritional and sensory properties [121]. In food products, microorganisms are inhibited by reducing water activity, maintaining low or high temperature, reducing the pH, and by addition of competitive microbes and preservatives. The hurdle effects are obtained by combining any of two or more mentioned parameters. The utilization of hurdle technology in food products must ensure food safety and adequate shelf life. The addition of antimicrobial agents in combination with other technology like MAP, controlled atmosphere packaging (CAP), and other incorporated additive provides hurdle technology for protecting food quality from deterioration, enhancement of the shelf life for longer storage without altering conventional parameters including moisture, oxygen barriers, and physical protection.

12.3.1 Functions of Antimicrobial Packaging

The added antimicrobials into packaging material display retardation against microbes through three different modes such as release, absorption, and immobilization. Generally, the food packaging system mostly consists of packaging material, food, and headspace inside the package. Figure 12.5 represents the food packaging system containing headspace (free volume), food, and package as well as

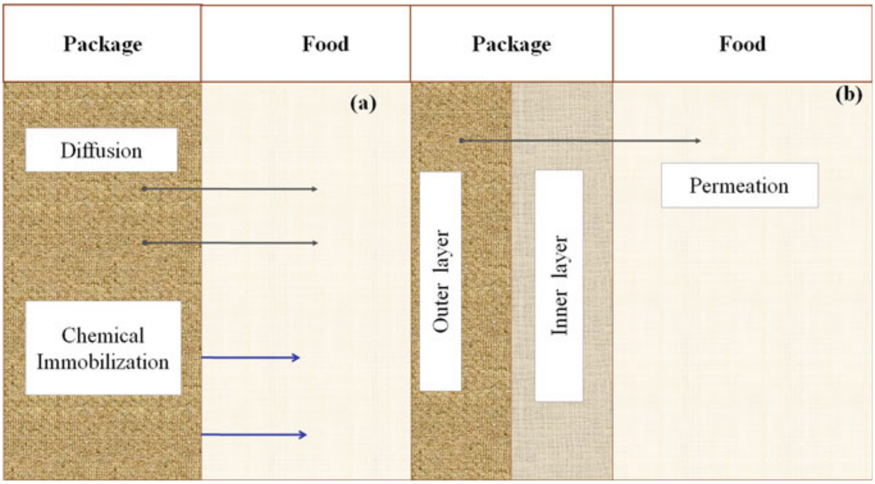

Fig. 12.5 Schematic diagram of various modes of construction of packaging material with food and migration of antimicrobials toward food matrix. **a** Migration of antimicrobials in one-layer system of package and food, and **b** two-layer system packaging with food

a package/food system. The package/food system is mostly applicable for liquid or low viscous food. The common example of this type of food products includes wrapped cheese and aseptic brick packages. Besides, the common examples of package/headspace/food systems are bottles, flexible packages, cans, cups, and cartons. A brief description has been made on the mode of functioning of antimicrobials as discussed in this section.

Release of Antimicrobials

In release mode, the antimicrobials slowly migrate from the packaging material directly into foods or headspace inside the packages (Fig. 12.6). Subsequently, these released antimicrobials retard the growth of pathogen and spoilage-causing microorganisms. In package/food system, the embedded antimicrobials are migrated through diffusion between the packaging material and food and at the interface (Fig. 12.6). Further, the diffusion and partitioning are the principle migration phenomena occurring inside the package/food system [122]. The phenomenon of evaporation is the main mechanism of migration in package/headspace/food system, where volatile antimicrobial agents are used. The equilibrated substances may diffuse into the food. Additionally, in comparison with the non-volatile substances, volatile substances can migrate through the headspace and air gaps between the packaging film and the food substances.

Absorption Mode

In absorption mode, antimicrobial restricts the supply of essential factors including moisture, oxygen, carbon dioxide, and pH, which are required for the growth of microorganisms inside the food system and eventually retard the growth of

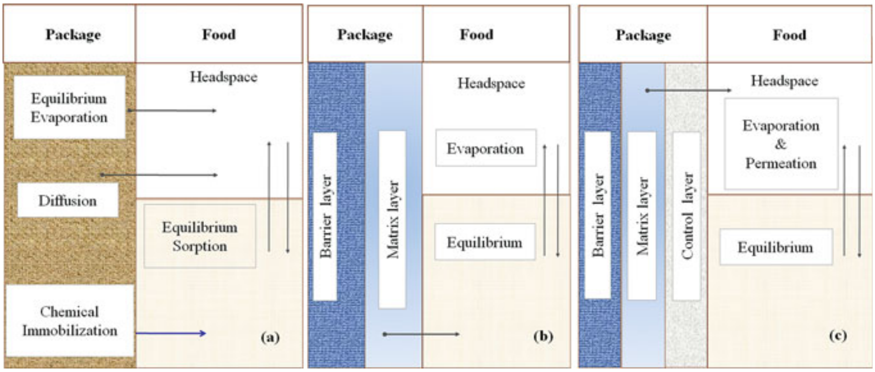

Fig. 12.6 Schematic diagram of various modes of construction of packaging material with food and migration of antimicrobials toward food matrix. **a** Packaged food with headspace, **b** two-layered packaging along with headspace and food, **c** three-layered packaging along with headspace and food

microbial counts. In addition, the antimicrobial agents can control and further act as carbon dioxide and oxygen scavenger, moisture absorber, and ethanol emitters, which indirectly prevent the growth of microorganisms.

Immobilization

Immobilization is another mode of antimicrobial action besides absorption and release. The immobilization system does not release antimicrobial agents and, however, suppresses the growth of microorganisms by contacting the food surface. Several chemical agents predominantly enzymes, antibiotics, or fungicides are immobilized on the polymer packages and inhibit the microorganisms through surface contact with food materials without diffusional mass transfer (Fig. 12.6a, c). Among available, lysozyme and glucose peroxidize are common antimicrobial agents covalently immobilized onto packaging material for cheese, beef, and culture media. In this context, the immobilization system is more effective in liquid foods due to the occurrence of direct contact of food with an immobilized antimicrobial system which allows greater surface area.

12.4 Different Types of Antimicrobial Packaging

The available various types of antimicrobial packaging include volatile antimicrobial agents in sachets/pads, polymers incorporated with volatile and non-volatile antimicrobial agents, coating of antimicrobials onto polymeric films, immobilization of antimicrobial agents via ionic or covalent linkages on polymer film surfaces, and natural antimicrobial polymers.

12.4.1 Sachets or Pads Containing Antimicrobial Agents

Sachets or pads are loosely bound to the inner wall of the food package in case of AP. Volatile antimicrobials can be used in sachets for inhibiting the growth of microbes. The common sachets used in antimicrobial packaging contain oxygen absorbers, moisture absorbers, and vapor of ethanol emitters [1]. Moreover, the oxygen scavengers are utilized to hinder the occurrence of oxidation and inhibit the growth of microorganisms, whereas moisture absorbers retard the growth of microbes by lowering the water activity in bakery, pasta, and meat product packaging. The ethanol emitters containing sachets are used to prevent mold growth by generating ethanol vapor mostly in bakery and fish products [123].

12.4.2 Antimicrobial Packaging with Direct Incorporation of Antimicrobial Agents

Nanocomposites and composites of polymers are developed by incorporating the antimicrobial agent directly, wherein nanodimensional antimicrobials are much effective as mentioned earlier. The commercial application of antimicrobial agent-incorporated polymer has been observed in pharmaceuticals and for other applications and recently, in food packaging system. In case of food packaging, the two most predominant categories of polymer matrix directly incorporated with antimicrobial agents are non-edible and edible food packaging. The AgNPs and Ag–zeolites can be incorporated into several conventional polymer matrixes such as PE, PP, nylon, and others, which hinder the enzyme activity of microorganisms, thereby reducing the growth of microorganisms. Furthermore, the concept of using biodegradable polymers is growing nowadays due to the growing concern of minimizing plastic generated hazards. Among biodegradable polymers, PLA has attained much attention in comparison with other available biodegradable polymers. Furthermore, antimicrobial enzymes, proteins, antioxidants, EOs, natural phenols, and metals like Cu have wide applications in developing composites, however, though research is required for complete commercialization. A variety of nanomaterials are used to develop nanocomposites containing a maximum of 5% w/w nanoparticles for improving several characteristic properties such as barrier properties, mechanical strength, antimicrobial activity, and others. The improved properties of nanomaterials are obtained through the addition of other food-grade additives as synergistic effects. In case of edible packaging, antimicrobial agents-incorporated polymers have been utilized by developing edible films and coatings. Nanotechnology has been implemented in developing edible nanocoating and nanofilm for obtaining better antimicrobial performance. In this context, nisin-incorporated edible films in combination with EDTA are used to reduce the effective growth of *E. coli* [124].The fabrication of edible food packaging and effect of nanotechnology in developing food packaging material are discussed in below section. In addition, application of nanocomposites in direct contact with foods has been approved by USFDA [125].

12.4.2.1 Application of Edible Antimicrobial Food Packaging

As discussed in Chap. 2, the edible food packaging materials can be defined as continuous matrices of edible substances including protein, polysaccharides, and lipids. The two main broad categories of edible packaging are edible coating and edible films, where the preparation of edible coating material with the release phenomena of antimicrobial agents has been represented in Fig. 12.7.

Antimicrobial Edible Coatings

The application of thin layer of selected edible formulation in liquid form onto the whole food surface can be defined as edible coating and is obtained by using different techniques including spraying, dipping, and immersion [126].The edibility of coating or film is attained when all the agents of packaging systems such as biopolymers, plasticizers, and other additives are in food grade as well as all the processes and required equipment in developing coatings and films must be approved for food processing purposes [127]. Among available edible coating approaches, the predominant coating techniques have been described below.

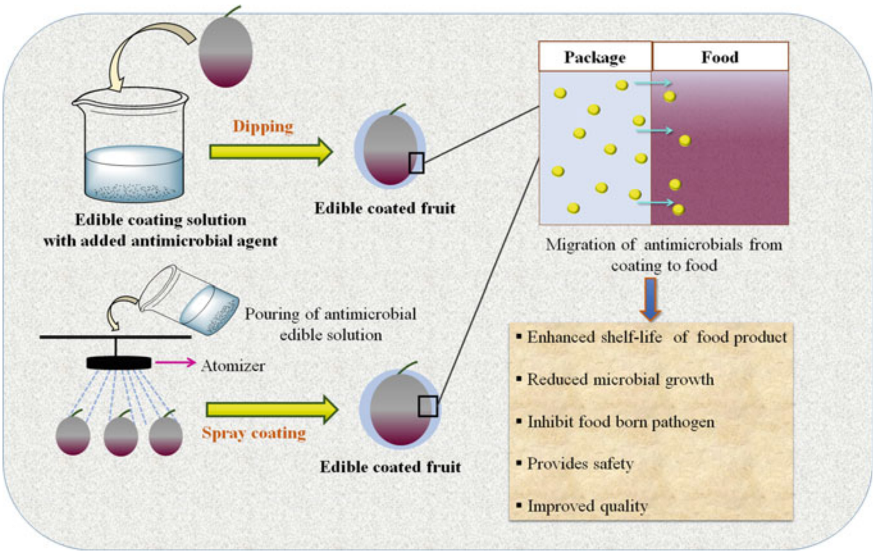

Fig. 12.7 Schematic diagram of edible antimicrobial coating with migration of active molecules

Spraying

Besides some of the existing conventional techniques, one of the best techniques of applying edible coating is spraying, which provides high-quality product at a lower cost. Generally, the conventional technique involves high temperature, leading to valuable losses of volatile antimicrobials. On the contrary, a uniform coating, controlled thickness, and multilayer coating can be achieved through the spray technique. Besides, this technique provides no contamination of coating solution, a temperature control facility, and continuous automated production. Therefore, spraying-based edible coating possesses stability of volatile antimicrobial agents via controlled temperature and better release of antimicrobials through controlled thickness of coating [128]. Spray coatings can combine hydrophilic and hydrophobic substances by two ways either forming emulsion solution before atomization or formation of bilayer after two sprays, which has some disadvantages of requiring more steps of double drying and spraying [129]. Interestingly, spraying of low viscous coating solutions can be done by applying high pressure. Further, electrospraying is able to produce uniform particles (<100 nm) from the distributed drop size, whereas sprayed coating solution can provide particle size up to 20 μm from polymer and biopolymer solution. Further, time and temperature have an impact on the formation of polymer coating during spraying [130]. Spraying converts bulk liquid into droplets through atomization. Further, this technique increases the liquid surface area; therefore, rapid evaporation can occur during the process. The homogeneous and uniform coating with controlled thickness allows quick release of antimicrobial agents into the surroundings and helps to evaluate the release kinetics of the antimicrobial additives.

Besides, electrospraying is another available spraying technique which includes atomization of coating solutions using air spray, pressure, and air-assisted atomization. Spraying technique is advantageous for heat-sensitive antimicrobial agents, wherein with a controlled heating system, antimicrobials can be delivered to the food system. In this context, an edible coating of alginate with added carvacrol and methyl cinnamate as natural antimicrobials has been applied on fresh strawberries by spraying technique [131]. The coated strawberries delivered firmness with original color and minimum reduction of weight loss in comparison with uncoated strawberry. Similarly, another sprayed edible coating of tapioca starch incorporated with green tea extracts, a natural antimicrobial agent upon fruit-based salads, romaine hearts and pork slices has shown restricted growth of aerobic microorganisms and yeasts [132].

Dipping

Dipping is another technique for edible coating which has been used for fruits, vegetables, and meat products [133]. However, this technique provides thick coating layers. The thickness of the film depends on the various factors of coating solution including density, viscosity, and surface tension [134]. A membranous film is formed after dipping the food products into the developed aqueous solution followed by drying. This process contains immersion, deposition, and evaporation from which excess solvent evaporates and a thin film is formed on food products. However, in the case of the dipping technique, a diluted coating solution is required for obtaining the better results. For instance, a significant amount of residual coating material is generated and the process requires further drying step. Therefore, this technique is restricted from the use of industrial applications [135]. Besides, the various examples of dip coating have been reported in the literature. The oyster mushroom has been coated by dipping into sodium alginate and sodium acetate solution and kept under MAP condition; as a result, enhanced shelf life has been attained [136]. A similar observation has been reported by Hamzah et al. on dip coating of papaya using k-carrageenan, and coating on carrots using solution of sodium alginate can offer extended shelf life [137].

Spreading

Spreading, which is also known as brushings, can provide controlled thickness through the attached blade at the lower part of the spreading device. This technique is required drying as the second step, which can be obtained by blowing hot air. The available reported literature suggests that protein and polysaccharide-based films can be developed by spreading technique [138]. The two main parameters of spreading are wetting degree and spreading rate. Several factors affecting the spreading approach include surface roughness and geometry of substrate, temperature, relative humidity, and properties of coating solution (viscosity, surface tension,, and density) [139]. It has been reported that spreading of highly viscous coating solution is much difficult than less viscous liquids. In addition, this technique is an alternative to film preparation. The inhibited growth of gram-positive (*L. monocytogenes*) and gram-negative (*E. coli, Salmonella*) microorganism is observed in CS-coated non-edible PE film by spreading technique [140]. A remarkable antimicrobial activity has been displayed by the sprayed coated surface of non-edible LDPE and cellulose-based active packaging films where the coating materials contain antibacterial peptide solutions [141]. In addition, the selection of coating techniques mainly depends on the type of product, coating thickness, properties of coating solution, and drying technique.

Nanocoatings

Edible coatings have been applied over a wide variety of food products including fruits, vegetables, chocolate, cheese, candies, bakery products, French fries, and meat products [142–144]. The incorporation of nanoparticles into edible coating/films provides better physical properties; however, few studies are available on the use of nanocoatings on food products. In addition, oxygen diffusion has been restricted after the addition of clay MMT or clay nanoparticle into pectin non-edible film [144]. In similar studies, nanocomposite of gelatine and MMT has been used for improving the physical properties of non-edible packaging material [145]. Another nanocomposite of CS incorporated with clay nanoparticle has been developed having better stability [146]. Moreover, nanoparticles can be utilized as a suitable carrier for delivering antimicrobials and additives. The addition of inorganic nanofiller in edible coating showed retention of flavor, taste, improved texture, appearance, increased stability, and reduced chances of spoilage. Besides, nanocoatings also provide the extended shelf life of food products for longer storage. For instance, a US based Company Sono-Tek Corporation developed an edible antimicrobial nanocoating on bakery goods [21].

Antimicrobial Edible Films

An edible film is a thin layered packaging material which is used for wrapping of foods [147]. As mentioned in Chap. 1, the first edible film was fabricated in Japan from soymilk, namely yuba, and used as a food preservation method in the fifteenth century [148]. Before that, in the twelfth century, China developed waxes and applied it over the oranges and lemons to hinder water loss [149]. Further, food products were coated with fat during the sixteenth century for controlling the moisture. During the 1930s in the USA, hot-melt paraffin waxes were applied to coat citrus fruits and further, an emulsion of carnauba wax and oil in water was used for coating of fresh fruits and vegetables during the 1950s [150]. Nowadays, the edible coating is utilized in various sections including as a carrier of antimicrobial agents and coatings of chocolate, fruits, nuts, and casing for sausages.

Moreover, proteins, polysaccharides, and lipids are the three major components of edible films. Proteins-based edible films utilize mainly wheat gluten, collagen, corn zein, soy, casein, and whey protein, whereas polysaccharides used in edible films are alginate, dextrin, pectin, cellulose derivative, and CS [151]. Further, films containing lipids are waxes, acylglycerols, and fatty acids [152]. Composite films have been developed with the presence of both hydrophilic and hydrophobic substances. The addition of food-grade plasticizers improves the final property of film which further decreases the brittleness and increases the flexibility of the film. Plasticizers are used in protein-based films, which reduce the protein interaction and enhance the chain mobility and intermolecular spacing of polymer chain [153]. Besides, plasticizers with high quantity can improve the barrier properties and mechanical strength, and decrease the elasticity. In addition, resistance, rigidity, mechanical strength, cohesiveness, and barrier properties of film are improved by the incorporation of cross-linking agent [154].

Several film-forming techniques have been developed for edible coatings such as thermal gelation, solvent removal, and solidification of melt. Hydrocolloid edible films are produced by the solvent removal technique. The formation of continuous structure is obtained in this technique, which can further be stabilized through the chemical and physical interactions between the molecules. Moreover, polymers can dissolve in a various solvent including water, ethanol, acetic acid along with other food additives including plasticizers, cross-linking agents, active components, antimicrobial agents, and others. The film-forming solution is casted on a support followed by drying and peeling as discussed in earlier chapters. During the development of protein films, heating is necessary for protein gelation and coagulation through denaturation, and/or precipitation and finally, rapid cooling. However, the denaturation of proteins breaks disulfide linkages within the protein molecule [155]. The disulfide bonds reformed together after casting of film-forming solution and linked with polypeptide chain together to produce the film structure.

Besides, lipid-based films are formed by melting and drying. Moreover, the edible antimicrobial films are used to improve the quality and safety of the food products along with extended shelf life, which minimizes the packaging loss. In addition, edible films are mainly applied to fish, meat, fruits, vegetable, fired products, and others.

Nanolaminates

Nanolaminates are composed of two or more layers of nanodimensional materials which are linked together by physical and chemical interaction. The layer-by-layer assembly deposition technique is one of the best methods for the development of nanolaminates. In this technique, the charged surfaces are coated with interfacial films containing multiple nanolayers of different materials [156]. Nanolaminates possess many advantages in comparison with conventional techniques for fabrication of edible coatings and films. This technique allows the use of various adsorbing substances for the preparation of different layers such as natural polyelectrolytes (protein and polysaccharides), charged lipids (phospholipids and surfactants), and colloidal particles (micelles, vesicles, droplets) [157]. Further, active functional agents including antimicrobials, antibrowning agents, antioxidants, enzymes, flavors, and colors can be incorporated into the films. The release of active compounds enhances the shelf life, while maintaining the quality of coated foods. The characteristics of nanolaminated coatings depend on the quality of film-forming solution and the entire coating solution, which has been made with edible ingredients. The different parameters of nanolaminates can be controlled through the film-forming solution, total number of dipping steps, dipping solution, and different other parameters including pH, ionic strength, dielectric constant, temperature, and others [158].

12.4.3 Natural Antimicrobial Polymers

Natural polymers with inherent antimicrobial properties applied to the food packages via film or coating include CS and poly-1-lysine polymers. The coating of CS on fruits and vegetables restricts the growth of fungi and made a barrier for nutrients of fresh produce against microorganisms [159]. The films and coatings of CS can also act as a carrier of antimicrobial agents including organic acids, essential oil, herbs, and spices [160]. These polymers inhibit the growth of microbes by damaging the cell wall and membrane through which essential intracellular components of microbial cells leach out [161]. In addition, polyamide films show antimicrobial properties after treating with UV irradiation due to the increasing concentration of amines over film surfaces [162]. Acrylic polymers also have bactericidal properties enhancing the shelf life of fruits and vegetables [163].

12.4.4 Polymers Immobilized with Antimicrobials Through Ionic or Covalent Linkage

The development of antimicrobial packaging system by immobilization technique involves an interaction with functional groups of both polymer and antimicrobial agents. The antimicrobial agents are embedded with functional groups such as enzymes, peptides, polyamines, and organic acids, whereas polymers accompanied with functional groups are ethylene vinyl acetate, ethylene methyl acrylate, ethylene acrylic acid, ethylene methacrylic acid, ionomer, nylon, PS, and others [161]. Besides functional groups, spacer molecules are also required to link antimicrobial agent onto the polymer surfaces. Spacer molecules help to move the antimicrobial agents, where the active site gets attached with microorganisms present on the food surfaces. However, during immobilization, antimicrobial activity per unit area reduces to some extent due to the altered conformation and denaturation of proteins, and peptides in the presence of solvents. In this context, various substances are added to protect and increase the activity of antimicrobial agents in polymers [164]. The release of antimicrobials from polymers to food surfaces depends on the types of bond in which ionic bonding permits slow release and in case of covalent bonding, there is a negligible occurrence of diffusion of molecules [161]. The addition of immobilized beta-galactosidase and glucose oxidase in packaging materials activates the lactoperoxidase system in milk, which extends the shelf life of milk [165]. Gram-positive bacteria are affected by the polymeric films immobilized with lysozyme and chitinase [161]. Further, more studies are undergoing with various immobilized enzymes and antimicrobials onto polymeric surfaces for developing antimicrobial packaging.

12.4.5 Polymer Surfaces Coated with Antimicrobials

The heat-sensitive antimicrobial agents are not suitable during the polymer processing; therefore, the coating is necessary to protect the agents. Besides, antimicrobial agents can be added to the polymer by the cast film method. In this regard, one such example is poultry products, which can be packaged using PE films coated with nisin and methylcellulose [166]. Similar studies include the application of a coating of nisin with zinc onto PE, which has been used in the packaging of poultry products [167]. At the early stage, waxes along with fungicides are a common coating material for fruits and vegetables to maintain the texture during the storage life. Another coating application includes (i) wrapping of potatoes with quaternary ammonium salts with wax and cheese and (ii) wrapping of sausages using coated wax paper and cellulose casing with sorbic acid, which is the first developed antimicrobial coating [152, 168]. Moreover, enzymes can also be coated along with adhesives on polymer surfaces [152].

12.5 Factors Affecting the Development of Antimicrobial Packaging

There are various factors which are responsible for developing effective antimicrobial packaging material. Among several factors, the selection of both matrix and the antimicrobial agent is the main factor of the fabrication of antimicrobial packaging system. Based on the selection of matrix materials and active agents, the physico-mechanical properties of the package can be modified. In this regard, the several factors affecting the antimicrobial package property have been listed in this section.

12.5.1 Chemical Nature, Process Condition, and Residual Antimicrobial Activity

The chemical nature of the packaging films is found to get altered due to the addition of antimicrobial agents. However, the effectiveness of antimicrobial agents may get reduced during film formation, distribution, and storage. In this way, the antimicrobials are selected based on the heat sensitivity of the antimicrobial substances during the processing of film (such as extrusion). Further, shearing force and pressure involved in the processing of film are also a factor to develop better antimicrobial edible packaging. The preparation of master batches of the antimicrobial agent in the resin is one of the solutions for minimizing the heat sensitivity problem [2]. For example, the addition of potassium sorbate powder (antimicrobial agent) into LDPE resin for the development of master batch includes extrusion processing, which can be obtained as pelletized form. Subsequently, these pellets can be incorporated into LDPE matrix to minimize heat decomposition [169]. Besides, other physical operation including lamination, printing, and drying needs to be characterized quantitatively. Some antimicrobial agents are volatile in nature which may lose during storage.

12.5.2 Characteristics of Antimicrobial Substances and Foods

The chemical interactions between food components and antimicrobial agent are another factor which affects the film characteristics. The antimicrobial agents and their release phenomena from packaging materials are also influenced by the food substances. Moreover, the activity of the antimicrobial agents can be altered by interacting with the food physicochemical properties. In this context, the pH of food products can alter the growth rate of targeted microorganisms as well as the degree of ionization of antimicrobial activity of organic acids and their salts [2]. It has been reported that at low pH, mold growth can be inhibited significantly by benzoic anhydride-incorporated non-edible LDPE film [170]. Moreover, the diffusion of sorbic acid can be reduced in edible methylcellulose/palmitic acid film by increasing pH [171]. The growth of different types of microorganisms occurs during the storage of food products at various conditions based on the chemical nature of food components. Additionally, water activity also influences the chemical stability and antimicrobial activity of the incorporated antimicrobial agents. For instance, the release rate of potassium sorbate from methylcellulose/HPMC film containing palmitic acid has reported to be more at higher water activity value. Therefore, the release kinetics of antimicrobial agents need to design based on all the intrinsic factors of food so that optimum inhibitory effect can be obtained.

12.5.3 Chemical Interaction of Additives and Film

The molecular weight and polarity of the polymer matrix can be altered after incorporation of food additives (antimicrobial agent); as a result, the physico-chemical properties of film may get altered. In this regard, the addition of high molecular weight and lyophilic antimicrobial agents is more compatible with LDPE polymer as being hydrophobic in nature [170]. Moreover, the solubility, ionic charge, and molecular weight of antimicrobial agents influence the diffusion rate of these additives from the packaging film [172]. The diffusion rate of ascorbic acid, potassium sorbate, and sodium ascorbate in calcium/alginate film at different temperatures (8, 15, and 23 °C) has been studied, where the highest diffusion rate of ascorbic acid and lowest rate for sodium ascorbate has been found at all temperatures, due to the different ionic states of added antimicrobials [173].

12.5.4 Storage Temperature

The antimicrobial activity of antimicrobial agents can be influenced by the storage temperatures. In general, storage under higher temperature allows an increased rate of migration of antimicrobial agents from the film and further, the antimicrobial activity of the packaging film slows down due to the higher diffusion rate. The diffusion rate of antimicrobials should be monitored in such a way that it can deliver optimum antimicrobial activity throughout the storage life of food, thereby extending the shelf life of food products [170].

Mass Transfer Coefficient

The simplest mode in migration of antimicrobial compounds from the package to the food is diffusion. In a multilayer packaging system, a thin layer coating of antimicrobial agents can be applied, where the migration and release phenomena are controlled by the thickness of the film. Further, the control release rate and migration phenomena of antimicrobial agents in food packaging systems containing one or more layers can be described by a mass transfer model (Fig. 12.6). This mathematical modeling can predict the release profile of antimicrobials at different time durations where the agents remain above the critical effective concentration.

12.6 Effect of Antimicrobial Packaging Against Foodborne Microorganisms

The risk of having foodborne illnesses can be caused for eating any spoiled food products, where the food may degrade in terms of nutritive value at any stage, and considerably, the consumers receive the end food products after going through a long food supply chain from the field to fork. The raw fruits and vegetable products undergo different stages of processing before transporting to end market such as harvesting, handling, processing, packaging, and transportation. The adverse impact of foodborne microorganisms on human health is occurred mainly due to the presence of pathogens. As discussed in Chap. 4, the most commonly available foodborne pathogens are *E. coli*, *L. monocytogenes*, *S. aureus*, *Campylobacter jejuni*, *Salmonella* spp., and others, found in fresh produces such as fruits, vegetables, fish, meat, and packaged food products. Interestingly, sometimes these pathogens can grow in adverse conditions by increasing resistivity against antimicrobials, adaptation capability in a new environment during transportation, and others. In this context, minimizing the impact of foodborne pathogens and delivering the safe quality food to the consumer are the prior concerns. The different food preservation techniques have been developed to minimize the risk of foodborne illness, where antimicrobial edible packaging is a type of new approach among all. The developed edible film and/or coating incorporated with antimicrobial agents can provide better affectivity against pathogen having a direct contact with food surface and further provide a protective boundary around the food throughout the storage life. It has been reported that essential oils and organic extract as well as inorganic nanoparticles have revealed the potential effect in minimizing the foodborne pathogens against specific targeted microorganisms based on the type of food products. As discussed in Sect. 12.2, the inhibition of microbial activity by the antimicrobial agents is obtained via damaging the protective cell wall or membrane of a microbial cell, altering the genetic components like DNA, generating ROS which oxidize the cell components and further lead to the death of microbes. Moreover, the edible antimicrobial packaging provides safe and quality food due to the efficacy of antimicrobial agents which further deliver many beneficial micronutrients to the food.

12.7 Research and Developments in Nanotechnology-Aided Antimicrobial Edible Food Packaging

As discussed earlier, the generally used antimicrobial components for developing edible coatings and films are CS, polypeptides (lactoferrin, natamycin, nisin), organic acids (malic acid, lactic acid, acetic acid, citric acid, sorbic acid, tartaric acid), acidulants (acetic acid, lactic acid, malic acid), EOs (thyme, clove, cinnamon essential oils, garlic, capsicum), plant extracts, mineral salts, oligosaccharides, etc [174, 175]. The application of edible food packaging such as edible coating acts as a delivery agent for several functional ingredients to attain the maximum shelf life of fruits and vegetables [176]. The edible packaging materials such as edible films and coatings act as a matrix material for the incorporation of antimicrobial agents, where the antimicrobial agents-aided composite materials are applied on fruits and vegetables for improved storage life by inhibiting the growth of microbes and other components. However, the other emerging application of edible films and coatings includes texture enhancer, incorporation of antimicrobial agents, incorporation of nutraceuticals, which helps in the increased shelf life of fresh food products [174]. In this regard, the antimicrobial activity in edible films and coatings against pathogenic microorganisms is attained using EOs; however, the water solubility and thermal sensitivity of bioactive compounds in EOs limit their use in food products. Additionally, the fabrication of nanoemulsion has some beneficial properties of improved solubility and activity, as direct incorporation of EOs in developing active films and coating may have the shortcomings of insolubility and reduced activity. The food compatible sources can be used to develop nanosized solubilizates (nanosized benzoic acid and sorbic acids), which can be used to develop antimicrobial packaging [177]. In this regard, the edible films and coatings incorporated with nanoparticles such as TiO_2, AgNPs, ZnO, and others are extensively utilized for preserving fruits and vegetables, where the mechanism of action of antimicrobial agents includes electrostatic interaction (between free metal ions and cationic polymers) and charged cell membranes [178]. Similarly, the addition of nisin and CS in developing HPMC-based films is effective in inhibiting several food deteriorative microorganisms [179]. In this regard, a brief discussion has been made on the available antimicrobial edible food packaging with the aid of organic and inorganic nanostructured materials and nanoemulsion approaches.

12.7.1 Organic and Inorganic Nanomaterial-Based Antimicrobial Edible Food Packaging

The polymer nanocomposite incorporated with organic and inorganic fillers is used to develop antimicrobial edible films and coatings for highly perishable food products such as fruits and vegetables, meat and meat products, and fish products.

The nanocomposite based on tragacanth/HPMC/ beeswax incorporated with AgNPs can be used as an edible active packaging [180], where the nanocomposite films provide some strong antibacterial activity against some pathogenic bacteria. Additionally, the edible films based on pullulan films with AgNPs, ZnO nanoparticles, rosemary, and oregano help in inactivating several pathogens (*Listeria monocytogenes, S. aureus,* and others) during storage of turkey deli meat [181]. The application of AgNPs on chicken sausages as edible coating has an ability to inhibit lactic acid bacteria for 30 days, which helps in increased shelf life [182]. The use of AgNPs can provide a broad antimicrobial spectrum against fungi, certain viruses, protozoa, gram-negative and gram-positive bacteria. Moreover, the coating on shiitake mushroom using alginate/nano-Ag can reduce the mesophilic, yeast, and mold counts [183]. The CS-based edible films loaded with ZnO nanoparticles also deliver an excellent antibacterial activity against *E. coli* [184]. The edible films based on sweet potato starch, lemon waste pectin, and nanotitania also help to improve the packaging attributes such as mechanical properties, barrier property, and thermal properties [185]. The antimicrobial property of nanomaterials is more efficient against microbial molecules and cells for having a higher surface-to-volume ratio in comparison with the higher scale materials [186]. The use of alginate, green tea extract, and grape seed extract to develop active edible films deliver antiviral activity against murine norovirus and hepatitis A virus [187]. Further, a research reports the development of food-grade edible films using *Salvia macrosiphon* seed mucilage and nanoclay targeting several kinds of food products [188]. Additionally, NCS-incorporated tara gum-based edible films also deliver antimicrobial activity, where NCS has better activity against *S. aureus* than *E. coli* [189]. However, a higher concentration of NCS may reduce the antimicrobial activity in nanocomposite-based films due to the agglomerating effect. However, bulk CS is more effective to provide antimicrobial activity in comparison with NCS-based nanocomposite films. Interestingly, the conjugated amino acids with nanopolymers can also be used as an active paper packaging materials, and an edible coating, where conjugated amino acids with nanobiodegradable polymers provide antibacterial (in a broad-spectrum) and antifungal activity [190].

12.7.2 Nanoemulsion-Based Antimicrobial Active Edible Food Packaging

Nanoemulsion-based edible coating on solid food products can act as a delivery agent for antimicrobials to improve the shelf life of food products [191]. The edible films based on basil seed gum incorporated with *Zataria multiflora* EO-based nanoemulsion have antimicrobial activity against potential foodborne pathogens [192]. Interestingly, the improved antibacterial activity of the nanoemulsion material (basil seed gum incorporated with *Z. multiflora* EO) can be obtained by reducing the particle size. In this way, the nanoemulsion-based films help in increasing the shelf life of food products by delaying the release of volatile components. Additionally, the shelf life of sliced bread can be improved by using the edible films based on methylcellulose and nanoemulsions (developed via ultrasonication) of clove bud and oregano EOs and further, the essential oils help in reducing the yeast and mold counts in sliced bread during 15 days of storage [193]. On the other hand, active packaging can also be formulated using the nanoemulsions (oil-in-water emulsion formed by mixing for 5 h at 1300 rpm) of soy protein (dispersed phase), carvacrol, and cinnamaldehyde. [194]. The edible coating containing aloe vera and eugenol-based nanoemulsion helps in maintaining the properties of shrimp [195]. The nanoemulsified edible coatings (citral-based coating) on fresh-cut melons can provide antimicrobial protection and reduce the microbial count up to 5 log, which can also enhance the shelf life till 13 days [196]. The several lipid-based nanodelivery systems such as nanoliposomes, nanoemulsions, solid–lipid nanoparticles, and nanostructured lipid carriers are extensively used for the delivery of several antimicrobial agents to control or inhibit the foodborne bacteria [197]. In this regard, the shelf life of Beluga sturgeon fillets can be improved with the aid of jujube gum and nettle oil-loaded nanoemulsion-based edible coating, where the used coating materials are effective against foodborne bacteria [198]. The fabrication of edible nanocoating on strawberry using limonene liposomes increases the shelf life for more than 2 weeks [199]. Besides, the edible coatings based on lemongrass EO-based nanoemulsion provide very effective antimicrobial activity (against *E Coli* and psychrophilic bacteria) and can enhance the shelf life of fresh-cut apples up to two weeks [200]. Moreover, the edible films based on EO-loaded nanoemulsion and sodium alginate can provide tailored physicochemical and antimicrobial property [201]. The nanoemulsion based on sodium alginate (continuous phase), Tween 80, glycerol, and EOs (thyme, sage, and lemongrass) as dispersed phase can be fabricated following few processing steps such as formation of emulsion using Ultra-Turrax (processing condition: 17,500 rpm, 2 min), and the formed emulsion can be processed to develop nanoemulsion under microfluidization (using microfluidizer, processing steps: 150 MPa, 3 cycles). Interestingly, the edible films based on thyme EO-based nanoemulsion can deliver good antimicrobial activity in reducing 4.71 log reduction of *E. coli* after 12 h (Table 12.2).

12.8 Safety Evaluation in Nanotechnology-Based Antimicrobial Edible Packaging

The AP has the primary goal of assuring the safety of food products. Additionally, in nanotechnology-based antimicrobial packaging, the safety concern and risk assessment are crucial concerns for wide consumer acceptance. The available international regulations are applied to produce safe packaging materials, which are accepted by the consumers and industry. In this regard, the risk evaluation of nanotechnology-aided edible food packaging is a critical challenge for food safety

Table 12.2 Application of nanotechnology-aided antimicrobial active edible food packaging

Sl. no.	Composition of packaging	Type of edible packaging	Antimicrobial property	References
1	Sodium alginate (continuous phase) EOs: thyme, lemongrass, sage oil (dispersed phase)	Nanoemulsion-based edible films	• Thyme EO-based edible films have the strongest antimicrobial activity against *Escherichia coli* with 4.71 Log reduction after 12 h	[201]
2	Tara gum (matrix materials) CS and CSNP (filler materials)	Antimicrobial edible film	• CSNP-reinforced films are more effective against *Staphylococcus aureus* than *Escherichia coli* • Antimicrobial activity of NCS-incorporated films has reduced antimicrobial activity at high concentrations	[189]
3	Basil seed gum *Zataria multiflora* EO	Nanoemulsion-based edible films	• Antibacterial activity of nanoemulsion can be increased by decreasing the particle size • The nanoemulsion delivers antimicrobial activity against gram-positive and gram-negative bacteria • The films can be used against foodborne pathogens	[192]
4	Methylcellulose Clove bud Oregano EO	Nanoemulsion-based edible films	• Use of EOs reduces the yeast and mold counts • The reduced droplet size delivers increased antimicrobial property • Increased shelf life of sliced bread	[193]

(continued)

Table 12.2 (continued)

Sl. no.	Composition of packaging	Type of edible packaging	Antimicrobial property	References
5	Pullulan AgNP ZnO nanoparticle Oregano oil Rosemary oil	Antimicrobial edible films for Turkey deli meat	• Good in the inactivation of meat spoiling pathogens such as *L. monocytogenes* and *S. aureus* • Addition of AgNPs and oregano essential oils is more effective against some pathogens as compared to ZnO nanoparticles and rosemary oils	[181]
6	CS ZnO nanoparticle	Active edible films	• Good antibacterial activity against *Escherichia coli*	[184]
7	Ginger EO-loaded nanoemulsion	Edible coating on chicken breast fillets	• Increased antimicrobial activity with increasing ginger EO concentration • 6% of Ginger EO-based nanoemulsion provides decreased total aerobic psychrophilic bacteria in chicken breast fillets (refrigerated storage) for 12 days storage	[202]
8	Thymol nanoemulsion, quinoa protein, CS	Antimicrobial edible films	• Coated strawberry has reduced yeast and fungal load under commercial storage environment • Provide enhanced shelf life of strawberry	[203]
9	Cellulose acetate ZnO nanoparticle	Edible films	• Strong antibacterial activity against *E. coli* (model bacteria)	[204]

(continued)

Table 12.2 (continued)

Sl. no.	Composition of packaging	Type of edible packaging	Antimicrobial property	References
10	Tragacanth, HPMC, beeswax AgNP (filler materials)	Edible films (food active packaging)	• Have strong antibacterial activity • Can be used as food active packaging	[180]
11	CS Lysozyme Nano-Ag	Edible protective hydrosols	• Applied on the surface of meat can inhibit several microbes with improved properties • Improved antioxidant activity	[205]

EO Essential Oil; *CS* Chitosan; *CSNP* Chitosan Nanoparticle; *NCS* Nanochitosan; *HPMC* Hydroxypropyl Methylcellulose

and analysis. However, the food safety analysis is done against pesticides, additives, drugs, foodborne pathogens, heavy metal reduction, allergens, and further inhibits the formation of biofilm. The use of nanostructured materials such as inorganic nanomaterials for developing antimicrobial edible packaging has received a wide interest due to enhance the shelf life of food products specially highly perishable food products. The several inorganic nanomaterials should be ingested and incorporated within the permissible limits, otherwise may create adverse health issues. The adverse impact of Zn nanoparticle includes damage to a biological system such as lipid peroxidation, protein impairments, organelle inflammation, DNA damage, and others. Additionally, the several health issues such as cytotoxicity, neurotoxicity, and genotoxicity may be caused by the use of several antimicrobial agents which depend on chemical composition, surface chemistry, surface energy, etc. In case of biodegradable food packaging, migration between food contact materials is a crucial problem, which may create serious health effects if the migration of components exceeds the limits. The migration in food packaging is mainly measured in terms of overall migration limit and specific migration limits, which has been discussed in the later chapters. Besides, the various quality management and food safety tools include good hygienic practices (GHP), good manufacturing practices (GMP), hazard analysis and critical control point (HACCP), Sanitation Standard Operating Procedures (SSOP), and others.

12.9 Conclusions

The chapter focuses to discuss the nanotechnology-aided edible food packaging facilitated by antimicrobial agents. The antimicrobial agents-aided edible food packaging has attracted a great deal of interest due to its potential in enhancing the shelf life of food products and further reduced the risk of pathogens in stored food products. The AP can also be considered as a novel food preservation technique which has an ability to combat the various existing preservation approaches. Several polymeric nanocomposites and nanoemulsion-based edible food packaging based on EOs and biopolymers are widely used in developing edible antimicrobial packaging.

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Chapter 13
Advanced Packaging Technology for Improved Delivery of Edible Packaged Products

Tabli Ghosh and Vimal Katiyar

13.1 Introduction

The consumer need for convenient and ready to eat healthy food products has raised the fabrication of novel packaging technologies such as modified atmospheric packaging (MAP), controlled atmospheric packaging (CAP), active, and intelligent packaging. The advanced packaging technology helps in enhanced product life, quality and further regulate the freshness of food products via reduced respiration process, and moisture infusion, ethylene scavengers, and others. The aspects of advanced packaging technologies are commonly associated with existing technologies, food attributes, globalization of packaging markets, and modern lifestyles, environmental and legal aspects (bioresourced, biodegradable, non-toxic, recyclable), consumers need and society. The global market of food packaging technology includes edible packaging, micro packaging, smart, antimicrobial, water soluble, self-cooling and self-heating packaging system, etc. In 2020, the trends in food packaging includes packaging with technology-enabled solutions, sustainability, increased portability, transparent and clear labelling and minimal design. Among available, the focused food packaging technology are active, smart, ascetic, biodegradable, MAP, controlled atmospheric storage (CAS), and others. The design and packaging technology are used based on the targeted food products such as dairy & dairy products, bakery products, fruits and vegetables, meat and meat products, convenience food, etc. Besides, the innovation in packaging approaches include functional barrier, high chemical barrier material innovations, intelligent supply chain, modified atmosphere, and others. In this regards, the various packaging equipment are form-fill-seal, labelling & coding, wrapping & bundling, cartooning, and others. In addition to the food packaging, the other packaging to

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move materials include assembly, automation, transportation, warehousing, order picking, etc. However, the structural design software for packaging are ArtiosCAD (good for cardboard and corrugated packaging), Impact by Arden software, Kasemake (produced by a British software house), EngView Packaging Designer Suite, Packmage, and others. The packaging designs and applied packaging technology are selected based on food products, property, consumer demands and others, where several strategies are applied to further improve the product life. Based on this discussion, the current chapter focuses to discuss the advanced packaging technologies such as modified atmospheric packaging, controlled atmospheric packaging, active packaging, and intelligent packaging.

13.2 Application of Packaging Technology for Improved Product Life

As mentioned earlier, the inclusion of packaging serves food protection in terms of physical, barrier, mechanical actions, and others for transporting food products to the targeted consumers. Among the available packaging materials, the sustainable packaging materials are replacing the available conventional packaging materials to be used as secondary packaging materials for several food products. The modification in properties of sustainable packaging is attained with the aid of nanomaterials as reinforcing agents to obtain tailored-made property. The use of nanotechnology in secondary packaging materials has received a great interest to provide active (such as use of nano-antimicrobial agents) and smart packaging (such as use of nanosensors, nanomaterials). Further, the Global market is continuously intended to improve the quality of food products via modifying the packaging based storage. In this regards, the different types of packaging include anti-corrosive, pharma, plastics, and flexible packaging, etc. Moreover, from very early days, the various packaging technologies (as shown in Fig. 13.1) such as MAP, controlled atmospheric storage (CAS), active packaging, smart packaging, etc. are used for maintaining the quality of food products. The application of various packaging technologies helps to extend the shelf life of commodities for maintaining modified or controlled atmospheres for food products. Considerably, the packaging technology can be applied to primary (edible coated food products) and

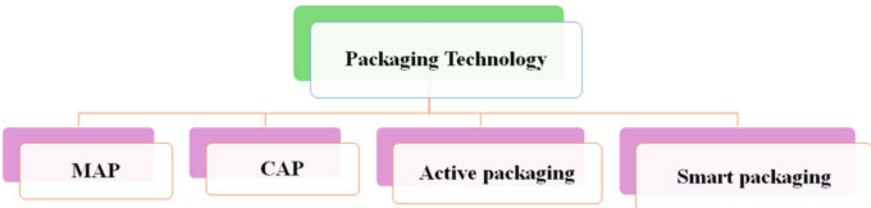

Fig. 13.1 Various packaging technologies for extended shelf life of food products (*Note* MAP: Modified atmospheric packaging; CAP: Controlled atmospheric packaging)

secondary packaging materials for transporting edible coated food products. However, the fundamentals of available advanced packaging technologies such as MAP, CAS, active and smart packaging and related strategies to extend the shelf life of food products have been detailed in the below section.

13.3 Modified Atmospheric Packaging

The MAP technology is extensively applied to prolong the food storage life via passive and active MAP [1]. MAP based packaging technology generally involves the alternation of gaseous atmosphere (gaseous concentration of O_2 and CO_2) inside the package. In the early of twelfth century, the transportation of lamb meat from Australia to England was done with the aid of MAP based packaging [2]. However, the first report on MAP was found in the year 1927 (with reduced O_2 amount and increased CO_2 amount), where MAP was used to enhance the shelf life of apples. As shown in Fig. 13.2, the various extended application of MAP to enhance the product shelf life are passive MAP, active MAP, high-oxygen MAP, controlled MAP, and intelligent MAP, which are proposed to extend the shelf life of various commodities. In passive MAP, there involve the natural interplay of gaseous concentrations inside the package, which held due to the respiration of food products and permeability of packaging films. In active MAP, the gaseous environments are actively modified with the aid of gas flushing systems or gas barrier systems to modify the packaging environments. Considerably, gas scavengers are also used to modify the gaseous environment of the package actively. The active MAP is more preferable than the passive MAP to provide modified atmosphere for food products. The high oxygen MAP can support the freshness of fruits and

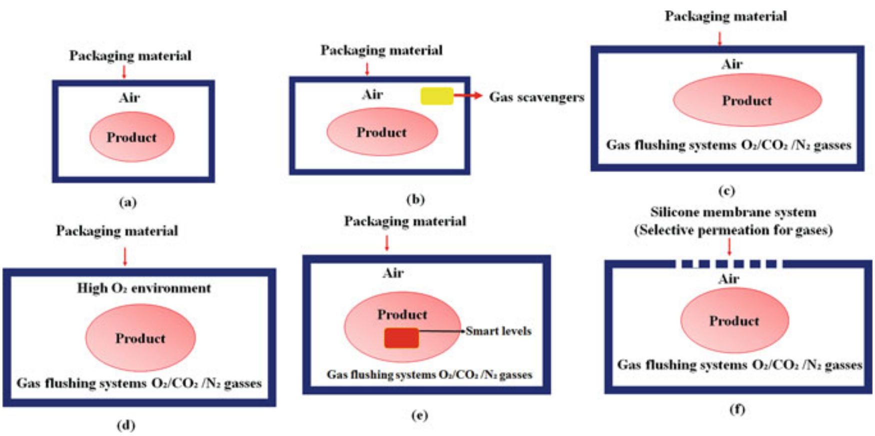

Fig. 13.2 **a** Passive MAP; **b** Active MAP with gas scavengers; **c** Active MAP with gas flushing systems; **d** High oxygen MAP; **e** Intelligent MAP; and **f** Controlled MAP

vegetables, and can inhibit the growth of anaerobic reactions, and certain bacteria. However, the MAP conditions should avoid high oxygen and carbon dioxide conditions for successful packaging of food products with maintained product quality. Considerably, the designing of MAP for food products need the mass transport properties of polymeric films at several temperature ranges, and respiration rate properties of food products (oxygen consumption rate and carbon dioxide evolution rate), where the mathematical modelling is very useful in selecting particular packaging films for MAP of food products [3, 4]. On the other hand, controlled MAP involves the controlling of the environment of packaging system with the aid of gas scavengers and others. Further, in intelligent MAP, indicators or smart labels are involved to detect the inner condition of the packaging. Thus, the strategies in storage of food products are applied to extend the product life, where the specific technique holds many benefits and limitations over other existing techniques. The significance of using MAP includes reduced economic loss, easy transportation of food products over longer distances, potential increase in shelf life, improved quality, etc. [5]. Besides, the limitations of using MAP are the increased cost of maintaining the systems, continuous temperature control, require different gaseous compositions for different produces, require special equipment for successful MAP of products, etc. Further, the limitations of MAP for available produces include inappropriate selection of packaging films, cost of MAP technology, maintaining package integrity during storage and transportation, and others.

13.3.1 Strategies in MAP for Improved Product Life

The gaseous conditions surrounding the package is modified or controlled using O_2, CO_2 and N_2 gasses, whereas, other gaseous components such as nitrous oxides, nitric oxides, ethylene, chlorine and others are also used for altering the gaseous compositions. The various available polymers, film types for MAP includes (i) Films such as ethylene-vinyl alcohol (EVOH), polyvinylidene chloride (PVDC), PA, microporous, microperforated, ethylene vinyl acetate (EVA), polyurethane (PU), polyethylene (PE), etc. (ii) edible films: pectin, wheat gluten, CS, zein, sodium caseinate, methylcellulose-palmitic acid, etc.; (iii) smart films: oxygen scavengers with oxygen indicators, antibody based detection systems; and (iv) antimicrobial films [6]. Additionally, some of the packaging films for MAP are thermally stable at lower temperatures (which depends on the various packaging components), where the use of this kind of packaging material may create anaerobic conditions inside the package at higher temperature which are undesirable conditions of food products. Additionally, the uncontrollable gaseous conditions may result in increasing CO_2 level inside the package which may further result in mold growth inside the package. The widely used packaging films for MAP includes low density polyethylene (LDPE), PVC, non-perforated PVC, biorientated PP-1 (BOPP-1), BOPP-2, BOPP-3, laminated PE, polypropylene (PP), unperforated PP, oriented PP, semi-permeable plastic bags, PP, perforated PE bags, etc. [7].

13.3.2 MAP for Perishable and Other Food Products

The MAP based technology has received a great interest in improving the handling chain of fresh and fresh-cut foods [2], lightly processed fruits and vegetables [8]; meat and poultry products [9, 10], value added products (broccoli florets, asparagus tips) [11], and others. The various reports available for food products are bhimkol [3], fig fruit [4], cherry, raspberry, strawberry [11], sardines [12]; blueberry [13]; pomegranate [14]; shredded lettuce [15]; chicken breast meat [16]; ostrich meat [17]; fresh-cut apple, fresh-cut pear, sliced carrot, blueberry, raspberry, papaya, fresh-cut melon, fresh-cut jackfruit [2], steaks from large beef cuts [18]; fish and fishery products [19]; etc., where inclusion of MAP helps in improving the product quality. The applied MAP technique for several vegetables products include broccoli, green asparagus, sea asparagus, celery sticks, fresh-cut peppers, summer truffles, white mushrooms, etc. [2]. The MAP based technology is extensively used for agricultural products specifically for produces which are having several problems such as high respiration rate (application of MAP can control the respiration rate), browning of cut fruit products, increased ethylene biosynthesis, microbial attacks, products which are prone to water loss, water condensation, etc. [11]. The consumer demands of lightly processed food products including shredded cabbage, cut carrots, cut lettuce are increasing, which can be packaged in MAP and under certain levels of relative humidity for improved storage life [8]. Thus, there is a need to predict the film permeability of selected packaging film for lightly processed food produces as the modified atmosphere inside the package depends on the respiratory behavior of food produces, gas exchange property, and permeability of film materials, etc. In this regards, the permeability of packaging materials should allow gaseous exchange to maintain the gaseous concentration of O_2 and CO_2 gas, which further depends on the temperature and storage time. The reduced level of O_2 helps in decreasing respiration rate, reduced oxidative reactions, reduced fermentative metabolism, reduced browning effect, reduced ethylene production rate, etc. Further, the lightly processed food products should undergo reduced weight loss under MAP, where the weight loss is a critical parameter in case of lightly processed food products. The MAP should contain CO_2 levels below injurious levels and the CO_2 evolution rate depends on the partial pressure of O_2 and storage temperature, where the decreased in CO_2 evolution rate obtained with decreasing storage temperature. The application of MAP has an ability in providing low oxygen storage and high carbon dioxide storage throughout the storage life [13].

Further, the application of active and modified packaging for meat storage can maintain product quality, where the active components of oregano essential oils affect metabolic activity and growth of microbial associations [9]. The addition of essential oils act as a preservative in meat products with enhanced shelf life via delaying the microbial spoilage, inhibition of microbial metabolites production, etc. The essential oils have many beneficial properties such as antioxidant, antibacterial, antifungal, and others which make it a good preservative for extending meat storage life [9, 20]. Other than essential oils, CS is also used as an antimicrobial agent to improve the product life of meat products. In this regards, the meat products may simply dipped in the solutions of respective antimicrobial agents for obtaining improved product life before MAP. The MAP of ostrich meat using various gaseous concentrations of O_2, CO_2 and N_2 can affect the physicochemical, microbiological, sensorial quality during the storage condition (4 °C and 10 days), where the microbial growth can be delayed using high CO_2 usage and further high O_2 may convert myoglobin to oxymyoglobin, which develops undesirable flavours [17]. Further, the several considerations for meat products in MAP based storage include blooming and residual O_2, gas mixtures, oxidative stability, headspace requirements, tray options, merchandising, productivity, etc. The attributes of meat products in packaging includes pigments color, metmyoglobin reduction, flavor attributes, etc. Among available packaging technologies, the shelf life of meat products can be improved with the aid of MAP, where various categories of MAP provide various properties of meat products [1]. The MAP of meat products are effected by the presence of oxygen in the gaseous environment of the packaging, where MAP of meat with oxygen has an ability to promote the oxidation of lipids and pigments. The meat products include various levels of degradation such as off-flavor, off-odor, discoloration, textural changes, nutrient degradation (myoglobin degradation, oxidation of various components), etc. The quality of meat products depends on the meat texture, appearance, nutritional components, color, flavor, etc. The various factors such as product type, headspace, package type, storage temperature, storage period, etc. have an influential effect on MAP of meat products. The suitable plastic film attributes for meat packaging include gaseous barrier properties, strength of packaging material, optical properties, etc. The several packaging options for meat packaging include high oxygen MAP, low oxygen vacuum packaging, low oxygen MAP, air permeable packaging, etc. The conventional packaging materials used for packaging meat and poultry products include PVC, PVDC, PP, High density polyethylene (HDPE), LDPE, linear low density polyethylene (LLDPE), EVA, EVOH, PA, PET, polystyrene (PS), etc.

The combined effect of CS or essential oils with the aid of MAP help in improving the quality of meat products through various ways such as lipid oxidation, inhibit growth of microbial spoilage, improve textural properties, etc. [16]. Further, the application of CS, essential oils and MAP help in improving the shelf life of chicken breast meat by 14 days. The oxygen MAP based storage for meat products provide a negative impact on myoglobin stability, promote lipid oxidation, other sensory properties, etc. However, high oxygen concentration can retain bright red color of meat products, which is accepted by the consumers [18, 21]. Further, a minimum of 55% oxygen content is suggested for MAP of meat products to prolong color stability, where 70–80% oxygen is widely used for MAP of meat products. Considerably, the carbon dioxide content of 20–30% is favored for inhibiting bacterial growth. In this regards, the inclusion of antioxidants, and low oxygen storage are recommended for beef storage to reduce oxidation induced degradation. However, the MAP of meat products require a maintained gas and moisture barrier to provide enhanced shelf life of meat products. The application of MAP and oregano essential oils (0.1%) can improve the shelf life of fresh chicken meat (5–6 days) in comparison to individual oregano essential oil and MAP packaging [20]. The meat spoilage during MAP based storage may be spoiled due to *Enterobacteriaceae* which can produce foul-smelling diamines and sulfuric compounds [10]. This kind of bacteria such as *Hafnia alvei, Pantoea agglomerans*, *Serratia liquefaciens*, may develop objectionable odors in meat products. The meat products such as fresh pork, beef muscle, ground beef, buffalo meat, chicken breast, ground ostrich meat, ostrich steaks, sheep meats, lamb meat are also packed using MAP [2]. The factors which are taken into considerations during meat product storage are surface color, lipid oxidation, pH of meat, microbiological analysis (mesophiles and psychrotropes), sensory analysis (appearance, flavor, juiciness, tenderness, oxidation flavor, overall acceptability, etc.), etc. [22].

Additionally, fish is generally recognized as highly perishable food items, and the shelf life is very less which can be enhanced with the aid of MAP, and vacuum-packaging under refrigeration storage. MAP is also considered as a kind of vacuum packaging, which includes the removal of air inside the packaging materials, or includes the replacements of gaseous environment (air content) inside the packaging by flushing or vacuum. The application of packaging technology can maintain organoleptic property, reduce microbial attack, improve texture, etc. The fish quality is generally determined by trimethylamine (TMA) content, total volatile bases (TVB), nucleotide ratios, etc. [12]. Further, high histamine content of fish can cause allergic reaction in human body. The application of MAP and vacuum packaging can provide reduced formation of histamine, TMA, and TVB and others which can enhance the shelf life of fish products. The aquatic products including salmon fillets, sea bream, Chinese shrimp, fresh sea bass fillets, atlantic salmon, tuna, rainbow trout fillets, etc. are also can be packaged with the aid of MAP [2].

13.3.3 Gas Exchange Mechanism in MAP

In MAP based packaging, the equilibrium gaseous concentration in terms of O_2 and CO_2 should attain within shortest period of time, which help in obtaining maximum shelf life of food products. The generalized mechanism for MAP of fresh produces are represented in Fig. 13.3. In this regards, the generalized Eq. (13.1) illustrates the rate of oxygen accumulation inside the headspace which is obtained by the difference between O_2 entry rate into the package space and the oxygen consumption rate by the packed food products. Similarly, the CO_2 diffusion rate from the internal package space to the external package space is represented in the Eq. (13.2), where the CO_2 accumulation rate in package headspace is given by the difference in the CO_2 evolution rate of the fruit products and CO_2 permeate rate from the internal space to external environment.

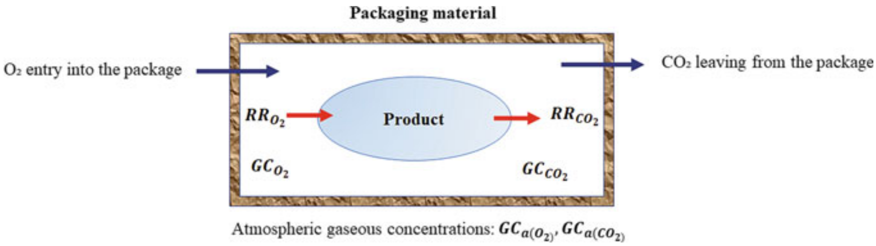

Fig. 13.3 Gas exchange mechanisms in MAP of fresh produces at storage

$$A_{\mathrm{pf}} K_{p,\mathrm{O}_2}\left(\mathrm{GC}_{a(\mathrm{O}_2)} - \mathrm{GC}_{\mathrm{O}_2}\right) - W_p * \mathrm{RR}_{\mathrm{O}_2} = V_e\left[\frac{d\mathrm{GC}_{\mathrm{O}_2}}{\mathrm{dt}}\right] \quad (13.1)$$

$$W_p * \mathrm{RR}_{\mathrm{CO}_2} - A_{\mathrm{pf}} K_{p,\mathrm{CO}_2}\left(\mathrm{GC}_{\mathrm{CO}_2} - \mathrm{GC}_{a(\mathrm{CO}_2)}\right) = V_e\left[\frac{d\mathrm{GC}_{\mathrm{CO}_2}}{\mathrm{dt}}\right] \quad (13.2)$$

where, $\mathrm{GC}_{\mathrm{O}_2}$ and $\mathrm{GC}_{\mathrm{CO}_2}$ are O_2 and CO_2 concentrations inside the packaging materials, respectively; $\mathrm{GC}_{a(\mathrm{O}_2)}$ and $\mathrm{GC}_{a(\mathrm{CO}_2)}$ are atmospheric O_2 and CO_2 concentrations, respectively; A_{pf} is the area of the packaging materials through which diffusion of gases held; K_{p,O_2} and K_{p,CO_2} are the permeability of O_2 and CO_2 gases through packaging films, respectively; W_p is the weight of the stored food products; V_e is the headspace volume (free space volume) inside the package; $\mathrm{RR}_{\mathrm{O}_2}$ and $\mathrm{RR}_{\mathrm{CO}_2}$ are the respiration rates of fruit materials in terms of O_2 and CO_2 gases, respectively; and dt is the difference in storage time between two successive respiration data.

13.3.4 Effectivity of Several Factors in MAP

The MAP based system should be developed which are consumer friendly, and has an ability to provide good quality products, and economic in nature. The packaging materials should maintain the favorable gaseous composition at various temperature ranges, which may be better obtained via active MAP. The film type such as permeability and physiology of fruit (respiration of fruit products) affect the steady state oxygen and carbon dioxide levels. The cost of MAP technology is also a limitation in using the MAP based techniques, where the cost of packaging films and creating a modified gaseous conditions inside the package are the other limitations.

13.4 Controlled Atmospheric Packaging: A Packaging Strategy for Food Products

The CAP includes the controlling and monitoring the environment surrounding the food packages to provide a suitable environment in terms of gaseous environment. As shown in Fig. 13.4, the available systems for controlled atmosphere includes (i) Oxygen control systems: It includes the use of external gas generator, gas separator systems, liquid nitrogen atmospheric generators, hypobaric storage and others; (ii) Carbon dioxide control systems: This includes the scrubbing action which helps in carbon dioxide removal such as water, caustic soda, activated charcoal, hydrated lime, and molecular sieves; (iii) Ethylene control systems: This may involve the removal of ethylene, where ethylene get oxidized to obtain water vapor and carbon dioxide [6]. CAP is generally used for controlling the ripening of food products and slow spoilage of fruits and vegetables. The several strategies for CAS of food products include various routes such as (i) CO_2 scrubbing technique to control the CO_2 concentration inside packaging, where the evolved CO_2 due to the respiration of fruit products should be removed to prevent CO_2 related injury for sensitive fruits and vegetables; (ii) Water scrubbing technique to remove water from packaging system; (iii) Nitrogen purging system is used to alter the gaseous composition in terms of oxygen; (iv) Atmosphere control innovations; (v) 1-Methylcyclopropene as an inhibitor of ethylene action: Ethylene increases the rate of ripening in fruit products, etc. [23]. In this regards, some of the available CAS based storage system has been discussed here. The survival of *Salmonella Enteritidis* on cherry tomatoes can be inhibited or reduced via adopting the packaging techniques such as MAP, CAS and air storage with or without gaseous ozone treatment [24]. The specific microorganism can survive during MAP and CAS storage of tomatoes, which greatly dependent on the storage temperature, load of contamination and others. The application of ozone treatment can offer a positive impact in inhibiting the growth of *S. enteritidis*, however, the surface color, organoleptic loss and nutritional quality, may be reduced and degraded due to ozone treatment. Further, the combined storage of food products at low temperature and CAS provide various benefits such as reduced rate of respiration of commodities, reduced rate of ethylene production rate, reduced nutritional compositions with ripening stages, etc. [25]. The CAS of snow pea pods provide more benefits than MAP and air storage (storage temperature for all the condition 5 °C). Further, a research reported the keeping quality of chilled pork when packed in CAP and

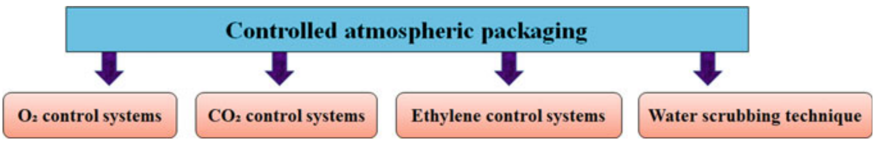

Fig. 13.4 Approaches for controlled atmospheric packaging system

vacuum conditions, where carbon dioxide and vacuum packaged pork loin (chilled storage at -1.5 °C) has a shelf life of 15 and 12 weeks, respectively [26]. The storage life of chilled pork about 9 weeks due to maximum growth of lactic acid bacteria and off-flavor development. The CO_2 storage helps in inhibiting the growth of anaerobic bacteria and is known to provide the inhibiting action against microbial growth, further the development of bacteria which may grow under anoxic conditions can also be retarded. In this regards, the vacuum and CO_2 CAP are preferable for lamb and beef products for improved shelf life. As discussed earlier, the high oxygen storage is preferable for color stability of red meat, thus, besides, microbial counts and shelf life, consumer acceptance is also a factor while deciding the storage conditions of meat products. However, the rapid CO_2 evolution in cooked beef product may cause pores in the product when packed in CO_2 based CAP [27]. The sodium carbonate-soluble pectin of peach fruit can undergo structural changes (can be visualized by atomic force microscopy (AFM)), which can be inhibited with the aid of CAP with lower concentration of oxygen and higher concentration of carbon dioxide [28]. In 2007, Morales et al., has studied the effectiveness of CAS (storage conditions: (i) 2.5% O_2 and 3.9% CO_2; and (ii) 1.5% O_2 and 2.5% CO_2 at 1 °C) and fungicide treatments on the accumulation of patulin in apples storage, where the production of patulin can be monitored at various O_2 and CO_2 concentrations [29]. Further, during CAP of sliced roast beef, the growth of *Listeria monocytogenes*, *Aeromonas hydrophila*, *Yersinia enterocolitica* can be monitored to extend the shelf life [30]. The CAP is very useful in monitoring the quality of food products via optimizing the processing conditions of the storage for targeted food products.

13.5 Active Packaging and Its Related Aspects

The active packaging is facilitated with the incorporation of certain active compounds into the packaging systems, which helps in maintaining the product quality against undesirable product conditions for prolong shelf life. According to the European Union Guidance to the Commission Regulation, the active packaging further can be defined as the type of food packaging having an additional function besides offering the protective barrier against the external environment [31]. This kind of packaging includes the use of additives which are developed from the natural resources and can absorb the food produced chemicals or can release active components into the food to provide safeguard against food degrading factors. The extensively used active components in active food packaging include antioxidants, essential oils, chitosan and nanochitosan (a category of biopolymers), flavorings, etc. In this regards, the available strategies to develop active packaging are displayed in Fig. 13.5, which includes the use of antimicrobial agents, addition of sachets or pads (having active response towards undesirable conditions), use of antimicrobial polymers, use of antioxidant agents, etc. The active packaging is performed using oxygen scavengers, carbon dioxide absorbers/emitters and

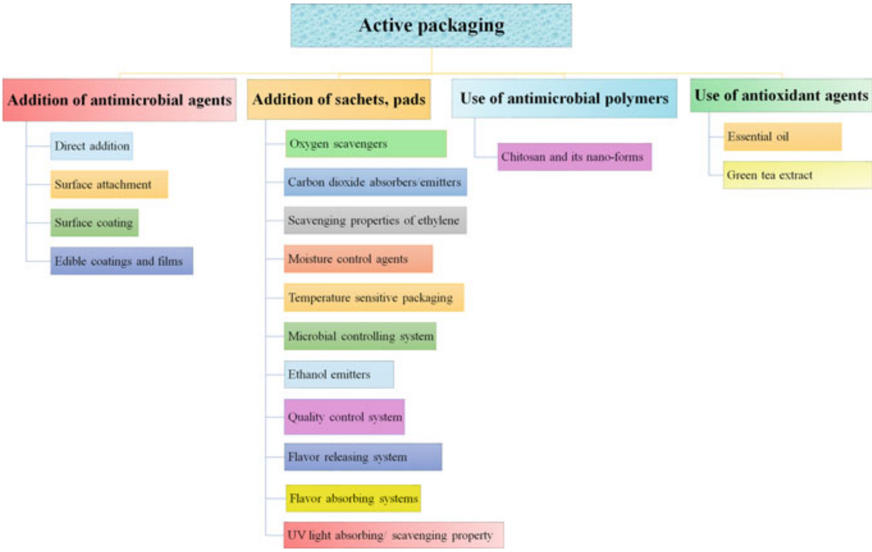

Fig. 13.5 Strategies to develop active packaging

scavengers, ethylene scavengers, moisture controlling agents, temperature controlling system (temperature sensitive packaging), microbial controlling system using moisture absorbers, ethanol emitters, quality controlling system, flavor releasing system, flavor absorbing systems, ultraviolet (UV) light absorbing/ scavenging property, etc. [32, 33]. The antioxidants are one of the widely used components in edible and non-edible food packaging to prevent degradation of various kinds of food ingredients such as prevents lipid oxidation, and considerably, has an ability in controlling the oxidation of myoglobin. In this regard, several synthetic and natural antioxidants are utilized to develop active packaging systems. The development of active packaging includes the use of freshness enhancers or active additives with the packaging materials which provides a preservative action to the food products [34]. The application of antioxidants such as oregano essential oils and green tea extracts for developing active packaging for foal meat can enhance the shelf life of foal meat under refrigeration storage [35]. The application of antioxidants can act against microbial spoilage, protein oxidation, lipid oxidation and further, helps in retaining the product color and textural properties, etc.

13.5.1 *Strategies in Active Packaging*

The oxygen scavengers or oxygen absorbing systems are used as a replacement to the gas flushing system to extend the shelf life of commodities. The mode of action of oxygen absorbing active packaging system can be obtained via enzymatic systems (such as alcohol oxidase-ethanol vapor) and chemical systems (ascorbic acid oxidation, iron–sulfur, ferrous carbonate, catechol, etc.) [32]. As discussed earlier in this section, the high oxygen concentration may increase oxidative degradation, microbial growth, color changes, and nutritive degradation, so the use of oxygen absorbing agents in active packaging can maintain the quality of the food products. The oxygen scavengers are generally based on using ascorbic acid oxidation, iron powder oxidation, enzymatic oxidation, photosensitive dye oxidation, etc. The various forms of oxygen scavengers that are widely used in food packaging include closure liner, sachet, film, card, label, etc. The available oxygen scavengers are FreshPax® (Multisorb Technologies Inc., USA), Ageless® (Mitsubishi Gas Chemical Co., Japan), ATCO® (Emco Packaging Systems, UK, Standa Industrie, France), Cryovac® OS2000™ (Cryovac Division, Sealed Air Corporation, USA), etc.

Several gaseous composition and moisture retaining system in Active Packaging. The commercially available O_2 scavengers for active packaging are (i) ATOX, Artibal SA, Spain (type: film coated with antioxidant); (ii) Shelfplus® O_2, Albis Plastic GmbH (Masterbatch); (iii) ATCO®, Laboratories STANDA (Label); (iv) OxyGuard®, Clariant Ltd., Switzerland, etc. The oxygen absorbing systems are required for cooked meat products, bakery products, grated cheese, fruit and vegetable juices, nuts and oils, fried snacks, etc. [36]. The commercially available CO_2 emitters for meat packaging systems are (i) Ageless G, Mitsubishi Gas Chemical, Japan; (ii) Freshpax, Multisorb Technologies, USA; (iii) Freshlock, Multisorb Technologies, USA; (iv) Verifraise package, SARL Codimer, France, etc. The ethylene scavengers used in active food packaging include Green Pack, which is a sachet of potassium permanganate in silica, where the component silica has a property of absorbing ethylene, whereas, the component permangananate oxidizes the absorbed ethylene to acetate and ethanol [37]. The moisture scavenging systems are required for mushrooms, fresh fish, strawberries, grains, maize, etc. [36]. The moisture control systems or moisture absorbers in packaging systems are required to reduce the water activity of packaged food products, which in turn reduces the microbial growth in food products. The packaging systems may have a problem with condensations due to continuous respiration of fruits and vegetables under close packaging systems. In this regards, the use of antifog additive may be beneficial to provide transparency in the packaging system. The available moisture control systems are Toppan™ (Japan), Peaksorb® (Australia), etc. The ethylene absorbing systems are required for climacteric fruits and vegetable products, which have a fast respiration process resulting in a fast degradation process [36].

Antimicrobial Agents in Active Packaging. As discussed in earlier chapter, the use of various antimicrobial agents for the development of active packaging has been discussed. The availability of active packaging with the aid of antimicrobial agents is available in the forms of edible and non-edible packaging materials. The classes of antimicrobial packaging are (1) use of sachets incorporated with antimicrobial substances, which can generate antimicrobials during undesirable conditions inside the packaging; (2) the antimicrobial agents can be directly incorporated within the packaging system; (3) edible coating of food products using antimicrobial agents; (4) Polymeric materials with antimicrobial activity. The natural antimicrobial agents that have attained a great interest include plant based extracts, essential oils, spice based extracts, and others. The commercially available antimicrobial active packaging systems are (i) Aglon^{TM}, Agion Technologies, USA (active compounds: silver zeolites); (ii) Backtiblock®, NanoBioMaters, Spain (active compounds: silver); (iii) Bioka, Bioka Ltd., Finland (Glucose oxidase); (iv) Biomaster®, Addmaster Ltd., UK (active compounds: silver); (v) Zeomic^{TM}, Sinanen Co., Ltd, Japan (Active compound: Silver); (vi) IonPure® Solid Spot LLC, USA (Active compounds: Silver), etc. [31]. The emitting sachets and absorbent pads are extensively used in antimicrobial food packaging systems, where some sachets generate and release antimicrobials and some carry and release antimicrobials [38].

Use of Active Components in Active Packaging. The lipid oxidation is a common problem in food products which creates unacceptable texture, off-flavors, off-odors, color changes, etc. [39]. The antioxidant based food packaging involves the use of various classes of antioxidants such as (i) Primary antioxidants (free-radical scavengers) include the use of various active agents such as (a) synthetic active agents such as butylated hydroxytoluene (BHT), butylated hydroxyanisole (BHA), propylene glycol, etc. (b) Natural agents such as Plant based extract, essential oils, flavonoids, etc. (ii) Secondary antioxidants include the use of chelators (lactoferrin, Ethylenediamine tetraacetic acid, citric acid, etc.), UV absorbers (benzophenones, benzotriazoles, phtalocyanine, TiO_2, etc.), oxygen scavengers (metal based powders, ascorbic acid, catechins, oxygen consuming spores, etc.), singlet oxygen quenchers (carotenoids, polyphenols, tocopherols, and others), etc. The other active components of active packaging of meat products include tea polyphenols, thymol, carvacrol, eugenol, rosemary, lactic acid, nisin, oregano extract, CS, etc. [40].

Additionally, the antioxidant packaging systems are available as sachets and labels, coating, multilayer active films, covalent immobilization, etc. The challenges and shortcomings of antioxidant based active packaging include antioxidant activity and stability, attributes of food products such as acidity, nutritional compositions, engineering properties of packaging materials, problems concerning migration and permissible limits of toxic components of packaging materials, safety and regulations of active packaging, etc. In this regards, the non-migratory active packaging is more preferable in comparison to migratory active packaging materials. Interestingly, there are several materials which are extensively utilized for the delivery of antimicrobial compounds such as synthetic polymers (petroleum based polymers such as LDPE, EVOH, etc.), bio-based polymers (polysaccharides, proteins, lipids, PLA, Polyhydroyalkanoates, etc.) and others [41]. The systems for antimicrobial migrating and non-migrating systems are flushing and gas/vapor emission, coating and films with antimicrobial agents, impregnating polymers with antimicrobial agents, electrospun nanofibers, etc. [41]. In this way, the active packaging can be used as primary and secondary packaging materials for food products.

13.6 Smart or Intelligent Packaging

The smart or intelligent packaging includes the sensing of the product properties or the package environment during storage and transportation, which helps in providing the information to consumers and producers about the product status without deteriorating the package. To obtain the status of product quality inside the package is a critical problem for every buyer, where the customer buys the products without knowing the product conditions, where the involvement of smart packaging may resolve this problem to some extent. The aim of smart food packaging includes the extension of product shelf life, monitoring freshness, and further the packaging systems display the information relating the quality, etc. Further, there are many opportunities for smart packaging including new business model, cybersecurity, real time capabilities, nanotechnology, etc., which make it a potential candidate for customer friendly packaging techniques. Interestingly, the Hazard Analysis and Critical Control Points (HACCP) and Quality Analysis and Critical Control Points (QACCP) systems can also be improved using intelligent packaging systems [42].

13.6.1 Strategies in Smart Packaging

This kind of packaging includes the use of indicators, sensors, security tags and radiofrequency identification tags (RFID), barcodes, etc. [43]. The various kinds of strategies used in smart packaging are represented in Fig. 13.6. The indicators include freshness indicators, time-temperature indicators (TTIs), integrity indicators, RFID, etc. The attributes which should be notified for the fabrication of smart indicators include easy to understand, clear colour changes, reasonable price, irreversible color change, and should have a relation with quality changes in products, etc. [44]. The TTIs are simple, economic and efficient to be used for fabricating intelligent packaging materials. The TTIs are widely used to study the storage life of perishable food products such as milk, perishable fruit products and others. The TTIs are generally categorized into three types such as critical temperature indicators (works above or below a reference temperature such as protein denaturation), critical temperature/time integrators, and time temperature integrators [45]. The widely used TTIs include MonitorMark™, Timestrip®, Fresh-Check®, Checkpoint® and others. A study reports the development of intelligent food packaging using anthocyanin (source: Red cabbage) aided active CS/Poly-vinyl alcohol (PVA) films as TTIs [46]. The anthocyanin is a naturally available pigments, which is extensively utilized in various chemical forms to develop TTIs. The various anthocyanin forms include cyanidin (Source: apple, pear, peach, cherry, red cabbage, blackberry, etc.), pelargonidin (Source: potato, strawberry, banana, etc.), cyanidin and delphinine (Source: purple carrot, green bean, pomegranate, eggplant, etc.), cyanidin and peonidin (Source: sweet cherry, purple sweet potato, plum, etc.), peonidin (Source: mango), petunidin and malvidin (Source: red grapes, bilberry, etc.) [43].

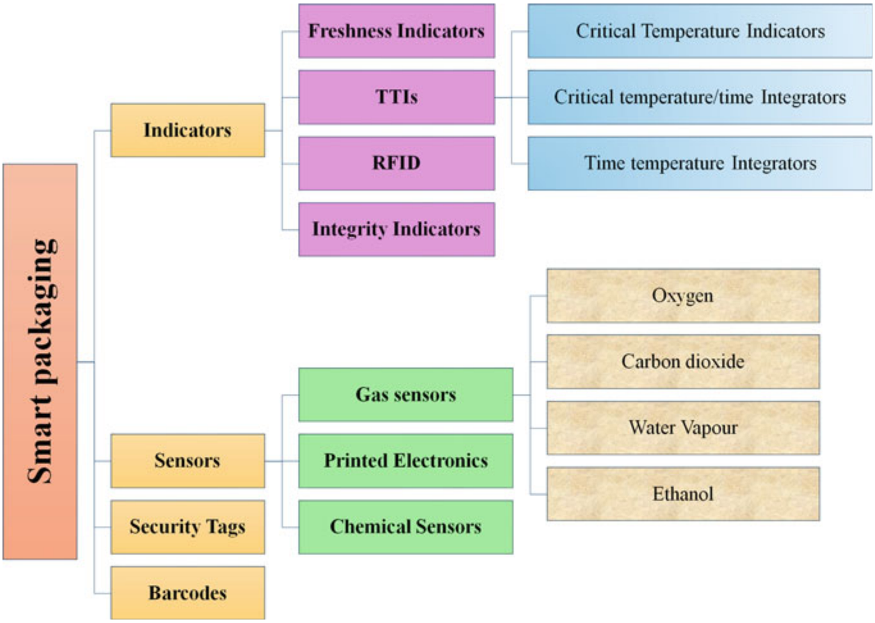

Fig. 13.6 Strategies to develop smart packaging materials (*Note* TTIs: Time-temperature Indicators; RFID: Radiofrequency identification tags)

Additionally, the different sensors for intelligent packaging include gas sensors (such as oxygen, carbon dioxide, water vapour, ethanol etc.), printed electronics, chemical sensors, and others. The smart packaging techniques utilize the sensor based approach for food products, pharmaceuticals, and other products [47]. A research reports the use of smart packaging for maintaining the quality of MAP packed bread, where intelligent oxygen sensors and active ethanol emitters are utilized [48]. The ethanol emitters in smart packaging are helpful for controlling mycological growth which in turn improves the shelf life. The CO_2 sensors are classified into two types such as optical sensors and electrochemical sensors, which are based on the type of transducer, where the electrochemical CO_2 sensors can be of various types such as amperometric, potentiometric, etc. Carbon dioxide sensors for smart packaging include conventional and innovative optical CO_2 sensors. The innovative optical CO_2 sensors are wet optical CO_2 indicators (pH based), principle in pH dye based indicators, fluorescent CO_2 indicators, dry optical CO_2 sensors, sol-gel based optical CO_2 sensor, photonic crystal sensors, color changing metals, etc. [49]. Further, the smart packaging materials are also applicable to check the storage study of edible coated food products and various strategies are applied to develop smart packaging materials for both edible coated and uncoated food products. Interestingly, several eatable materials are utilized to develop smart packaging materials, where, the consumer can eat the product with the food products by decreasing waste generations.

13.7 Conclusion

The advanced packaging technology are used to obtain high shelf life using high level of technical expertise. In the current trends of advanced packaging systems, the several packaging technologies include MAP, CAP, active and smart packaging. The several MAP based techniques for improved shelf life of commodities include high oxygen, controlled and intelligent MAP, which are introduced to further improve the shelf life of uncoated and edible coated food products. In MAP assisted food storage, the permeability and physiology of perishable fruit products influence the steady state oxygen and carbon dioxide levels. However, the shortcomings in MAP technology are cost of packaging materials and creating the modified atmosphere with the aid of other gaseous components. The CAP is another advanced packaging technology which helps in monitoring the food products quality, by controlling the atmospheric conditions in headspace. The active packaging is fabricated using various active additives for enhanced freshness such as enzymatic systems (such as alcohol oxidase-ethanol vapor) and chemical systems (ascorbic acid oxidation, iron-sulfur, ferrous carbonate, catechol, etc.). The inclusion of strategies in smart or intelligent packaging system involves the use of indicators, sensors, security tags and radiofrequency identification tags (RFID), barcodes, etc.

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Chapter 14
Edible Food Packaging in Targeted Food Preservation

Kona Mondal, Tabli Ghosh, and Vimal Katiyar

14.1 Introduction

Edible films and coatings can be an ideal replacement for existing conventional polymer due to its remarkable biodegradable and renewable properties. However, petroleum based polymers have been utilized from early days as food packaging material, where, the generation of massive environmental hazardous waste paves the route towards developing green sustainable materials as alternatives. In this regards, edible films and coatings has gained much interest in the last decades by the researchers and industrialists. The edible films and coatings possess many advantages such as low cost, biodegradable, biocompatible, non-toxic, eatable, resistant from mechanical and physical shock, rigid, heat sealable, and versatile nature. However, edible films and coatings are not completely considered as a substitute for existing petroleum-based conventional packaging, thus continuous research and developments are going to combat with exiting packaging materials. The traditional synthetic polymers are nonrenewable and mostly non-biodegradable, and contribute in the massive accumulation of waste, which are generated by continuous disposal of plastics from food packages. In order to minimize the generated waste plastic problem from food packaging material, some alternative need to be identified. In this context, use of biodegradable and renewable food packaging material can be a remedy. On the other hand, as a replacement of non-renewable sources, biobased polymers are not necessarily biodegradable and all biodegradable polymers are not necessarily edible. Therefore, edible films and coatings can be an ideal candidate for providing additional support in this critical circumstance for its renewable and biodegradable characteristics. In this regards, Fig. 14.1 represented the base materials for edible films and coatings, which should

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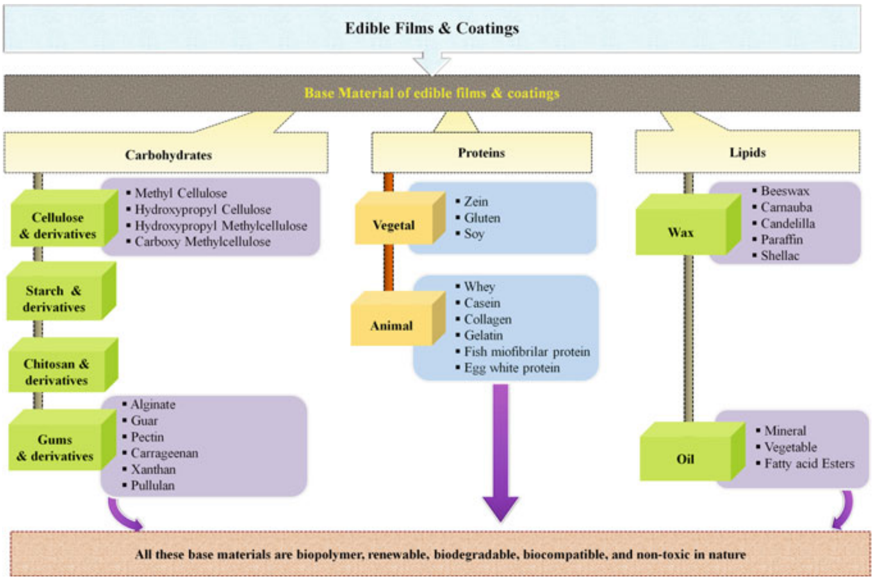

Fig. 14.1 Targeted base materials for edible films and coatings with their characteristics attributes

have the characteristics attributes of biodegradability, non-toxicity, biocompatibility and wide availability.

Edible films and coatings are also used as alternatives for prolonging the product storage life while keeping unaltered nutritional quality, sensory property, and further, maintains the food safety in a sustainable way. Besides, as discussed in earlier chapters, edible films and coatings can be utilized as a carrier material for several bioactive compounds such as nutraceuticals, antimicrobials, antibrowning agents, flavors, colors, antioxidant, and others, which makes it a value-added product by delivering required specific bioactives in food product [1]. The added or natural antimicrobial characteristics inhibit the growth of spoilage causing foodborne pathogens and other microorganisms, thereby, extending the shelf life of food products [2]. The edible films and coatings act as a barrier against moisture, gases, and volatiles via acting as a semi-permeable membrane which again aids to improve the product shelf life as well as the quality. It can be a cost-effective technique as biopolymers are derived from renewable sources and reduce the use of conventional plastics. For instance, Fig. 14.2 describes the major functional and characteristic properties of edible films and coatings. Moreover, the components use for developing edible films and coatings must be food grade and should be recommended as generally recognized as safe (GRAS) elements and further, should be approved by US Food and Drug administration (USFDA) for ensured product safety. In general, the term edible film and edible coating are found to be similar, however, the mode of fabrication and use are different. In this regards, Fig. 14.3 represents the differences between both the approaches of edible food packaging such as films and

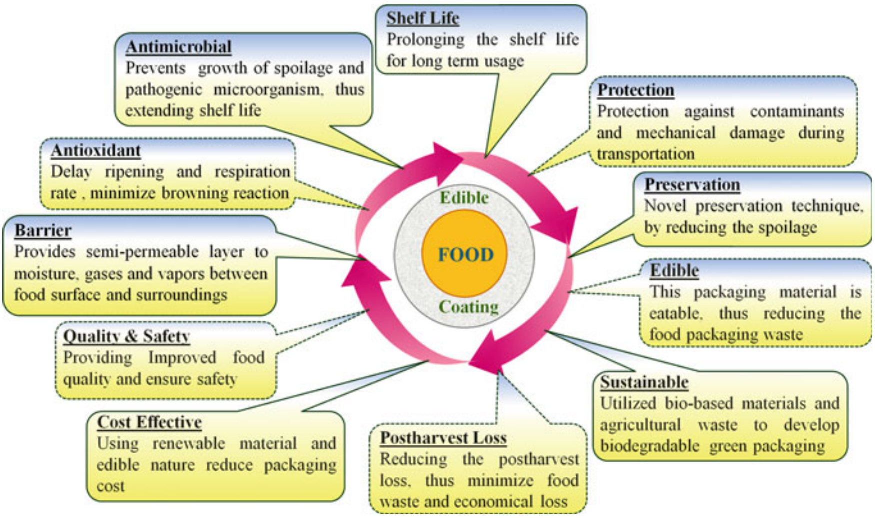

Fig. 14.2 Functional and characteristics attributes of edible films and coatings

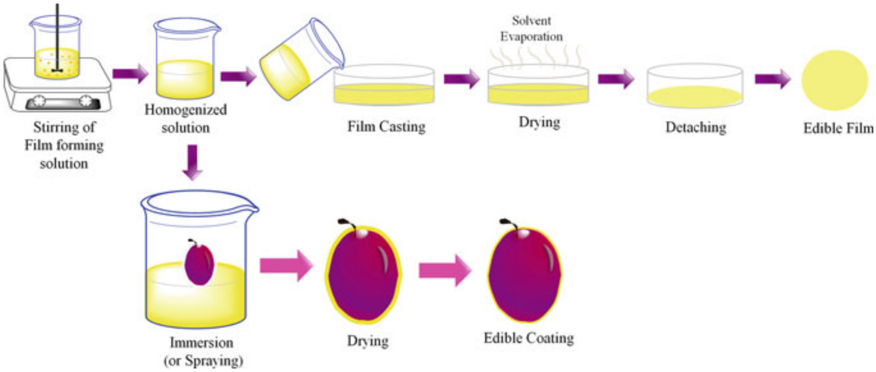

Fig. 14.3 Approaches of edible films and coatings for improved shelf life

coatings. Usually, as mentioned in Chap. 1, edible film is developed and applied on the food products by covering food surface, or can be placed between food components or can be wrapped into edible pouches. Other hand, edible coating is developed onto the food products via direct contact using different methods such as dipping, spraying, brushing and others as discussed in Chap. 2.

As discussed in Chap. 2, the biopolymer based matrixes of edible films and coatings are mainly polysaccharides, proteins and lipids (Fig. 14.1) which must possess cohesive structure and should be compatible with various solvents, plasticizer, cross-linkers, and nanoreinforcement agents for fabricating effective edible films and coatings [3]. In this context, water, ethanol and their combination are commonly used as solvents for developing edible films and coatings via solution casting method with controlled drying temperature as high temperature enhances the rate of solvent evaporation which causes uneven surface formation with inappropriate cohesiveness and structure [4]. Furthermore, plasticizers are added to improve flexibility and to reduce the brittleness, whereas cross-linkers and nanoreinforment agents are incorporated to improve barrier and mechanical properties and inherent characteristics of biopolymers for developing tailored-made edible films and coatings with effective and attractive characteristics. Several polysaccharides, protein, and lipid-based edible films and coatings has been developed for various food products such as fruits and vegetables, meat, fish and other processed foods, which significantly offer improved shelf life, and further, provide improved mechanical and barrier properties without altering sensorial properties during long storage [3, 5]. The edible films and coatings are mostly transparent in nature and commonly does not interact with sensory characteristics. Besides, one of the most noteworthy properties of edible films and coatings include that it acts as bio-preservatives by improving the food and agricultural economy via minimizing postharvest loss. In this regards, India is the principally agriculture-based country where postharvest loss is the common problem which now can be addressed by using edible films and coating based biopreservative technique thereby improves the socio-economic structure of the country.

The several targeted food products are preserved using edible films and coatings. The highly perishable foods such as fruits, and vegetables, meat and various meat products, fish and different fish products, cereals, dairy, and fried products are highly nutritious due to the presence of various macro and micro-nutrients which allow easy growth of microorganism, thereby shortened the shelf-life. In this circumstance, edible films and coatings provide a barrier layer for controlling the permeability of water and gas molecules, thereby, maintain off-flavor generation, lipid oxidation, ethanol production, respiration rate, and microbial growth which eventually extended the shelf life via preventing the growth of spoilage causing microorganisms. However, based on the food products, the applied edible packaging or barrier requirements are different. In this regards, in the sixteenth century, larding process was used to coat meat products for enhancing the shelf life in England [6, 7]. Subsequently, in nineteenth century gelatin coating was patented for preservation of meat product in US patent [5]. This brief history of development of edible coating depicts that concept of edible films and coatings are not new rather it was not appreciated as a preservative technique in early days, however, in twentieth century, people has considered edible films and coatings as a value-added technique. However, different food products require different type of edible packaging materials for improved property. In this regards, fruits and vegetables require low water vapor and oxygen permeability in order to retard desiccation and respiration rates, whereas, if anaerobic environment occurs due to presence of very low amount of oxygen, then off-flavor generation and ethanol production may occur [8]. Similarly, for other food products the condition varies. As described in earlier chapters, the incorporation of various nanostructured organic and inorganic compounds and agricultural extracts into the edible films and coatings formulation offers improved mechanical, thermal and antimicrobial properties and deliver micro-nutrients to the food material. Besides, processed food products such as fried and baked foods also have short life, where, edible films and coatings can be an effective candidate for extending their shelf life by retarding the growth of spoilage causing microorganisms. Based on the discussion, the chapter mainly addresses the application of edible films and coatings on perishable and processed foods such as raw and minimally processed fruits and vegetables, meat and meat products, poultry products, sea foods and fish products, various dairy, bakery and fried products as well as describes their importance and beneficial effects on food products. The chapter also describes the required property of edible packaging for targeted food attributes, and several food attributes can be preserved applying various types of film materials.

14.2 Advances in Edible Films and Coating for Fruits and Vegetables

14.2.1 Nutritional Benefits, Spoilage and Preservation Approaches of Fruits and Vegetables

Fruits and vegetables are highly nutritious, perishable food products and widely recommended to incorporate in a healthy diet, since, is a rich source of several minerals, vitamins, dietary fibers, antioxidant, polyphenols, phytochemicals, and other bioactive compounds which provides essential molecules for maintaining a healthy cell metabolism in human. However, depending on the origin of fruits and vegetables, the categorization of fruits and vegetables are varied. However, the color of fruits and vegetables are also a considerable factor for categorization, where, color can depict the presence of available pigments such as lycopene, anthocyanin, carotenoids, etc. In this regards, the orange colored fruits and vegetables such as carrot is a rich source of carotenoids, however, dark green vegetables also contain high amount of carotenoids. Further, citrus fruits are strongly recommended for rich source of vitamin C, whereas, other fruits and vegetables such as strawberries, green peppers, white potatoes, guava, and others are also recommended for the same. Besides, fruits and vegetables are rich source of starch such as potatoes, corn, avocado, and dried beans. In addition, the whole fruits and vegetables are favored for consumption, by the health directives due to the low content of fat and sodium, which, further capable of nourishing the deficient micronutrient in individual. The diet full of calcium, potassium, dietary fiber, and vitamin D, is highly recommended by the Dietary Guidelines for Americans (2010) [9], which can be served by consuming specific fruits and vegetables. Interestingly, the fruits and vegetables are low in energy, therefore, contribute fewer calories to the diet. Similarly, other international organizations including WHO (World Health Organization, 2002) and FAO (Food and Agricultural Organization, 2003) have strongly recommended the inclusion of fruits and vegetables into the diet as it is associated with the reduction of health risk, cardio-vascular disease, low immune systems, blindness, birth defects, ceasing of mental, and physical growth, diabetes and various forms of cancer [10]. It has been evaluated by the WHO (2002) that chances of occurring mortality risk for diseased condition is more in individuals with lower intake of fruits and vegetables. Further, this organization has reported the lower intake of fruits and vegetables responsible for chances of 19% cancer (gastrointestinal), 31% ischemic heart disease, and 11% stroke. Therefore, consumption of fruits and vegetables are strongly recommended for each and every individual. In addition, the unprocessed raw whole fruits and vegetables consumption are mostly directed by the authorized bodies, however, cut and minimally processed food products undergo no or very minimum loss of nutrients and are unaffected from the use of chemicals, thus capable of providing whole nutritious food. However, the consumption of cut fruits and vegetables has gained popularity as a nutritious ready-to-eat (RTE) product due to hectic life styles in the modern life. The freshly cut fruits and vegetables are treated with slight processing including sorting, cleaning, washing, steaming, peeling, coring, slicing, shredding, packaging and other required steps. Moreover, cut fruits are susceptible to browning due to rapid oxidation by the fruit enzymes in the presence of open environment thus antibrowning agents are mostly applied on cut fruits, whereas in case of vegetables the chances of occurring browning are less. In general, the average product life of freshly cut fruits and vegetables are 10–14 days, whereas the shelf life of stored prepared fruits remains for 1–2 days in catering industry and restaurants [11]. Few examples of commonly available fresh cut fruits and vegetables are apple wedges, sliced and chunk melons, cored and sliced pineapple, peeled citrus fruits, washed grapes, and de-capped berries, sliced kiwi fruits, fruit salads, trimmed spinach, chopped tomatoes, trimmed green onions, shredded lettuce, slice carrots, mixed salads, broccoli florets, and others.

The nutritious nature of fruits and vegetable allow extremely favorable condition for rapid and easy growth of microorganism by providing nutrient and water, and subsequently becomes unacceptable for eating. Besides, fruits and vegetables are getting contaminated at different stages of the development and during field-to-fork transportation by contaminated soil, water and manure, contaminated seed and harvesting equipment, and favorable condition for proliferation of microbes during transportation and storage. Further, owing to the fact that fruits and vegetables consists of living tissues, biochemical degradation may occur due to different reasons such as physical and mechanical damage during harvesting, handling, washing, processing and transportation, which damage the food products by enhancing the chances of microbial attack. The outgrowth of spoilage causing bacteria also occurs from the external damages including cracks, bruising, and puncture. The development of lesion further increases the risk of rapid outbreaks of spoilage causing microorganisms which also enhances the risk of cross-contamination during culling, washing, sorting, and packing before storing for longer period. Therefore, the quality maintenance of whole fruits and vegetables for prolonged time is difficult. The genera of commonly occurring microorganisms responsible for postharvest spoilage in whole fruits (Apple, Banana, Berries, Citrus, Grapes, Melon, Peach, Pears, and Pineapples) are *Pseudomonas*, *Erwinia*, *Xanthomonas* and *Acidovorax* [12]. Moreover, the two most predominant spoilage causing fungi, responsible for occurring wound on the fruits surface are *Penicillium expansum* and *Botrytis cinerea*, and insufficient cleaning of whole fruits before storage is the main source of the growth of the mentioned microorganisms [12]. Vegetables are more prone to have soft rot disease by the several pathogenic bacteria, among them six common soft rot causing bacteria are *Xanthomonas*, *Pseudomonas*, *Clostridium*, *Cytophaga*, and *Bacillus*, whereas the most effective bacteria is *Erwinia carotovora* subsp. *carotovora* causing massive pre and postharvest injury. The visualization of water type transparency in leafy plant and watery disintegration in non-leafy pants indicates occurrence of soft rot. The decay occurs by soft rot via degradation of pectin by the *Erwinia* pathogen derived-enzyme pectate lyase and the disease occurs at the temperature of 20 °C and above. Further, *Pseudomonas fluorescens* and *Pseudomonas viridiflava* are accountable for appearing soft rot disease at the temperature of 4 °C or below. Further, *Pseudomonas tolaasii* is another spoilage causing bacteria, which affect white mushroom (*Agaricus bisporus*) where the microbe creates blemishes on caps and stems thereby started decaying or infecting in mushroom [12].

The fresh-cut fruits and vegetables are also exposed to microbial attack by various Gram-positive, Gram-negative bacteria, yeasts, molds, fungi and spoilage causing pathogens. Mostly, the contamination occurs in fresh cut products originated from pre and post harvesting process and usage of contaminated machineries. The common mold *Geotrichum candidum* contaminates fresh-cut fruits from the fruit processing equipment. The decaying in fresh cut vegetables such as cut lettuce, celery products and others is caused by *Erwinia*, *Pseudomonas*, *Aeromonas*, *Bacillus* spp. etc. Besides, *Salmonella* and *Listeria monocytogenes* are common spoilage causing pathogen whereas several varieties of yeasts cause spoilage in fresh cut products (on sliced carrots) including *Candida*, *Cryptococcus albidus*, *Rhodotorula*, *Trichosporon penicillatum*, and *Saccharomyces cerevisiae*. Besides, the isolated fungi from salads are *Alternaria*, *Cladosporium*, *Penicillium*, *Alternaria*, *Cladosporium*, *Aspergillus*, and *Fusarium*. Moreover, the spoilage of whole and cut fruits and vegetables can be controlled and minimized via several existing methods and techniques including modified atmosphere packaging (MAP), controlled atmosphere packaging (CAP), refrigerated storage, providing suitable environment, while maintaining the standard guideline for minimizing the microbial hazards via following the rules of good agricultural practices (GAPs) by FDA. In this context, edible films and coatings are one of the value added packaging systems for extending the shelf life of whole and cut fruits and vegetables, are discussed in below section.

14.2.2 Effect of Edible Films and Coatings on Whole and Fresh Cut Fruits and Vegetables

The preservation of whole and cut fruits and vegetables by the application of edible films and coatings contributes several benefits such as improved organoleptic properties, appearance and texture, reduction in weight loss, reduction in rate of respiration and reduction in ethylene production. Thus, this delays the ripening rate, and further minimize the degradation caused by microbial attack and enzymatic browning, which reduces the loss of aroma, flavor and color. The application of this specific technique protect vitamins, micronutrients, antioxidants, and pigments, and also, reduce the risk of chilling injury during frozen storage, and finally delivers value added product via incorporating nutraceuticals and various bioactive molecule as food additives [3]. Besides, some drawbacks are reported by researchers including allergic reaction, food safety, less cost-effectiveness, and need of secondary packaging materials for maintaining the safety and quality as they are less physically and chemically resistant than conventional polymers. Several edible films and coatings have been developed and provide effective results in retarding the microbial growth with extended shelf life. Besides, various biopolymers based edible films and coatings with added food additives and their effect on whole and cut fruits and vegetables are discussed in Table 14.1. Among carbohydrates, chitosan (CS) and its derivative based edible films and coatings are much effective and popular. In order to obtain best characteristic property of edible films and coatings several natural food additives such as essential oils (EOs), plant extract, antioxidant, antimicrobial and antibrowning agents, nanomaterials and others are incorporated into carbohydrate, protein and lipid based matrices. In this regards, ascorbic acid incorporated CS based edible coating can be utilized for preservation of fruits and vegetables, where the developed coating can maintain the nutritional and sensory of pomegranate arils for 28 days at 5 ± 1 °C [13]. The CS/ascorbic acid based edible coating system on pomegranate arils reduces the fungal and bacterial growth and inhibits aerobic mesophilic count, thereby, extending the shelf life of coated aril as compared to uncoated arils (control). The coating has no effect on anthocyanin, organic acids, and sugars on the arils during storage, however, sensory score (color, taste aroma) and visual quality are higher for coated arils and acceptable after 25 days of refrigerated storage. The ascorbic acid incorporated CS based edible coating can extend the shelf life of coated arils up to 21 days under refrigerated condition, where, control is unacceptable within 10 days of storage. Similarly, CS incorporated with olive oil residues extracts (OOR) based edible films can be developed for extending the storage life and reducing the spoilage causing pathogen on apple and strawberry fruits under refrigerated condition (4 ± 1 °C) [14]. The developed CS/OOR edible film is capable of inhibiting spoilage causing pathogen *Penicillium expansum* and *Rhizopus stolonifer*. Further, fresh cuts products can also be preserved by developing transglutaminase crosslinked whey protein/pectin based edible films while applying on fresh cut apples, potatoes and carrots [15]. The addition of enzyme transglutaminase helps to preserve phenolic and carotenoid in

Table 14.1 Several applications of edible films and coatings on whole and freshly-cut fruits and vegetables

Types of fruits and vegetables	Packaging materials	Effects	References
Pomegranate arils	CS/Ascorbic acid	• Active edible coating • Improved shelf life and sensory attributes • Reduced growth of fungi and bacteria via antimicrobial properties	[13]
Apples, strawberry	CS/Olive oil residues extract	• Active edible film • Improved shelf life and antioxidant activity • Effective against spoilage causing pathogen • Improved antimicrobial activity	[14]
Fresh cut apples, potato, carrots	Whey protein/ Pectin/ Transglutaminase	• Active edible film • Reduction in weight loss • Preserved antimicrobial activity • Extended shelf life • Preserved hardiness and chewiness	[15]
Strawberry	NCS, NCS/cu	• Edible coating • Delayed ripening rate • Enhanced shelf life • Prevented microbial counts • Preserved bioactive and sensory properties • Reduced weight loss	[16]
Banana	CS, CSNPs	• Edible coating • Delay in ripening process • Improved shelf life • Maintained quality and sensory attributes	[17]
Cucumber	CSNPs/ZEO	• Active edible coating • Improved antioxidant and antimicrobial properties • Enhanced shelf life • Maintained quality attributes	[18]
Tomato, chilly, brinjal	CSNPs	• Edible coating • Improved shelf life • Remarkable antimicrobial activity against plant pathogen	[19]
Fresh cut eggplant	SPI-cys	• Active edible coating • Controlled enzymatic browning with maintained visual appearance • Improved storage life up to 8–9 days at 5 °C • Provided an approach to enhance the storage life at atmospheric condition	[20]
Freshly cut mango	Alginate/ascorbic acid/citric acid	• Active edible coating • Carrier for antibrowning agent • Significant reduction in browning effect • Enhanced antioxidant activity and improved colored appearance during storage for up to 12 days at 4 °C	[21]

(continued)

Table 14.1 (continued)

Types of fruits and vegetables	Packaging materials	Effects	References
Fresh cut broccoli	CS	• Edible coating • Remarkable antimicrobial activity against mesophilic, psychrotrophic, yeast and molds, lactic acid bacteria and coliforms • Improved appearance, quality and sensory attributes via inhibiting yellowing and opening florets	[22]
Shiitake mushroom	Gum Arabic/ natamycin	• Edible coating • Reduced yeast and mold growth • Extended shelf life up to 16 day with maintained sensory and textural properties	[23]
Shiitake mushroom	CS/Gum arabic	• Edible coating • Slowing down rate of decreasing ascorbic acid and soluble protein and increasing rate of TSS, reducing sugar, MDA and electrolyte leakage during refrigerated storage (4 ± 1 °C) for 16 days • Improved quality and sensory attributes	[24]

CS Chitosan, *Cu* Copper, *NCS* Nano chitosan, *CSNP* Chitosan nanoparticle, *TSS* Total soluble solid, *MDA* malondialdehyde, *ZEO Zataria multiflora* essential oil, *SPI-cys* Soy protein isolate-cysteine

cut products and prevents microbial growth. The remarkable effects of the developed edible films on the cut products results in reduced weight loss, maintained hardness and chewiness during the storage of 10 days as compared to control. As mentioned above, nanomaterials are also used in the preservation of fruits and vegetables via developing edible films and coatings. In this regards, nanochitosan (NCS) based edible coating with or without copper loading can be useful in order to extend the storage life of fresh strawberry at 4 ± 1 °C with 70% relative humidity (RH) for 20 days [16]. The developed coating system can inhibit polyphenol oxidase and peroxidase activity, which further reduce the respiration rate, weight loss, and preserve anthocyanin concentration, bioactive compounds. Besides, CS nanoparticles (CSNPs) based edible coating can maintain post-harvest quality when applied on banana for longer storage at ambient condition (25 ± 1 °C) [17]. Both CS and CSNP based edible coating are effective in maintaining weight, texture, sensory properties, total soluble solid and quality attributes. In addition, the developed edible coating helps to delay ripening rate and extend the shelf life of banana during storage. Moreover, *Zataria multiflora* extracted EO (ZEO) incorporated CSNP based edible coating on cucumber helps to improve the shelf life under refrigerated storage at 10 ± 1 °C, 90–95% RH for 21 days [18]. The ZEO aids better effects in developed coating system as a result provide improved antioxidant activity and antimicrobials effect, reduced respiration rate, and maintained quality and sensory attributes during storage. Further, the shelf life of tomato, chilly, brinjal can be extended via developing CSNP based edible coating [19]. The CSNP based edible coating system is able to provide remarkable antimicrobial properties against plant pathogens such as *Rhizoctonia solani*, *Fusarium oxysporum*, *Colletotrichum acutatum*, and *Phytophthora infestans*.

14.3 Advances in Edible Films and Coating for Minimally Processed Fruits and Vegetables

14.3.1 Nutritional Benefits, Spoilage and Preservation Approaches of Minimally Processed Fruits and Vegetables

With the growing trends of modern lifestyles, people are becoming health conscious and are more interested towards maintaining healthy life via consuming fresh foods without added harmful chemical agents. Based on the current circumstances, food industries have put a lot of interest towards manufacturing minimally processed fruits and vegetables to meet the consumer's needs for accessing convenient, less time consuming and healthy food products. Since, fruits and vegetables contribute lots of micronutrients and functional biomolecules for a healthy diet, as mentioned in the previous section. In this regards, the minimally processed fruits and vegetables encompasses all processing techniques including washing, sorting, trimming, peeling, coring, slicing, low-level irradiation, individual seal packaging and others that does not have effect on the quality of fresh produces [25]. For instance, the minimally processed fruits and vegetables remain biologically and physiologically active with living tissues that are able to respire. Generally, minimally processed fruits and vegetables provide all the beneficial healthy component similar to the fresh produces, however, there is a chance of providing less nutrient due to loss of essentials during processing such as cutting, peeling etc. where tissue damage occurs which led to cease the production of valuable component.

Generally, minimally processed products possess high degree of perishability due to the effect of processing conditions. Further, minimal processing causes physiological changes in fruits and vegetables via disruption of cell wall and membranes, leading to damage of cell tissue, loss of ions, essential component, cell rigidity and alteration of membrane flux. Besides mechanical damage such as cutting activates the activity of certain enzymes polyphenol oxidase and peroxidase which leads to the formation of enzymatic browning and generation of off-flavor via reacting with phenolic compounds and oxygen. Whereas, the structural polysaccharide gets degrade by pectin lyase and polygalacturonase enzyme, leading to cell wall degradation which affects the textural property. The membrane lipids also get affected via lipoxygenase and lipase enzyme which are related to senescence. In addition, the oxidative and hydrolytic changes by enzymes further leads to loss of flavor, texture, palatability in regular and biological hindrance pathways. The cut surfaces of fruits and vegetables are usually high in moisture content which enhances the enzymatic activity. In this context, the cut injury also generates stress in cell tissue that leads to the formation of secondary metabolites and production of ethylene, which increase the ripening rate. Moreover, the above mentioned reasons allow easy microbial growth at the site of wound generated by mechanical injury and finally spoil the minimally processed products. Besides, the chance of occurring microbial spoilage is more in minimally processed products due to partial or total loss of skin which have exposed cell components containing high carbohydrate, protein, moisture and other components to the food spoilage causing bacteria thereby enhance the chances of foodborne illnesses [26]. The spoilage causing microorganisms affecting the minimally processed fruits and vegetables are *Erwinia herbicola*, *Pseudomonas* spp., *Flavobacterium*, *Xanthomonas*, *Enterobacter Agglomerans*, *Leuconostoc mesenteroides*, *Lactobacillus* spp., *molds*, and *yeasts* [10]. Therefore, advanced technology is required in order to prolong the shelf life while keeping the quality and safety unaltered in minimally processed fruits and vegetables. In this regards, MAP, CAP, refrigerated storage, packaging and other techniques have been utilized. However, in MAP, there is a chances of growth of *Clostridium botulinum* on fruits at higher pH of 4.8 thereby produce toxins, which lead to causing foodborne disease. In this context, edible coating can be an ideal candidate to resolve the drawbacks via coating the food surface which eventually reduce the chances of microbial growth in minimally processed fruits and vegetables [27].

14.3.2 Application of Edible Films and Coatings on Minimally Processed Fruits and Vegetables

The application of edible films and coatings on minimally processed fruits and vegetables control water loss, delay ripening, and respiration rate, maintain visual and textural appearance, reduce browning, prevent microbial growth, and ultimately enhance the product shelf life. In this context, aloe vera based edible coating on minimally processed kiwifruit can prevent the microbial contamination [28]. The coating material delays respiration rate and prevents mesophiles, yeast and molds count in sliced kiwifruit and extends the shelf life under refrigerated storage (4 ± 1 °C) as compared to uncoated sample. Further, the aloe vera coating minimizes the pectin degradation and maintains the texture and firmness in coated fruits. The edible film of protein-lipid based emulsion such as casein/beewax/stearic acid are able to minimize common problem of white blush in minimally processed peeled carrot due to surface dehydration [29]. Besides, the developed film system reduces the water vapor permeability and respiration rate. For instance, the chance of occurring microbial growth in minimally processed carrot can be reduced using starch-CS edible antimicrobial coating. This formulated edible coating system inhibits mesophilic aerobes, molds, yeast, psychrotrophs, lactic acid bacteria and total coliforms during the refrigerated storage at 10 °C for 15 days [30]. The postharvest quality of several minimally processed fruits such as strawberry can also be improved via starch based edible coating [31]. The starch based coating is able to reduce respiration rate and improve water vapor resistance thereby extending the shelf life. Edible active coating of agar incorporated with CS and acetic can act as an antimicrobial edible coating when applied on minimally processed garlic cloves stored at 25 °C for 6 days [32]. The developed film enhances the shelf life of coated garlic with maintained quality and color attributes by preventing microbial growth of yeast and fungi, retarding the respiration rate and reducing water vapor permeability as compared to uncoated garlic. Interestingly, minimally processed mango can be wrapped by mango based edible films for prolonging the shelf life of the mango fruit while storing under refrigerated (5 °C) and ambient temperature (30 °C) [33]. The tissues of ripe mango fruit are able to form edible films and coatings which provides good oxygen barrier and mechanical properties to wrap mangoes as well as provide reduce weight loss, ripening and extend shelf life of wrapped mangoes. However, future studies are required to improve the film property as mango based edible films and coatings are hydrophilic in nature thereby it is highly soluble which limits its application. The various application of edible films and coatings on minimally processed fruits and vegetables has been represented in Table 14.2.

14.4 Advances in Edible Films and Coating for Meat and Meat Products

14.4.1 Nutritional Benefits, Spoilage and Preservation Approaches of Meat and Meat Products

Meat is one of the animal originated foods, which is recommended for being an available nutrient source required for optimal growth and development of human [34]. Generally, it is a source of several nutrients such as high quality protein, essential amino acids, bio-available vitamins and minerals [5]. The valuable micronutrients of meat are iron, selenium, zinc, and vitamin B_{12}. Besides, meat offal such as liver is a rich source of vitamin A and folic acid [35]. Among available meat and meat products, red meat is the rich source of protein, which has high digestibility scores as compared to other plant proteins. The meat proteins are distinguished from other proteins due to the availability of all essential amino acids, thereby, provide enhanced food value. Further, essential amino acids are required for cell growth metabolism in human body and absence of these can lead to protein malnutrition. On the other hand, fat is another rich constitute of meat and meat product. However, the quantity of fat depends on the species and various cut of meat. Among all, beef and pork are consumed worldwide due to its high fat and fatty acid content. On the other hand, cooking can lead to loss of fat content in meat

Table 14.2 Various application of edible films and coatings on minimally processed fruits and vegetables

Types of fruits and vegetables	Packaging materials	Effects	References
Sliced kiwifruit	Aloe vera	• Edible coating • Retention of quality via maintaining texture and retarding pectin degradation • Delay respiration and yellowing rate • Reduced microbial count thereby enhancing shelf life	[28]
Peeled carrots	Casein/ Beewax/ Stearic acid	• Edible film • Formation of white blush reduced • Reduction in respiration rate • Improved water vapor resistance	[29]
Peeled and sliced carrot	Starch/CS/ Glycerol	• Edible active coating • Improved shelf life • Prevented growth of total coliform, mesophiles, yeast, molds, psychrotrophs, lactic acid bacteria and *Staphylococcus aureus*	[30]
Strawberry	Cassava starch	• Edible coating • Delayed respiration rate • Extended shelf life	[31]
Garlic	Agar-agar/ CS/acetic acid	• Edible active coating • Reduced moisture loss, rate of respiration, water vapor transmission • Inhibited growth of aerobic mesophilic and filamentous fungi	[32]
Mango	Ripe mango	• Edible film • Reduction in weight loss, ripening rate • Provide oxygen barrier and mechanical property • Prolonging shelf life	[33]

CS Chitosan

when subjected to grilling, broiling, and pan frying, which increases the polyunsaturated/saturated fatty acid ratio due to the less exposure of polyunsaturated fatty acid with heat as being present inside the cell membrane [36]. However, high consumption of meat increases the saturated fatty acid content in human that led to the formation of cardiovascular diseases. In addition, meat contains some amount of trans-fatty acid, which has the positive role in controlling obesity, diabetes, cardiac disease, etc. Depending on the food habit of animals, meat can also contribute to higher percentage of polyunsaturated fatty acid especially omega-3 fatty acid, responsible for protective nature of cardiac diseases and can be a replacement for fish oil. Besides, meat and meat products are outstanding source of vitamins and minerals including niacin, riboflavin, vitamin B_6, and B_{12} as well as several mineral contributing healthy growths in children and fulfill the deficiency of the above in human diet via consumption.

Since meat and meat products are highly nutritious, therefore, chances of causing spoilage are adequate. The chances of meat spoilage depend on the handling of livestock during pre and post-slaughter condition. The two most powerful controlling factor of meat quality is pH and temperature. The pH of meat varies from 5.4 to 5.7 in post-rigor condition based on the storage carbohydrate, and glycogen content. After slaughtering, meat immediately needs to be transferred in refrigerator (4–8 °C) for maintaining the quality [5]. Moreover, the meat and meat products are expected to degrade the quality during processing and storage via three main phenomenon including microbial spoilage, lipid oxidation, and enzymatic hydrolysis. The microbial contamination in meat products occurs from several sources such as (1) Naturally present microflora in intestinal tract; (2) Cross contamination during handling, processing; and (3) from the environment [37], which results in decreasing water holding capacity, off odor, changes in appearance and texture, degradation of structural components, and slime formation [38]. Further, the major factor for lipid oxidation are prooxidants (free iron in muscle), concentration of vitamin E, and fatty acids composition. The oxidative degradation of carbohydrate, protein, lipid, pigment, and vitamin produces hydroperoxides, aldehydes, and ketones, which led to loss of color and nutritive value [5]. However, the enzymatic degradation of carbohydrates, proteins, and fats result in tissue softening and formation of greenish discoloration, which allows microbial spoilage. Interestingly, under refrigerated condition (5 °C), the proteolytic enzyme can cause degradation, leading to loss of water holding capacity, amines production, and possible microbial growth [39]. Generally, meat and meat products are susceptible for several pathogenic and non-pathogenic microorganisms including *Salmonella* spp., *Listeria* spp., *Clostridium* spp., *Staphylococcus* spp., *Campylobacter* spp., *Pseudomonas* spp., *Acinetobacter*, *Brochothrix thermosphacta*, *Lactobacillus* spp., *Enterobacter*, *Escherichia coli*, *Listeria monocytogenes*, *Clostridium* spp., *Aeromonas hydrophila*, yeast, and mold etc. [5, 40]. Among available, *Salmonella* is the main spoilage causing pathogen in meat and meat products. Besides, *Listeria* is the most lethal among others and *Salmonella typhimurium* is common in ground beef [5]. All the mentioned microorganisms cause quality defects including generation of off-flavor, off-odor, and are responsible for causing foodborne illness. However, during cooking, most of all vegetative cells of pathogenic microorganisms gets destroyed, but, their toxins and spore remain active. Therefore, a barrier is required for inhibiting the growth of microorganism by keeping unaltered quality and safety. In this regards, the conventional physical and chemical methods of treating meat and meat products has some drawbacks including usage of synthetic chemical additives, and insignificant outcome. Further, consumers' demand for preservation with natural biodegradable preservatives such as edible films and coatings are considered as one of the suitable candidates for preservation of meat and meat products.

14.4.2 Attributes of Edible Films and Coatings on Meat and Meat Products

Several applications of edible films and coatings on meat and meat products and their effects are summarized in Table 14.3. Among available biopolymers, CS is extensively used edible packaging materials for meat and meat products due to the natural antimicrobial activity along with biodegradability. However, the various meat and meat products such as fresh pork meat are highly perishable and more prone to oxidation and microbial attack. In this regards, the CS and gelatin based coatings incorporated with grape seed extract (GSE) is able to suppress the oxidation and inhibit the microbial attack for 20 days under refrigerated storage (4 °C) [41]. In addition, GSE is a natural food additive and is incorporated to CS based edible coating system for enhanced effectivity. The GSE incorporated edible coating is more effective in inhibiting oxidation and microbial growth as compared to uncoated and CS-gelatin based edible coating. Besides, the specified edible coating system prolongs the shelf life of refrigerated fresh pork via (1) Minimizing the changes in pH value, (2) Preventing lipid and protein oxidation, and (3) Inhibiting the microbial growth. The incorporation of GSE enhances the antioxidant activity in order to minimize the protein and lipid oxidation, and further, provides red and yellow color effects to pork meat due to the presence of phenolic compounds in GSE. On the contrary, in some cases EO (basil and thyme) incorporated CS based edible films may not show significant activity as compared to only CS based edible films as added EO increases the oxygen permeability, therefore, color changes can be observed while applying on minced pork meat, however, the antimicrobial properties remain unaltered for both the case [42]. Thus, selection of EO and their effects on CS needs to be carefully examined. Similarly, CS based edible films can be used to control the growth of *Listeria monocytogenes*, a common pathogen in RTE meat product while applying on RTE roast beef during frozen storage at 4 °C [43]. Besides, antimicrobial activity, CS/gelatin/glycerol based edible coating system are able to control the color of beef steak by reducing myoglobin oxidation and, further, control the lipid oxidation [44].

Besides carbohydrate based edible films and coatings, whey protein isolate (WPI) based edible films can be able to improve the storage life of meat products such as bologna and summer sausage with the incorporation of p-aminobenzoic acid (PABA) and/ascorbic acid under refrigerated storage (4 °C) for 21 days [45]. This system is effective to inhibit the growth of *L. monocytogenes*, *E. coli* O157:H7, and *S. Typhimurium*. Further, beef is another highly nutritious source of meat and mostly affected by the microorganism if not stored properly. In this context, incorporation of natural antimicrobial agents (EOs) into the edible films system can be able to deliver noteworthy antimicrobial properties. Therefore, a designed system of soy protein based edible films incorporated with oregano and thyme EOs can provide significant antimicrobial effect when applied on fresh ground beef patties [46] under frozen storage at 4 °C.The designed system inhibits growth of *E. coli*, *E. coli* O157:H7, *Pseudomonas* spp., and *S. aureus* where, *L. plantarum* and

Table 14.3 Application of edible films and coatings in preserving meat and meat products

Types of meat and meat products	Packaging material	Effects	References
Fresh pork	CS-gelatin/grape seed extract	• Edible active coating • Inhibited pork oxidation and microbial spoilage • Extended shelf life	[41]
Fresh ground beef patties	Soy protein/ oregano/thyme EOs	• Antimicrobial Edible film • Remarkable antimicrobial activity against *E. coli*, *E. coli* O157:H7 and *S. aureus*	[46]
Beef slice	Medicinal plant based coating (LRSM)/AHEO	• Active edible coating • Extended shelf life • Improved antimicrobial activity and oxidative stability	[47]
RTE roasted beef	CS/acetic acid	• Edible film • Inhibited the growth of *Listeria monocytogenes*	[43]
Bologna and summer sausage	WPI/PABA/SA	• Edible film • Remarkable antimicrobial activity against food pathogen • Extended shelf life • Percentage of elongation increased while tensile strength decrease	[45]
Beef steak	CS/gelatin/glycerol	• Edible coating • Remarkable improvement in color stability in retail display • Reduction in myoglobin oxidation • Controlled lipid oxidation	[44]
Buffalo meat patties	Sodium alginate Calcium chloride	• Edible coating • Improved quality by decreasing the overall shear force, TBA and tyrosine value during frozen storage (4 °C) • Reduction in TPC, psychrophilic, yeast and mold count • Improved overall appearance, color, juiciness, flavor, texture and palatability	[48]
Pork patty	Pectin/green tea powder Irradiation	• Edible coating • Reduced lipid oxidation and increased radical scavenging property during 14 days' storage at 10 °C • Aerobic bacteria count reduced via edible coating and coated vacuum packed sample irradiation separately	[49]
Chevon sausages	Calcium alginate/ *Asparagus racemosus*	• Active edible film • Controlled lipid oxidation • Improved antimicrobial and antioxidant activity • Lowered TBARS and FFA values	[50]

(continued)

Table 14.3 (continued)

Types of meat and meat products	Packaging material	Effects	References
Fresh beef patties	Apple peel powder/ CMC/Tartaric	• Edible coating • Complete inhibition of lipid oxidation keeping appearance and other quality property unaltered under refrigerated storage • Improved antimicrobial activity against mesophilic aerobic bacteria, yeast, molds, *Salmonella enterica*	[51]
Beef steak	Alginate/Rosemary/ Oregano EO	• Active edible coating • Reduction in lipid oxidation, water loss • Improved antioxidant, antimicrobial activity and color properties • Oregano EO is more effective	[52]
RTE cooked pork chops	CS/bamboo vinegar	• Edible coating • Improved shelf life under refrigerated storage (4 ± 1 °C) for 12 days • Retained color properties • Improved antimicrobial activity against TVC, LAB, *Enterobacteriaceae* and *Pseudomonas* spp. • Improved antioxidant activity thereby controlling lipid oxidation	[53]

CS Chitosan, *EO* Essential oil, *LRSM Lallemantia royleana* seed mucilage, *AHEO Allium hirtifolium* essential oil, *RTE* Ready-to-eat, *WPI* Whey protein isolate, *PABA* p-aminobenzoic acid, *SA* Sorbic acid, *TPC* Total plate count, *TBARS* Thiobarbituric acid reactive substances, *CMC* Carboxymethyl cellulose, *TVC* Total viable count, *LAB* Lactic acid bacteria

P. aeruginosa are resistant against the antimicrobial edible film. Further, the film is not significantly effective against total viable counts, lactic acid bacteria, and *Staphylococcus* spp. Moreover, medicinal plant *Lallemantia royleana* seed mucilage extract based edible coating incorporated with *Allium hirtifolium* EO system can also extend the storage life of slice refrigerated beef (4 °C) during storage life (18 days) via inhibiting microbial load and lipid oxidation [47].

14.5 Advances in Edible Films and Coating for Poultry Products

14.5.1 Nutritional Benefits, Spoilage and Preservation Approaches of Poultry Products

Poultry products contribute high nutrition to the diet similar to meat and meat products. Generally, poultry products include chicken, turkey, duck, egg, etc.

Poultry products are also rich sources of protein, fat, and several micronutrients (vitamins and minerals). In addition, chicken breast contains high amount of protein around 34.5% with the energy value of 176 kcal with skin and 108 kcal without skin [36]. However, the skin of chicken meat is the principle source of fat in poultry meat. Generally, poultry meat without skin has fat content around 1–15% and the value can be more in meat with skin. The turkey legs contain higher amount of fat in comparison to chicken legs, however, chicken and turkey breasts contain similar fat content. Among poultry products, chicken breast is a rich source of niacin and vitamin B_6, where, turkey breast contains lesser amount. On the other hand, egg is also a rich source of protein, fat, riboflavin, folate, vitamin E, B_6, A and B_{12}, minerals (zinc, calcium, iron) and others. Besides, egg is also a rich source of cholesterol [54]. Therefore, the inclusion of poultry products in healthy balanced diet is highly recommended due to the nutritional benefits.

Since, poultry products are highly nutritious, thus, chances of occurring microbial spoilages are common. In this context, *Salmonella* and *Campylobacter* are the most popular microorganism for causing food poisoning in chicken [5]. Another, food pathogen in turkey meat causing foodborne illness is *Clostridium perfringens*. Besides, *Salmonella* also affects the turkey [5]. Therefore, numerous preservation technique has been developed for arresting the microbial spoilage with targeted enhanced storage life of poultry products with maintained safety and quality. In this context, besides, existing conventional approaches, MAP is widely accepted technique for preserving food products for a longer time. However, the preservation of poultry skinless breast fillets and thigh meat under refrigerated MAP condition allows the growth of lactic acid bacteria, *Brochothrix thermosphacta,* and *pseudomonas* [55]. Similarly, the refrigerated storage of poultry meat in high and low oxygen (O_2) MAP shows growth of *Brochothrix thermosphacta*, *Carnobacterium* spp. and *Pseudomonas* spp. in high O_2 and *Hafnia alvei*, *Carnobacterium* spp., Serratia spp., and Yersinia spp. in low O_2 MAP environment [56]. Therefore, considering the fact, edible films and coatings can be an ideal alternative for keeping poultry products safe for long term usage as direct contact of coating material and food may have high effect in controlling microbial growth as well as the quality.

14.5.1.1 Effect of Edible Films and Coatings on Poultry Products

In terms of maintaining the quality and safety of poultry products with enhanced shelf life, various application of edible films and coatings with their effective attributes are summarized in Table 14.4. The developed edible films and coatings include the use of carbohydrates, proteins, and lipids as matrix material with or without various edible food additives such as antimicrobials, antioxidants, flavoring agent, nutraceuticals, and others. The various food additives are incorporated to matrix materials to enhance the activity of developed edible films and coatings. In this regards, WPI based edible coating incorporated with oregano and clove EO can be used for improving the quality and extending the shelf life of chicken breast

Table 14.4 Application of edible films and coatings and their effectiveness for maintaining the quality and shelf life of poultry products

Types of poultry products	Packaging material	Benefits	References
Chicken breast	WPI/Oregano/ Thyme EO	• Edible coating • Doubling shelf life under refrigerated storage • Improved antimicrobial activity and quality of the product	[57]
Cooked sliced turkey meat	Sodium caseinate/glycerol	• Edible film • Reduction in lipid oxidation with less TBARS value	[59]
RTE-Turkey Frankfurter	WPI/nisin/GSE/ malic acid/EDTA	• Edible active coating • Excellent antimicrobial agent • Inhibited growth of *Listeria monocytogenes*, *E. coli* O157:H7, and *Salmonella typhimurium*	[58]
RTE-deli turkey meat	CS/lauric arginate ester/nisin	• Edible active film and coating • Antimicrobial activity against *Listeria innocua*	[60]
Chicken strips	MC, HPMC	• Edible film • Developed better quality in fried food with higher fat and lower moisture content while frying edible film coated chicken strips • Reduced degradation of frying oil	[61]
Chicken meat	Gelatin/nisin/ EDTA/ potassium sorbate	• Edible active coating • Significant reduction in drip loss • Improved shelf life and color	[62]
Chicken breast fillet	Nanoemulsion based sodium caseinate/GEO	• Edible active coating • Improved antimicrobial activity • Reduction in growth of aerobic psychrophilic bacteria during refrigerated storage for 12 days • Maintained color, lowest cooking loss and extended durability of the product	[63]
Chicken Wingettes	Gum Arabic/CS/ carvacrol	• Edible coating • Significant reduction in growth of *Campylobacter jejuni* and total aerobic count thereby ceasing of foodborne disease • Controlled color difference during refrigerated storage	[64]
Fried Chicken meat ball	Oat flour/wheat flour/corn flour	• Edible coating • Improved sensorial properties such as appearance, color and taste after frying coated chicken meat ball	[65]

(continued)

Table 14.4 (continued)

Types of poultry products	Packaging material	Benefits	References
Egg	CS/WPC/mineral oil/soybean oil	• Edible coating • Prolonged shelf life for 4 and 20 weeks during storage at 25 and 4 °C, respectively • Minimize weight loss	[66]

CS Chitosan, *EO* Essential oil, *MC* Methyl cellulose, *HPMC* Hydroxypropyl methylcellulose, *EDTA* Ethylenediaminetetraacetic acid, *GSE* Grape seed extract, *GEO* Ginger essential oil, *TBARS* Thiobarbituric acid reactive substances, *WPC* Whey protein concentrate

during frozen storage (4 °C) [57]. The EOs added system act as antimicrobial agent in the developed system where oregano is most effective. The EO incorporated edible coating has doubled the product life of chicken breast from 6 to 13 days by inhibiting the total mesophilic aerobic, *Pseudomonas* spp. and lactic acid bacteria counts. Similarly, WPI based edible coating incorporated with nisin, GSE, malic acid, and ethylenediaminetetraacetic acid can act as an effective antimicrobial agent for inhibiting microbes on RTE turkey frankfurter during storage at refrigerated temperature (4 °C) [58]. This combined WPI based active edible coating prevents the growth of *Listeria monocytogenes*, *E. coli* O157:H7, and *Salmonella typhimurium*. Another protein based edible films, sodium caseinate along with glycerol is able to reduce the lipid oxidation when used on cooked sliced turkey breast meat during storage at 4 °C for 4 days [59]. The casein wrapped sample shows less rancidity and better sensory property as compared to uncoated sample. Carbohydrate based edible films and coatings can also act as antimicrobial packaging when developed CS incorporated with lauric arginate ester, and nisin system is applied on RTE deli turkey meat for controlling foodborne illness, generated from post processing contamination [60]. Besides, the developed solution when applied over poly(lactic acid) (PLA) film for packaging of the final product shows inhibited growth of *Listeria innocua* similar to the direct coating onto the product. Therefore, the CS based solution can be acted as effective antimicrobial packaging in both film and coating based packaging. Besides, minimizing the microbial growth and extending the shelf life, edible films and coatings is able to reduce degradation of fried oil, while, frying methylcellulose (MC) and hydroxypropyl methylcellulose (HPMC) based edible films coated chicken strips hinder the migration of moisture and acetic acid into the frying oil from chicken strips [61]. The coating also improves the fried food quality by enhancing fat and reducing moisture content.

14.6 Advances in Edible Films and Coating for Sea Foods and Fish Products

14.6.1 Nutritional Benefits, Spoilage and Preservation Approaches of Fish Products

Fish products are a rich source of animal protein, essential amino acids (EEAs), omega-3 fatty acids, vitamins (riboflavin and vitamin D), minerals (iron, iodine, magnesium, potassium, zinc, sodium, calcium, chlorine), and others. The essential nutrients such as omega-3 fatty acids obtained from fish, and specifically from fatty fish, are a good choice to healthy heart and brain. However, the most recommended omega-3 fatty acids sources are salmon, shad, sardines, smelt, anchovies, etc. Besides, fish products are recommended to eat for several health benefits such as (1) Bone fortification, (2) Promotes eyesight, (3) Keep heart healthy, (4) Provide a healthy life, (5) Prevents cancer, (6) Provide glowing skin, (7) Boosts immune systems, (8) Acts as an antidepressant, (9) Lower cholesterol levels by lowering low density lipoproteins, (10) Prevent blood clotting, and others. Additionally, the salmon fish is considered a great source of required nutrients such as gluten-free, low-sodium, sugar-free, low in saturated fat and others, a good protein source, and contains omega-3 fatty acids. In the global markets, the available processed fish products include (1) Surimi: It is a Japanese food product and developed from white-fleshed fish; (2) Fish glue: It is prepared from several parts of fish such as bones, skin, and swim bladders; (3) Fish oil: It is a rich source of omega-3 fatty acids which has the components of eicosapentaenoic acid (EPA) and docosahexaenoic acid (DHA) which help to reduce inflammation in body; (4) Fish emulsion: Fish emulsions are produced from the leftover fluid from processed fish oil and fish meal; (5) Fish meal: The several fish based meals are prepared from whole fish, bones, etc.; (6) Fish hydrolysate, (7) Fish sauce, (8) Isinglass, (9) Tatami iwashi, and (10) others.

The fish products are susceptible to get degraded for being highly perishable, and the spoiled fish and fish products are hazardous for health which can cause serious health degradation. In this regards, the freshness of fish is commonly analyzed by general appearance (eyes, surface slime, firmness, discoloration, bell walls) and odor (gills, belly cavity, rigor mortis). The factors which cause spoilage to fish and fish products are high protein, fat and moisture content, bacterial contamination, breaking muscle tissue, careless handling, and others. Moreover, the spoilages are caused due to changes in nutritional components such as putrefaction (protein breakdown/oxidation of protein), sourness (due to producing lactic acid), rancidity, bacterial contamination, fat degradation, and others. The quality assessments of fish products are generally done by physical, subjective, organoleptic (flavor, texture, sight, etc.), chemical, and biological methods. However, the spoilage consists of three different processes such as mechanical damage (bruised flesh, burst guts, broken skin), bacterial contamination (temperature, water content, chemical substances), chemical decomposition (occurs in fat fishes such as mackerels, trout, grass carp) and enzymatic spoilage (autolysis). Additionally, the bacterial spoilage in fish products can occur at different temperatures such as (1) At chilling temperature by *Achromobacter, Flavobacterium, Pseudomonas*; (2) At higher temperature by *Micrococcus* and *Bacillus*; and (3) At atmospheric temperature by *Escherichia, sarcina, Clostridium*, Proteus. The physicochemical properties in determining fish spoilage are pH value, trimethylamine (TMA), total volatile basic nitrogen (TVBN), indole, skatole, hydrogen sulphide, volatile reducing substances (VRS), etc. Additionally, the lipid based spoilage in fish can be detected via various analysis (lipid markers) such as peroxide index, refractive index, rancidity, texture, electrical conductivity, and others. The specific spoilage organisms which cause spoilage in fish products include fish metabolites, undesirable appearance, undesirable flavor, undesirable odour, and others. The spoilages in fish products caused by several ways (1) Proteolytic microorganisms: Converts protein to amino acids, amines, and others; (2) Fermentative microorganism: Converts carbohydrates to acids, alcohols, and gases; and (3) Lipolytic microorganism: Converts fats to fatty acids and glycerol. The several preservation approaches for fish products are canning, drying, freezing, smoking, pickling, etc. However, the application of edible films and coating for improved property and product life of fish products has been discussed in the below section.

14.6.2 Effect of Edible Films and Coatings on Fish Products

The several available polysaccharides, proteins, and other bioactive agents are extensively used for improved shelf life of fish and fish products as shown in Table 14.5. Among available polysaccharide materials, CS and its nanostructured materials are mostly used in developing edible coated fish and fish products for improved quality and shelf life. In this regards, the antimicrobial enzymes as a natural biopreservative are utilized to develop antimicrobial packaging systems. In this context, the use of CS incorporated with lactoperoxidase (LPOS) system can be used for the preservation of fish, where the development of edible coating on rainbow trout fish can maintain the quality during storage life of 16 days at 4 ± 1 °C [67]. The application of edible packaging using CS and LPOS on rainbow trout help to reduce the microbial count in fish products such as *Pseudomonas fluorescens*, *Shewanella putrefaciens*, psychrotrophic, and mesophilic bacteria in comparison to uncoated products. Additionally, the application of edible coated fish products provides permissible consumable limit of total volatile basic nitrogen (TVB-N) of 22.07 mg 100 g^{-1}, whereas uncoated and CS coated rainbow trout samples provide exceeded level of TVB-N. The use of CS and CSNPs as an active edible coating on fish fingers improves the microbiological property in terms of psychrophilic bacteria, coliform bacteria, proteolytic bacteria and total bacterial count and others [68]. The use of NCS for developing edible coating on fresh silver carp fillets is more effective than CS for having better antimicrobial activity during storage period at 4 °C [69]. Moreover, the shelf life of *Huso huso* fish fillets can be

Table 14.5 Several edible films and coating materials for fish preservation

Sl. No.	Type of fish	Packaging materials	Property	References
1	Rainbow trout	CS LPOS system	Active edible coating Reduce microbial count Reduced level of TVB-N	[67]
2	Bream (Refrigerated storage)	Sodium alginate Vitamin C Tea polyphenols	Reduced chemical spoilage Increased overall sensory property Decreased microbial growth	[72]
3	Double filleted Indian oil sardine	CS	Inhibit bacterial growth Reduced oxidation of products Reduced formation of volatile bases Provide improved water holding capacity, textural properties and drip loss in fish products	[74]
4	Tilapia fillets	Gelatin Benzoic acid	Prolong shelf life Suitable for preserving tilapia fillets	[73]
5	Nile tilapia fillets	Fungal CS Pomegranate peel extract	Retard chemical spoilage Extended shelf life	[75]
6	Fish fingers	CS CSNPs	Edible coating Improved microbiological property Increased shelf life up to 6 months	[68]
7	Smoked fish	Protein sources	Monitored quality attributes Improved shelf life of 2–3 weeks	[71]
8	Silver carp fillets	CS NCS	NCS is more effective in preserving silver carp fillets NCS is more antimicrobial active	[69]
9	*Huso* fish fillets *(*Edible coating and MAP*)*	CSNPs Fennel essential oils	Improved shelf life up to 18 days Improved sensorial attributes	[70]
10	Silver carp fillets	CSNPs Orange peel extracts Pomegranate peel extract	Inhibit lipid oxidation in fish Safe preservatives for fish products	[76]

CS Chitosan, *NCS* Nanochitosan, *CSNPs* Chitosan nanoparticle, *LPOS* lactoperoxidase, *TBA* Thiobarbituric acids, *MAP* Modified atmospheric packaging

improved up to 18 days, by applying edible coating (CSNPs and fennel EO) and modified atmospheric packaging [70]. The use of CSNPs, orange or pomegranate peel extracts as an edible coating material on silver carp fillets can improve the shelf life and further beneficial for reducing lipid oxidation. Additionally, the various protein sources for edible coating of fish and fish coatings are SPI, WPI, egg white powder protein, wheat gluten, corn protein, gelatin, collagen, animal proteins from fish (rainbow trout, atlantic mackerel) [71]. The application of edible coating based on alginate, vitamin C, and tea polyphenols on bream (*Megalobrama amblycephala*) can provide improved shelf life at refrigerated storage of 4 ± 1 °C [72]. The chemical spoilage in terms of thiobarbituric acids (TBA), pH, TVB-N, water loss in bream fish can be reduced with the aid of coating materials. The edible coating of tilapia fillets using gelatin and antimicrobial agent (benzoic acid) helps to improve the shelf life at refrigeration condition, and also contains acceptable volatile basic nitrogen (VBN) after 7 days of storage [73].

14.7 Advances in Edible Films and Coating for Dairy Based Products

14.7.1 Nutritional Benefits, Spoilage and Preservation Approaches of Dairy Based Products

Dairy products are widely consumed for several health benefits such as a primary source of proteins and minerals. The nutritional components of dairy products are calcium, vitamin D, vitamin A, protein, magnesium, zinc, and others. Additionally, the intake of dairy products provides several health benefits such as (1) Healthy bones, (2) Healthy weight, (3) Reduced risk of gum diseases/strong teeth. Besides, the health benefits of dairy products include reduced heart diseases, hypertension, type 2 diabetes, stroke, and others. The available dairy products including fermented milk, paneer, yogurt, butter, cream, cheese, ice cream, custard, and others are developed from milk of mammals. The extensively eatable dairy based food products are khoya, basundi, bland, buttermilk, chaas, condensed milk, cream, ghee and others. However, there are some available alternatives for dairy products such as soya milk, nut milk, rice milk, oat milk, tofu, frozen rice deserts and others. Further, milk being a highly perishable food product has a very short shelf life, and thus, needs to process to obtain increased shelf life. The common technique to obtain improved shelf life of milk are pasteurization (high temperature short time; ultrahigh temperature, etc.), sterilization and dehydration. Besides, several milk based products may have some improved shelf life due to various processing techniques. Additionally, milk is widely preserved by several ways such as salting, dehydrating, freezing, canning, condensing, sweetened condensed milk, and others. The several types of spoilage in dairy products occur due to microbial spoilage and non-microbial spoilage. The spoilage causing microorganisms in different types of milk products are varied in different products such as (1) Raw milk: Microbes; (2) Concentrated milk: Spore forming bacteria, osmophilic fungi; (3) Cheese: Psychrotrophs, yeast, molds, coliforms and others; (4) Butter: Psychrotrophs, enzymatic degradation, others; etc. In this regards, the edible films and coating are applied to several types of cheese and paneer products for maintained quality and improved shelf life.

14.7.2 Preservation of Dairy Products Using Edible Films and Coatings

The aspects of developing edible coating on cheese products include (1) Increased shelf life via oxidation protection; reduced weight loss, prevent microbiological spoilage; (2) Sustainability for using edible packaging; (3) Protection against light, pH, gaseous components; (4) Acts as a carrier of bioactive components with antioxidant property, antimicrobial property etc. [77]. In Table 14.6, use of edible coating and films to some of the dairy products have been listed with their characteristics attributes. The several polysaccharides such as CS, galactomannan, agar in combination with other components such as plasticizers and oils are used to develop edible films and coatings on dairy products [78]. The polysaccharide coatings are a good alternative against synthetic coating materials for cheese, where tailored permeability in terms of oxygen, water vapour and carbon dioxide. The edible coating on several types of cheese such as Emmental, Saloio, Mozzarella, regional, Ras, and others are developed using CS. The cheese properties and shelf life can be tailored-made by reduced rate of water loss, maintained texture and using an effective edible coating material. The use of CS as an edible coating material on ras cheese is beneficial to develop high quality cheese with enhanced organoleptic properties and reduced microbial count [79]. The application of edible coating on "regional" cheese using galactomannan and CS provide improved shelf life with reduced gas exchange rate in terms of oxygen and carbon dioxide. Additionally, the several factors of "regional" cheese in terms of appearance, weight, moisture loss can be maintained through developing edible coating [80]. Interestingly, the storage temperature of cheese can significantly affect the attributes of cheese such as texture, color, microbial growth, moisture loss, and others. The galactomannan based edible coating on cheese has gas exchange rates between 0.195 to 0.635 mL kg^{-1} h^{-1} and 0.125 to 0.9 mL kg^{-1} h^{-1} respectively in terms of O_2 and carbon dioxide (CO_2). Moreover, the shelf life of paneer can be enhanced up to 13 days from 5 to 6 days using edible films of sodium alginate, cinnamon EOs, and calcium [81]. Paneer is a kind of highly perishable dairy product, where the quality attributes are easily degraded due to microbial attack and environmental conditions. In this regards, the several quality attributes of paneer are maintained with increased shelf life by the aid of edible films. The development of edible coating on single baked Mustafakemalpasa sweet using κ-carrageenan, CS, corn zein, and whey protein concentrate (WPC) can provide tailored attributes, where, the coated food products are further stored in polystyrene (PS) bags (storage temperature: 20 ± 1 °C) and shelf life can be extended to 10 days when coated with corn zein or WPC [82]. The storability of saloio cheese can be improved using edible films of CS and natamycin, where the physicochemical and microbial properties can be tailored and further, 1.1 log (CFU g^{-1}) of moulds/yeast counts can be decreased in comparison to uncoated saloio cheese [83]. The development of edible coating using WPI, glycerol, guar gum, sunflower oil and tween 20 in combination with antimicrobial components (natamycin and lactic acid; natamycin

Table 14.6 Several edible films and coating materials for dairy products

Sl. No.	Dairy products	Packaging materials	Property	References
1	Regional cheese	Galactomannan CS	Provide improved shelf life Reduced gas exchange rate Improved weight Improved appearance	[80]
2	Ras cheese	CS	CS (2%) provide high quality cheese Enhanced organoleptic attributes of cheese	[79]
3	Cheese	WPI Glycerol Guar gum Sunflower oil Tween 20 Antimicrobial components	Best sensory property with natamycin and lactic acid based edible coating Tailored microbiological property	[84]
4	Paneer	WPC Iron salts	Mineral fortification Enriched nutritional quality	[86]
5	Paneer	Sodium alginate Glycerol Calcium chloride CEOs Tween 80	Enhanced shelf life to 13 days Provide antioxidant activity Improved antimicrobial activity Maintained quality	[81]
6	Single baked Mustafakemalpasa sweet	Corn zein WPC	Increased shelf life from 3 to 10 days No significant loss in organoleptic properties	[82]
7	Saloio cheese	CS Natamycin	Improved storability Decreased yeast and mould counts (of 1.1 log (CFU g^{-1})) after 27 days of storage compared to uncoated sample	[83]
8	Bod Ljong cheese	CS Starch Glycerol Antimicrobial substances	Provide improved shelf life Antimicrobial components inhibit growth of microorganism	[85]

CS Chitosan, *WPC* Whey protein concentrate, *WPI* Whey protein isolate, *CEOs* Cinnamon essential oils and lactic acid; natamycin and chitooligisaccharide) provide improved property to cheese, where the best sensory property is obtained with natamycin and lactic acid incorporated edible coating [84]. Additionally, the shelf life of Bod Ljong cheese can be enhanced via application of edible coating of CS, starch, antimicrobial substances such as *Cornus officinalis* fruit extract and pine needle EOs [85].

14.8 Advances in Edible Films and Coating for Bakery Products

14.8.1 Available Bakery Products, and Strategies to Obtain Improved Property

The bakery products are a kind of cereal based products, comprising various proportions of major constituents of carbohydrates, proteins, insoluble dietary fiber, lipids, vitamins and minerals. The researchers and food industries are trying to improve the nutritional aspects of bakery products by fortifying with several nutrients for increased demands of healthier food products. Additionally, the bakery products including breakfast cereals, snacks, biscuits, cakes, muffins, are widely eaten by consumers for the crispy texture. However, the environmental conditions such as high moisture may change the texture of bakery products, and further, food-spoilage microorganism may also cause spoilage to bakery products. In this regards, the improved texture and modified attributes in bakery products can be obtained via several approaches. Further, fortification of bakery products is an another approach to modified the texture and acceptability. The available fortified bakery food products include vitamin fortified bread, mineral fortified bread, iron fortified bakery products, etc. The functional bread products are also developed using edible films and coatings as detailed in the below section.

14.8.2 Application of Edible Films and Coating for Improved Attributes of Bakery Products

The use of edible films and coating for improved attributes of bakery products has been represented in Table 14.7. The functional bakery products are developed via a new strategic way where probiotic edible films containing whey protein and sodium alginate are applied on the crust of pre-baked bread [87]. Additionally, the developed bakery products (30–40 g bread slices) with probiotic edible films can deliver the WHO recommended viable probiotic cell count to human host such as 7.57–8.98 and 6.55–6.91 log cfu/portion before and after in vitro digestion, respectively. The functional bread can be developed by incorporating microencapsulates of probiotics and starch coatings, where the physicochemical properties and sensory

Table 14.7 Several edible films and coating materials for bakery products

Sl. No.	Packaging material	Food products	Attributes	References
1	**Probiotic edible films** ALG WPC Probiotic (*Lactobacillus rhamnosus* GG)	Bakery products	Functional bakery product Inclusion of Probiotic in bakery products Whey proteins provide reduced *L. rhamnosus* GG viability losses during drying and losses.	[87]
2	Polysaccharide Lipid	Dry bakery product Crackers	Enhanced shelf life	[93]
3	Microencapsulates of *Lactobacillus acidophilus* Starch coatings	Bread	Provide functional bread Improved sensory property Increased water activity Reduced failure force	[88]
4	Waxy corn Starch Gum (Gellan gum and sodium alginate)	Rice cakes	Retard retrogradation during storage time Preserve water holding capacity Maintain bread texture	[90]
5	Triticale edible film	Muffins	Maintained quality Preserve appearance Edible films provide softer texture	[91]
6	Edible films MC Nanoemulsion of clove bud and Oregano EOs	Sliced bread	Enhanced shelf life EO reduce the yeast and mould count in sliced bread for 15 days	[89]
7	Transglutaminase-crosslinked whey protein/pectin films	Baked foods "Taralli" biscuits	Acts as a water barrier coating Prevents food matrix conversion in biscuits Prevent moisture absorption by baked products	[92]
8	Synbiotic edible films Konjac glucomannan Probiotic Prebiotic	Bread buns	Functional food products Tunable water vapour permeability	[94]

MC Methylcellulose, *EO* Essential oil, *ALG* Alginate, *WPC* Whey protein concentrate

properties can be modified [88]. The microencapsulated probiotics can be applied to the bread surface via edible coating or films, where, the probiotics can sustain in functional bread during baking and storage time. Additionally, the shelf life of sliced bread can be improved by applying edible films developed from MC, and nanoemulsions of clove bud and oregano EOs, where the EOs can help to reduce the yeast and molds counts for 15 days due to the antimicrobial effect [89]. Further, the edible coating based on blends of waxy corn starch and gum (gellan gum and sodium alginate) are reported to be applied on rice cake to retard retrogradation, improved sensory property, and water holding capacity and maintained cake texture [90]. Additionally, the application of triticale based edible films to muffin can provide improved appearance, freshness, and softer texture [91]. The edible coating to muffins can retard staling, with increased shelf life and maintained quality. The use of edible films developed from transglutaminase-crosslinked whey protein/pectin films can prevent moisture absorption in baked products such as biscuits (taralli) [92]. The application of edible coating on dry bakery products such as crackers provide improved shelf life [93]. Thus the films can be used as a water barrier coating for providing improved texture, and maintained property during storage life.

14.9 Advances in Edible Films and Coating for Fried Products

14.9.1 Available Products and Modified Attributes of Fried Products

The frying is considered as the cooking of food in oils or fats for improved texture and customer acceptability. The worldwide popular fried products include fish fries, chicken strips, breaded cutlet, pakora, tempura, fried dough, doughnuts, fried fish, fried chicken, fried potato, mozzarella sticks, deep fried pizza, deep fried turkey, and others. However, the oil uptake ratio and moisture retention property in fried food products need to be controlled as fried food products have high calories, which may cause health degradations. The fried food products may raise the low density lipoprotein in body, which is a bad cholesterol in our body. In this regards, the application of edible coatings on fried food products can reduce the oil uptake with improve product life as detailed in the below section.

14.9.2 Application of Edible Packaging for Improved Attributes of Fried Products

The application of edible films and coatings has many benefits in fried food products such as deep fat fried products provide reduced oil uptake due to edible coating applications [95]. The edible coatings on meat and fish products are extensively used as an oil barrier and reduced fat uptake to offer healthier fried products as represented in Table 14.8. In this regards, the hydrocolloids have thermogelling properties which can be strategically utilized to develop healthier fried food products with advantages

Table 14.8 Several edible films and coating materials for fried products

Sl. No.	Packaging material	Food products	Attributes	References
1	Deep-fat frying potato strips Dough discs	MC HPMC Sorbitol	40.6% reduction of oil uptake in fried potato strips 35.2% reduction of oil uptake in dough discs	[96]
2	Frying of coated chicken breasts	MC	Inclusion of 2.5% coating solution provide reduced frying and coating loss Frying of coated chicken breast with 2.5% MC provides higher cooking yield	[97]
3	Fried potato chips	CMC SPI	Reduced fat content Improved sensory property SPI is more effective than CMC	[98]
4	Deep fat fried batter of pork cutlet	Cellulose derivatives	Reduced oil uptake Increased moisture retention HPMC is more effective in providing improved property than MC	[99]
5	Deep fat frying of starchy products (Mashed potato balls of 47 mm)	Corn zein HPMC MC	MC has more effectiveness in reducing fat uptake than HPMC and corn zein based edible coating. Reduced oil uptake Reduced moisture loss	[101]
6	Deep-fat frying of shrimp (Active edible coating)	Basil seed gum Thymol oil	Coating provides 34.50% reduction in oil uptake compared to uncoated shrimp Coating provides 13.9% reduction in moisture loss compared to uncoated shrimp	[100]
7	Vacuum fried banana chips	Guar gum Xanthan gum	Reduced oil absorption of banana chips	[102]
8	Deep fat frying of cowpea paste	HPMC MC	Reduced oil uptake Soggy appearance with less brown color	[103]
9	Fried breaded product	HPMC MC Wheat gluten	Improved property in terms of reduced moisture content and reduced oil uptake The approach is beneficial for consumers and food industries	[104]

of less fat content, better nutritional content, improved sensory content, etc. The polysaccharide materials are widely used to deliver improved attributes of food products such as reduced oil uptake ratio. Some of the cellulose and cellulose derivatives are MC, carboxymethyl cellulose (CMC), HPMC, and others, which are focused to be used in fried products. The uses of MC based edible coatings are better for deep fat frying potato strips and dough discs than HPMC [96]. Additionally, there can be obtained reduced oil uptake ratio up to 40.6 and 35.2% in potato strips and dough discs, respectively. The frying of coated chicken breasts with 2.5% MC provide higher cooking yield with reduced frying loss [97]. The SPI based edible films are more effective than CMC for reducing fat uptake during frying of potato pellet chips [98]. Additionally, application of CMC and SPI based coating films for deep-fat frying of potato pellet chips provide improved sensory property and reduced oil uptake. However, the use of HPMC is more beneficial than MC in offering reduced oil uptake, moisture retaining property and sensory property (crispiness, hardness, color) in deep fat fried batter of pork cutlet [99]. Moreover, the application of edible coating of basil seed gum and thymol on shrimp provide reduced oil uptake and moisture loss by 34.50 and 13.9%, respectively in comparison to the uncoated shrimp sample [100]. The several improved attributes of shrimp can be improved via frying based processing such as overall acceptability, texture, chewiness, juiciness and others. The development of edible coating on mashed potato balls (47 mm) using corn zein, HPMC, and MC provides reduced fat uptake by 59, 61.4 and 83.6%, respectively during frying [101]. The inclusion of guar gum/xanthan gum based edible coating and post frying centrifuge step improve the quality of banana chips (vacuum fried) such as reduced oil absorption property, which further provide improved shelf life [102]. Interestingly, the oil uptake during akara production can also be reduced with improved appearance by developing edible coatings from HPMC, MC, corn zein, and amylose [103]. The application of edible coating on fried breaded products using HPMC, MC, and wheat gluten are beneficial for consumers and food industries as the coating helps to reduce the moisture uptake during deep frying [104].

14.10 Conclusion

The nutritious high perishable food products can be preserved using edible food packaging technology. In this regards, the meat and meat products, fish and fish products, dairy products contain proteins, vitamins and others, which helps in lowering the blood pressure and further, help to reduce the risk of heart attack. The perishable food products have very low shelf life, and can be preserved using edible films and coatings, without compromising the nutritional quality. For instance, nanotechnology aided edible food packaging plays a vital role in order to improve the inherent characteristics of biopolymers by incorporating effective nano-dimensional bioactive compounds for further improved quality of food products.

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Chapter 15
Characterization Techniques for Nanotechnology Assisted Edible Food Packaging

Deepshikha Das, Tabli Ghosh, and Vimal Katiyar

15.1 Introduction

The nanotechnology is a branch of science which deals with the nanoscale (range 1–100 nm) materials and is used in enhancing the attributes of microscopic and macroscopic materials. It is a booming area of technological advancement, where novel techniques and methods are used to fabricate nanodevices and nanobiomaterials [1]. These kind of nano-based materials are initially applied for developing microscopic devices in the electronic industries. Additionally, the nanostructure materials providing several noteworthy properties such as high surface-area, high strength, better diffusivity and catalytic properties, etc. are targeted for various multifaceted advanced application. In this regards, the development in the area of nanocrystalline materials are also being explored for edible food packaging application in relation to mechanical properties such as ductility and high strength, physicochemical, biological properties (biocompatibility and biodegradability) and microbiological properties. The different grain sizes of nanomaterials have been used by the physicists and chemists to produce novel materials [2]. The property of nanomaterials which are useful in food packaging industry depends on various factors such as particle-size, surface properties, improved bioavailability, dispersibility, water permeability, thermal stability and its antimicrobial property [3]. These properties determine the effect of nanomaterials upon human health and therefore it is utmost essential for proper characterization of the nanomaterials to understand the interaction of these with the environment and ensure their safety usage in the field of edible food systems [4]. The field of nanoscience aims to

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develop novel nanostructures in different forms such as nanocomposites, nanoemulsions, nanocapsules, nanospheres, and nanofibers in order to enhance the flavour, texture, shelf-life, and the colour of the food [5]. Recent reports and researches reveal that the use of nanomaterials will lead to a newer and smarter form of packaging system ensuring the safety aspect of the food materials [5].The various techniques involve for the characterisation of the nanomaterials are scanning electron microscopy (SEM), transmission electron microscopy (TEM), high resolution transmission electron microscopy (HRTEM), atomic force microscopy (AFM), scanning tunnelling microscopy (STM) that provide the size and shape of the nanomaterials. The interaction of these materials with the electromagnetic radiation helps to know about the concentration, size, crystal structure, and morphological behaviour of the nanomaterial as a function of its wavelength. This can be achieved by Raman spectroscopy, ultraviolet visible (UV-Vis) spectroscopy, attenuated total reflectance Fourier transform infrared spectroscopy (ATR-FTIR), dynamic light scattering spectroscopy (DLS), Zeta-potential spectroscopy, and X-ray photoelectron spectroscopy (XPS). There are several other techniques used for the analysis of specific materials (magnetic nanomaterial and nanogels) are Vibrating sample magnetometry (VSM), X-ray magnetic circular dichroism, etc. These are related to both physical and structural characterization [6].

15.2 Characterization of Nanostructured Materials

The characterization of nanostructure materials mainly depends on the size and shape, surface chemistry, aspect ratio, hydrophobicity, and other factors. The properties of the nanomaterials vary according to the changes in the sizes of the matter. These are basically layered films and possess different physical and chemical properties as compared to the macrostructured materials with similar chemical fabric. These kind of materials possess vivid potential to create novel inventions with well-designed shapes, structures and crystalline phases leading to the advancement in many areas of sustainable technologies. The different characterization techniques described for the analysis of nanomaterials are quite challenging being interdisciplinary in nature [6].

15.2.1 X-Ray Diffraction

It is a non-destructive technique used for determining the atomic and molecular design of any material. It is the most popular and widely utilized technique for the characterization of the NPs. It gives us a better understanding about the crystalline property of any material. It helps to gain information on the crystalline grain size, nature of the crystalline phase, unit cell parameters, and crystalline grain size. In this technique, the specimen is generally vacuum dried before it is subjected to analysis. The peak position and intensity of the particles can be determined by comparing with the standard patterns mentioned by the Joint Committee on Powder Diffraction Standards (JCPDS) database [6]. The shape and size variations of any particle is effective in determining the peak intensities of a crystalline material. Upadhyay et al. in the year 2016 reported the structural and magnetic properties of chemically synthesized magnetite NPs (ferric oxide Fe_3O_4) [7], where the X-ray line broadening results showed that the average crystallite size of the magnetic nanoparticles (NPs) are in the range of 9-53 nm and average crystallite size affects the magnetic parameter of the samples. The magnetic parameters of the analysed sample show greater dependence upon the average crystalline size of the material. Besides, Yan and group reported that AuNPs showed higher intensity corresponding to (111) plane than the (220) plane [8]. Additionally, Li et al. prepared CuTe nanostructures with different shapes, where, the relative XRD intensities varied depending upon the shapes of the nanomaterial [9]. In this way, XRD analysis is used to evaluate the chemical composition, and phase identification of crystalline nanomaterials and their modified/functionalized forms via identifying characteristics diffraction patterns.

15.2.2 X-Ray Absorption Spectroscopy

This is a widely used technique for determining the absorption co-efficient of a material which is a function of energy. Here, element transitions of direct energy levels occur from ground-state to the excited-state due to absorption of energy. Each element corresponds to the binding energy of its electrons making itself more selective in nature. In X-ray Absorption Spectroscopy(XAS), generally synchrotrons are used for acquiring the spectral absorption. There are other two methods of absorption that predicts the structural information of the elements. The extended x-ray absorption fine structure (EXAFS) is the most sensitive technique that studies the structural absorption, where the energies are considered greater than the intensity level required for electron release. It also tells about the information regarding the chemical nature of the element by reporting numbers, ligands and nearby atoms from the absorbing element. The x-ray absorption near edge structure (XANES) reports the density of partially filled electronic states of the metal site considering the excitation energy of an inner shell permitted by dipole interaction rules [10]. Many researchers reported XAS to investigate interactions within metals in order to examine several structural changes. Heinz and group (2016) used the HRTEM, XRD, EXAFS, and optical absorption spectra to find out the correlations between the structural and plasmonic properties of silver (Ag) atoms and their NPs aggregates [11]. The EXAFS of Ag K-edge spectra study indicated the atomic structure of Ag-Ag and Ag-O bonds, averaged over neutral and ionic states of Ag [11]. Pugsley et al. (2011) reported the formation of nano-Ge (nano germanium) particles by reaction between dissolved $GeCl_4$ (Ge^{4+}) and suspended Mg_2Ge (Ge^{4-}) in a diglyme solvent. Here *in situ* XAS have been used to study the kinetics mechanism of the reaction. The EXAFS and TEM analysis can provide the information on the formation of nano-GeO_2 particles. The EXAFS can also reveal the Ge-Ge distance (obtained value: 2.45 Å) by comparing it with the XRD analysis [12]. Chen and his group synthesized the cubic germanium oxide (GeO_2) decorated with sodium bis(2-ethylhexyl) sulfosuccinate (AOT) using the reverse micelle technique. *In situ* EXAFS have been used to examine the structural behaviour around germanium and its atoms in GeO_2NPs. Interestingly, they also observed that at higher temperature, the complete formation of GeS_2 took place due to the presence of sulphur content [13]. Ramallo-Lopez and his co-workers (2007) prepared palladium (Pd) NPs capped with *n*-alkyl thiol molecules and studied the structural and electronic behaviour of alkyl thiol-capped Pd NPs due to the sulphur-Pd interactions. The analysis with the help of XANES and EXAFS revealed the sulfidation of clusters of Pd that is caused by capping thiols, which occur both on the surface and in the bulk [14]. Pizarro et al. (2004) investigated metallo-protein with the help of XAS to evaluate the effect of the coordination charge on the absorption edge energy. The understanding of EXAFS in case of metal systems for unknown structures also provides a criterion for studying well-characterized model systems [15]. The theory of XAS also has been developed in such a manner to help us build understanding of the complicated molecules of known structures as shown by Rehr and Albers [16]. The importance of XAS over the X-ray crystallography provide information related to element structures for both powders and solution based samples. Also, these techniques are most easily applicable for ordered samples such as single crystals. These are specially used for determining structural information from multi-nuclear metal clusters which are linked with water oxidation in the photosynthetic oxygen evolving complex (OEC), such as Mn_4Ca clusters [17]. Cinco et al. (2002) developed another method where EXAFS measurements are used to gather information on both manganese (Mn) and calcium K-edges (Ca K-edges) for the OEC cluster [18]. Sharma et al. (2015) synthesized copper-oxide (CuO) NPs and Cu_2O/CuO and CuO/TiO_2 by a modified co-precipitation method. The combination of these two techniques of XANES and EXAFS evidenced the different oxide phase probing of the local electronic and atomic structure of these samples. Further, XANES provides information on oxidation and local structure of iron atoms. Small angle x-ray scattering (SAXS) technique reveals the information on the size of the particles. The combination of SAXS and XANES studies also helpful to understand the formation of maghemite NPs in water both structurally and chemically [19].

15.2.3 Small Angle X-Ray Scattering

It is an analytical technique used for the measurement of intensities of x-rays at smaller scattering angles (0.1–5°). SAXS is generally used for the determination of particle size distribution and shape. The schematic presentation of in situ setup employed for real time SAXS/Wide Angle X-Ray Scattering(WAXS)/UV-Vis measurements during the formation of Au nanoparticles (NPs) has been represented in Fig. 15.1. The SAXS analyses for size are statistically quite average than the TEM analysis for size. It is a low resolution based technique because of which in certain investigations, XRD or any other electron diffraction techniques becomes necessary for NPs characterizations. Wang et al. (2008) prepared platinum (Pt) NPs coated with poly(vinylpyrrolidone) (PVP), where they found size of the NPs obtained by SAXS are bigger in size as compared to the images obtained by TEM analysis. This is due to the presence of PVP coating that showed different scattering intensity. SAXS is quite sensitive to the fluctuation of size due to electron density around any particle whereas, XRD is sensitive towards the wider size region. SAXS technique provides the actual particle size while the crystallite size is yielded by XRD. It is important to note that the different size values of SAXS and XRD is basically due to different growth rate of NPs during thermal treatment [20]. Singh et al. (2011) studied the localized surface plasmon resonance (LSPR) behaviour in case of aqueous sols of polyvinyl alcohol (PVA) stabilized AgNPs. SAXS study enabled more quantifiable learning between the LSPR behaviour and the aggregation phenomenon of the correlations. The structure and property relationship is also much feasible since both applies scattering phenomena. Moreover, the size range of these NPs as shown by LSPR is quite similar when compared to SAXS analysis [21]. Bulavin and co-workers (2016) demonstrated a combined approach using SAXS, UV-Vis spectroscopy and QELS (quasi elastic light scattering) to characterize Ag sols in polymer matrices. The SAXS analysis exhibited monomodal scattering size distribution along with QELS and UV-Vis, which showed multimodal particle-size distribution. This is because SAXS uses small x-ray intensities ranging (1–5°), whereas the QELS and UV-Vis employs higher intensity angles with large particles ranging within 30-60 nm. There are some particles that couldn't be analysed as the properties are beyond the detection limits of SAXS. However, all the aforementioned methods were in good concord for analysis of the particle size and polydispersity index (PDI) in the case of relatively smaller size particles [22]. Generally, SAXS helps us to gather information regarding the concentration and size of NPs simultaneously with respect to time function. Liang et al. (2012) studied the process of formation of mesostructured (platinum-ruthenium) Pt-Ru NPs which was electrochemically reduced from their metallic salts and implanted in a microemulsion type of lyotropic liquid crystalline template (MLLC) [23]. A process of comprehensive evolution from metallic precursors to subsequent atom reduction, mesostructure formation and NPs aggregates formation have been studied by using *in situ* XRD, SAXS and XANES technique. Chen et al. developed a process in order to study the nucleation and growth kinetics of AuNPs as a function of concentration, temperature, ligand ratio and solvent type using SAXS, WAXS and UV–Vis spectra techniques [24]. A schematic representation of this has been shown in the Fig. 15.1.

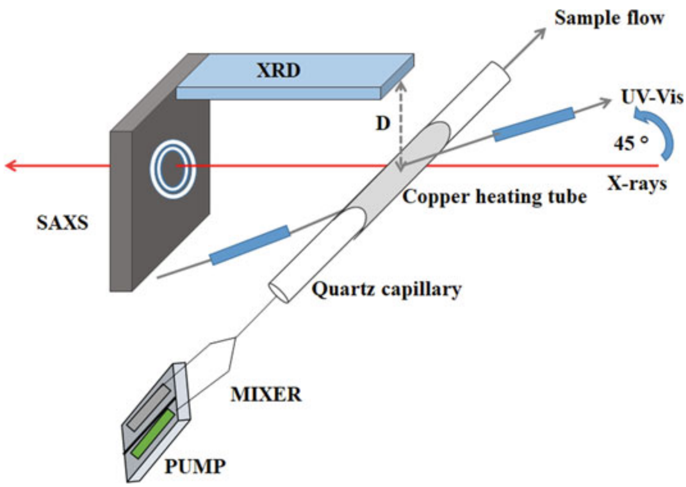

Fig. 15.1 Schematic presentation of SAXS/WAXS/UV–vis measurements during the formation of AuNPs

15.2.4 X-Ray Photoelectron Spectroscopy

It is a surface-spectroscopy analytical technique used for the measurement of elemental composition of the materials. This technique is used for the analysis of nanomaterials and provides information on the nanoscale surfaces. It works under the principle of photoelectric effect [6]. Here, the bombardment of electrons on the metal targets such as aluminium (Al) and magnesium (Mg) targets takes place in order to generate x-rays [25]. It is a technique that is used to explain the chemical analysis of any element, its elemental composition, electronic structure, oxidation states, ligand exchange interaction and surface functionalization of nanomaterials. The x-rays are entirely incident and absorbed by an atom of the material. The electrons that are ejected from the sample depend upon the binding energy of the core electrons of the atoms. If the photon energy is more than the binding energy, the electrons emit from the surface of the elemental surface. The kinetic energies of the ejected electrons can identify the elemental structure of the materials. The XPS technique is quite sensitive to the chemical framework of the atoms as it detects the chemical nature of the elements [5]. In a study related to core-shell NPs, Shard demonstrated direct and empirical method to interpret the shell thickness of spherical core-shell NPs with the help of XPS data [26]. Similarly, Sarma et al. (2013) reviewed the benefit of using XPS in order to study about the internal structure of NPs. While comparing it with TEM and TEM/Electron Energy Loss Spectroscopy (EELS) that uses lateral spatial for characterizing elements which acts perpendicularly to the probing electron beam, whereas, XPS probes act along the direction of electron beam [27]. Barros et al. (2015) used XPS technique to study the surface framework of NPs and also the oxidation of copper species in order to interpret the degradation of the Amaranth food dye as heterogeneous catalyst by electro-Fenton process. The copper spinel oxides reveal that the copper species are excluded to the NPs surfaces [28]. Also, XPS is a powerful technique to study the proteins and peptides. Belsey et al. (2015) used two approaches for the analysis of protein coatings on the surface of gold (Au) NPs. The XPS study helped to understand the adsorption of proteins on the Au surface. The characterization of the molecular interface was done by analysing the thickness of the NPs coatings [29]. The dielectric properties of nanomaterials can also be analysed with the use of XPS technique. In this regard-, Tunc et al. in demonstrated an easy method for controlling the charge that is developed over Au/silica NPs. The XPS analysis of these NPs helped for the facilitated detection, location and identification of the charges in a non-contact manner [30]. In another work, Prieto et al. (2012) studied the Ag, nickel (Ni) and bimettalic Ag-Ni NPs with sizes less than 35 nm acquired by derived seed-mediated growth method on transparent and conductive indium tin oxide (ITO) substrates by XPS, XRD and optical spectroscopy. The XPS analysis provides the oxidation states of Ag and Ni, where the surface of Ag NPs are not oxidized, and the Ni NPs were oxidized to Ni oxide (NiO) and Ni hydroxide ((Ni $(OH)_2$). It highlights the fact that it could also identify the amorphous species, while XRD failed to detect the peaks for NiO and $Ni(OH)_2$ [31].

15.2.5 Dynamic Light Scattering Spectroscopy

It is a technique used for the analysis of the size of NPs in colloidal suspensions or solution with size ranging from nano to submicrometer ranges. In this, a beam of monochromatic light passes through the sample solution as a laser and the fluctuations occur as a function of photon auto-correlation. The experiment requires minimum sample and its non-invasive, which provides information about the complex behaviour of concentrated polymer solutions. The NPs are dispersed in a continuous Brownian motion. The Stokes-Einstein assumptions are utilised to determine the NPs hydrodynamic diameter. The system pre-conditions the samples in which the calorimetry experiment is performed. The instrument is operated under temperature conditions of 4–60 °C [5], as represented in Fig. 15.2. It is finally connected to the software that depicts the information about the size distribution of various components in the sample. In DLS, a low concentration of the sample is required so as to avoid the multiple scattering effects [32]. The DLS technique is quite sensitive as compared to SAXS analysis. Tobler and co-workers tried to highlight the fact that DLS is much more sensitive towards aggregation of NPs in case of silica NPs(SiO_2). Both the analysis method confirmed the ionic strength and silica concentration for the NPs formation [33]. Lim et al. reviewed the characterization of magnetic NPs with the help of DLS technique. Here they discussed how various factors such as particle shape, colloidal stability and concentration affect the size distribution and colloidal stability of magnetic NPs. Also, by comparing with other sizing techniques such as TEM and AFM, they discussed both pros and cons of using DLS. The self-assembly

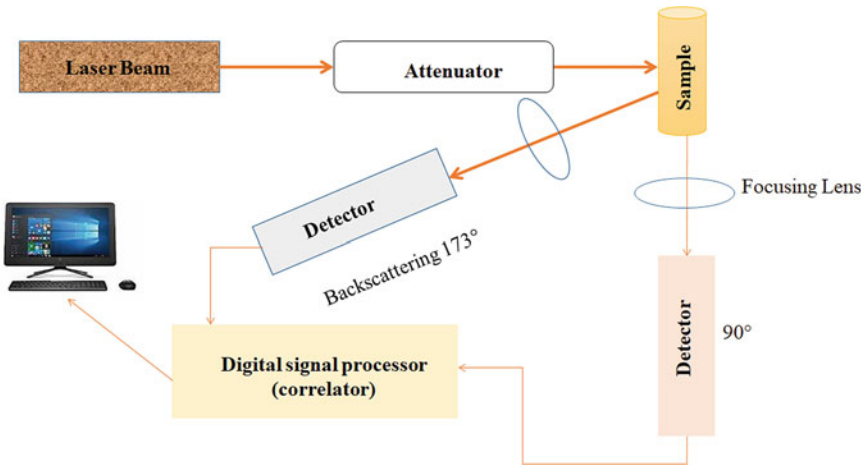

Fig. 15.2 Schematic representation showing the instrumentation of DLS

of the NPs can also be analysed by DLS [34]. The differential calorimetry technique, DLS is well known for its feasible operation for monomodal suspensions. The one limitation for the DLS is its necessity for the samples to undergo Brownian motion. Therefore, in case of polydisperse heterogeneous samples, its resolution is quite low because larger particles scatter more light. Although it measures anisotropic structures that sometimes assume spherical shaped particles, DLS requires transformative assumptions while interpreting data for polydisperse samples [15]. Additionally, the dimensions (average hydrodynamic diameter) of nanoparticles and its aggregates in aqueous suspension can measured using DLS [35]. The nanoparticles having wide size ranges are difficult to analyse by DLS as this technique is less sensitive towards smaller particles. Driskell and co-workers made a novel approach in using one-step screening method for characterizing antibody conjugated AuNPs. The advantage of using DLS as compared to the classic colorimetric technique is to determine the specificity of antibody-antigen aggregates. It also offers an important improvement in comparison to Enzyme-linked immunosorbent assay ELISA (enzyme-linked immunosorbent assay) assays for proteins [36].

Murdock et al. (2007) characterized range of NPs such as metals, metal oxides and carbon-based materials. The cell culture studies along with DLS measurements were used in order to assess the toxicological effects such as agglomeration changes due to the presence or absence of serum in cell culture media. The DLS measurements revealed that the NPs in solution necessarily do not justify their size [37].

15.2.6 Transmission Electron Microscopy

TEM is a high-resolution technique for structural and chemical analysis [36]. Here, an electron beam is transmitted through an ultra thin sized sample and interacts while passing through it. The TEM facilitates the direct imaging of the structure of the atom of the solid and the surface by collecting information about the size, crystallinity, shape of the particle. It creates a very high-resolution image of approximately0.1 nm. It is also an important tool for chemical analysis as the beam of the electron passes through a very small diameter (>3 nm) of a NPs. When the beam of the electron interacts with the sample, it helps in the determination of some scattered and unscattered electrons [31] which are detected by the detector. The preparation of a sample for TEM analysis is a complex method. The sample must be of very thin in size because the electrons which are directly transmitted through the sample is generally of size less than 150 nm. For a very high resolution, the size must be within 30 nm size range [5]. Dubiel and colleagues reported that EXAFS showed higher accuracy than HRTEM in the determination of lattice parameters in case of Ag NPs embedded in silicate glasses [38].

15.2.7 Raman Spectroscopy

It is a non-destructive chemical analysis technique that is based on the phenomenon of light scattering. In this, the intensity of the scattered light is such that one in a million of the photons will group with the molecular vibrations of the sample and emit diverse wavelengths of light. These low intensity of light when interact with the molecular vibrations results in a shift of the photon energy [39]. The shift energy comes into effect, when the laser beam of light interacts with the electron cloud of molecules [40]. The entire process depends upon the inelastic scattering of the monochromatic light rays which is known as the Raman scattering. The Stokes Raman effect is generated when a molecule gets energized from the ground state level to a virtual energy state level and then relaxes back into the vibrational excited state level [41]. The studies of superparamagnetic $NiFe_2O_4$NPs prepared by sol-gel auto-combustion method is characterized by Mossbauer, Raman and X-ray analysis. The study reports about the single-phase nanosized $NiFe_2O_4$ with average crystallite size of 9 nm showed superparamagnetic properties. The Mossbauer spectra study showed that the NPs consisted of mixed spinel structure and canted spin order at 5 K, whereas the bulk particles depicted collinearity in order with perfect inverse spinel structure. The sizes of the particles were controlled by maintaining the heat treatment temperature. The results based on X-ray diffraction, Raman and Mossbauer analysis concluded that the particles are in a single phase with this process of preparation. The significant broadening and shifting of the Raman bands of the NPs with decreasing of the particle are also observed [42]. The surface enhanced Raman spectroscopy (SERS) technique is quite sensitive analytical method for detecting or quantifying trace amounts of pesticides in raw vegetables and fruits [43].

15.2.8 Fourier Transform Infrared Spectroscopy

It is a method of analysis which is based on the measurement of the absorption of electromagnetic radiation with wavelengths ranging (4000–400 cm^{-1}). In this, the infrared light rays falls into the prism in such a manner that the molecule absorbs the IR radiation and becomes active due to the modification of the dipole moment. The spectrum is then recorded which depicts the position of the bands and specific functional groups; providing information of molecular structures and interactions [15]. This technique is an efficient tool for fingerprinting method. It is also used for analysis of nanomaterials and various other materials [5]. Shukla et al. (2003) reported FTIR analysis of ferrous-platinum (FePt) NPs to examine the surfactant bonding in presence of oleic acid and oleylamine [44]. FTIR spectroscopy is generally used to analyse multifunctional NPs and polymer samples having different groups of stretching peaks that is related to aforementioned molecules. Table 15.1 shows a selected few infrared vibration ranges for some of the most common groups generally found in case of NPs [45]. The attenuated total reflectance (ATR) technique challenges the new aspects of the infrared analysis such as sample preparation and spectral reproducibility. It aims for both solid and liquid sampling by faster sampling, reproducibility and minimising spectral variance. Shankar et al. studied the FTIR analysis for Ag nanocomposite films with polylactic acid and lignin and recorded the spectra for 32 scans per sample at a resolution of 4 cm^{-1} [46] and similarly studied for chitin nanofibril-reinforced carrageenan nanocomposite films using ATR-FTIR technique with frequency range 4000–500 cm^{-1} [47]. A combined approach of thermogravimetric analysis (TGA) technique with FTIR study on the effect of magnetic cellulose nanofibers (mgCNF) on chitosan (CS) based edible nano-coating showed the development of different gaseous components at various IR ranges within 4000–450 cm^{-1}. The appearance

Table 15.1 The infrared vibration ranges and their corresponding functional groups for NPs

Frequency range (cm^{-1})	Functional groups
2970–2950 or 2880–2860	Methyl C–H stretching (asym./sym.)
1680–1620	C=C alkenyl stretching
3130–3070	Aromatic C–H stretching
3400–3380, 3345–3325	N–H aliphatic primary amine, NH stretching
1650–1590	N–H primary amine, NH bending
3570–3200 (broad)	O–H hydroxyl group
1470–1430 or 1380–1370	Methyl C–H bending (asym./sym.)
1050	C–O stretching
1090–1020	C–N, primary amine, CN stretching
1050–990	Aliphatic phosphates (P–O–C stretching)
1365–1340 or 1200–1100	Sulfonates
800–700	Aliphatic chloro compounds, C–Cl stretching

of smaller amount of gaseous products during the analysis of mg-CNFs provided a route for the usage of melt-extrusion technique, blown film technique and injection molding in case of biodegradable films. Here, the iron mound NPs paved a way for developing sustainable materials for food packaging applications which is related to evolution of fewer greenhouse gases [4].

15.2.9 Atomic Force Microscopy

It is a microscopy technique used for creating three dimensional images of NPs surfaces at higher magnification. It was developed by Gerard Binning and Heinrich Rohrer at IBM research laboratory in the year 1986 [48]. It is designed to measure the friction, magnetism, and height of the sample. In this AFM, it is conducted between the probe and the sample as a function of their mutual separation. This method can be used to find the sample's Young's modulus. The probe is like a sharpened tip which is connected towards the end of a cantilever, made of silicon nitride. AFM can scan under different modes between the probe and the sample i.e. contact, non-contact and tapping (intermediate or oscillating mode) method [5]. The tapping mode is most common for characterizing the NPs. Additionally, there are other parameters that influence the final topological values such as tip curvature radius, and surface energy and elasticity of the NPs. A representation of AFM set up has shown in Fig. 15.3. Alternatively, non-contact method is preferred in case of sensitive sampling and can be influenced by tip-sample forces [5]. AFM technique can also be used for measuring other properties such as imaging, mechanical properties such as stiffness, electrical and conductivity properties. This technique has the advantage of not requiring surface modification or coating prior to the analysis of the sample. The topological analysis of ion-doped Y_2O_3NPs (≤ 6 nm) performed by AFM did not require any prior modification before analysis [5].

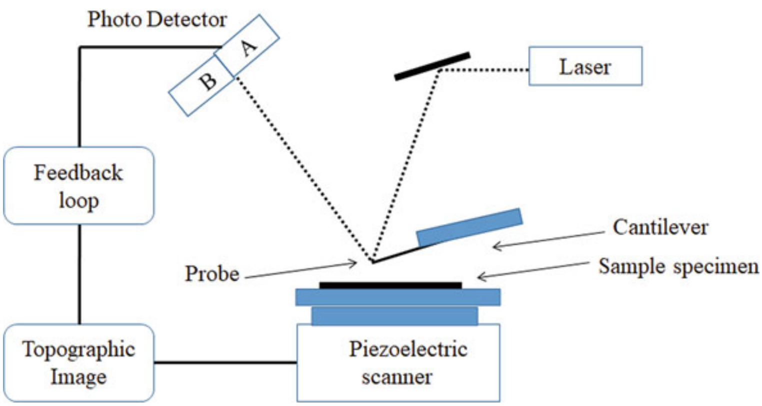

Fig. 15.3 Representation of AFM set-up

Further, AFM has been used to study the formation mechanism of uniformly patchy, hollow and rectangular shaped nanoplatelets made of polymer mixtures [49]. De Moura et al. (2012) reported AFM images of AgNPs for an antimicrobial effect of cellulose-based active food packaging, in a contact mode, by using silicon nitrate tip with a spring constant of 0.06 N/m [50]. Dhar et al. (2016) reported the fabrication of four varieties of cellulose nanocrystals (CNCs) through acid hydrolysis and its effect on the mechanical, thermal and surface properties of polylactic acid bionanocomposite films [51]. The morphological study of the different CNCs in terms of length, diameter and aspect ratio can be evaluated using FESEM and AFM micrographs.

15.2.10 Scanning Electron Microscopy

It is a widely used imaging technique employed for the characterization of nanomaterials. The SEM uses the electrons for imaging the surface of any material instead of light as involved in light microscopy. The image resolution depends on the interaction between the specimen and the electron beam. At times, due to aggregation of particles, the NPs of the sample becomes infeasible to differentiate. When an incident beam of light strikes the sample, the interaction of atoms with its surface leads to the formation of secondary electrons or back-scattered electrons and special X-rays depending upon the surface morphology and chemical framework of the sample. The back-scattered electrons reverted from the sample is responsible for the formation of an image of the sample. The SEM analysis forms a very high resolution image (HRSEM) that can capture size range up to 5 nm. The HRSEM is able to find the nanoscale features of the nanostructured material respective to their properties and application. The SEM is used to analyse the surface morphology of polymer nanocomposites, NPs, nanofibers, and nanocoating. In classical SEM, a beam of monochromatic electrons emitted is generally condensed and accelerated by accelerating anode [5]. Mazzaglia et al. (2009) studied the combined system by using field emission SEM (FESEM) and XPS measurements for AuNPs/amphiphilic cyclodextrin. The morphological information depicted the nature of interaction between (thio hexyl carbon chain) $SC6NH_2$ and (thiohexadecyl carbon chain) $SC16NH_2$ with AuNPs onto the silicon surface [52]. De Britto et al. reported the morphological and particle size study of N,N, N-trimethyl CSNPs as a vitamin carrier system [53]. The NPs are casted on the microscopy slide on top of the specimen stub, which was further Au coated in order to make it conductive and to avoid the disturbance in the sample due to the electron beam. The images revealed smooth and spherical morphology of the NPs. Additionally, Fig. 15.4 depicts the schematic representation of SEM instrument.

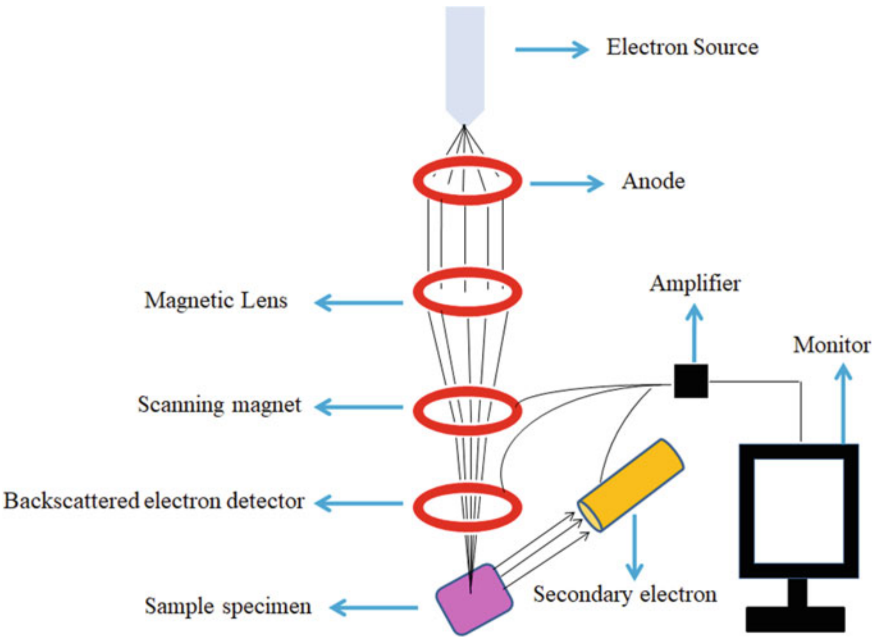

Fig. 15.4 Representation of FESEM set-up

15.2.11 Nuclear Magnetic Resonance

It is one of the techniques used for the quantitative and structural determination of nanomaterials. It is based on the phenomenon that in presence of strong magnetic field, the nuclei possess non-zero spin that causes differences between 'spin up' and 'spin-down' energy states. NMR spectroscopy is generally used to study the interactions of diamagnetic or anti-ferromagnetic NPs. This method does not employ for characterization of ferri- or ferromagnetic materials as wider saturation magnetization causes shift in the signal frequency and relaxation times. As a result of this, a prominent broadening of the signal peaks occurs showing the measurements practically infeasible to be interpreted [15]. A review on NMR techniques for noble metal NPs have been studied by Marbella and Millstone [54]. The report depicted the usage of NMR in various areas and how it will help towards molecular scale investigation of formation of NPs and its morphology in situ. It is particularly useful for analysing both formation and final characteristics of noble metallic NPs. Besides, easing to study the chemical evolution of precursors of ligands, the NMR is also used to investigate the work of capping ligands for particle shape determination [54]. The proton ^{1}H NMR chemical shift is totally sensitive towards the surrounding electronic environment; electronic structures, and bonding environment of the nucleus. This results change in neighbouring spin positions due to the changes in the chemical shift which helps to assess chirality of nanoclusters. NMR is also favourable for the measurement of hydrodynamic radius of metal NPs in relation to the other available standard techniques such as TEM and DLS. Similar to DLS, NMR spectra are used to determine particle diffusion of NPs. NMR also helps to extract diffusion coefficient of well dispersed species according to Brownian motion and the hydrodynamic size of the particles can be calculated through Stokes-Einstein equation. It is important to note that NMR analysis in case of polymer hybrid particles can only exceed size up to 100 nm and in case of metallic NPs, the dimensions include up to 1–5 nm [55]. Hens and Martins (2013) reviewed a tool for the investigation of surface chemistry of colloidal NPs using proton ^{1}H NMR analysis [56]. A study reported the analysis of AuNPs where ^{2}H NMR was used to characterize the intramolecular ligand dynamics in *d15*-(PPh3)-capped AuNPs. The ^{2}H NMR is known for its simplicity and capacity to differentiate dynamics in both amorphous and crystalline domains in case of organic compounds that are isotropically deuterons [57]. In this regard, Smith et al. used NMR to study the extent of ligand exchange between definite kinds of thiolated molecules on the surface of AuNPs [58].

15.2.12 Vibrating Sample Magnetometer

It is an analytical technique used to determine the magnetic property of materials. It is used to record the magnetization field, magnetic hysteresis loops for magnetic nanomaterials, where different parameters in the form of saturation magnetization (M_S) and the remnant magnetization (M_r) are obtained. The magnetic properties of NPs are studied as a function of its magnetic field, temperature and time. Upadhyay et al. (2016) reported the characterization of synthesized magnetic NPs using XRD, TEM, and VSM analysis. The magnetic parameters showed that the magnetic properties are depended upon the average crystallite size of the material and the ratio of the coercive field at 20 K to 300 K increased sharply with the decrease in crystallite size [7]. Ghosh et al. successfully fabricated mgCNF for edible food packaging through a single-step co-precipitation route, where the iron (Fe) particles are adsorbed onto the surface of CNF. The VSM analysis of the samples revealed that the nano-coated materials depicted magnetic hysteresis, from which it could be inferred that the there is a loss in their superparamagnetic properties and further loading of mgCNF maintained their magnetic properties [4]. Andrade and group prepared size-controlled magnetite NPs using direct reduction-precipitation method in the presence of tetramethyl ammonium hydroxide. The examined samples are found to be superparamagnetic in nature as shown by the zero coercivity and zero remanence on the magnetization loop. The saturation magnetization was found to be a linear function of the size of the NPs [59].

15.3 Characterization of Edible Films and Edible Coatings

An edible film or coating is defined as a thin, continuous layer of edible material developed from edible components on food products. The aim of edible food packaging is to produce natural biopolymer based coated films with specific properties, which may be consumed with the food. The basic function of edible film or coating is to provide a barrier against the transfer of water, gas and lipids; to serve as a carrier of food ingredients and additives (pigments, flavours and so on) and to provide mechanical and thermal stability.

15.3.1 Film Thickness

The film thickness is considered as a crucial parameter for the calculation of mechanical properties and water vapour permeability (WVP). The thickness of films and coatings depends upon the preparation method, sample quantity, and drying conditions. The effectiveness of thickness on edible films property have been reported by many authors. In this regard, the measured thickness of sodium alginate (ALG) and pectin composite based films is used to determine the physical property of the edible films, where, the thickness of the films are measured using electronic gauge with a precision of 1 μm. Simultaneously, five thickness measurements are done at the central and at the four corner of the films, where a mean value was determined and reported [60]. However, the ALG-pectin films thickness changed significantly ($p < 0.001$) from 12.5 to 26.8 μm depending upon the composition of the film-forming mixture. The higher amount of pectin can enhance increased thickness of the composite films. The increased amount of pectin can provide increased thickness of the composite films. Also, da Silva et al. (2009) analysed the amount of glycerol effect on ALG-pectin films having a thickness of 45–70 μm [61]. García et al. measured the film thickness of using SEM analysis, where a correlation was also found between the thickness of the reported chitosan-starch (CS-starch) films, which affected both barrier and the mechanical properties [62]

15.3.2 Barrier Properties

The barrier properties determine the ability of the edible films to improve the shelf life property of various food products. It helps to prevent the oxidation of films by protecting the properties of food such as odour, colour, flavour and the nutrient content. The incorporation of compounds such as essential oils (EOs) into the polymer matrix also contributes in modifying the barrier property. The gas transmission rate (GTR) is a measurement of different types of gases and it depends upon the structure of the edible film [63]. The edible biodegradable films also act as a barrier to control the transport of moisture, gaseous components (oxygen and carbon dioxide), lipids and flavours in order to prevent the deterioration of the quality of the food and simultaneously increase the shelf life of the food products. The determination of barrier property of any edible film is done by two kinds of measurements including water vapour transfer rate and oxygen transfer rate.

15.3.2.1 Water Vapour Permeability Rate (WVPR)

The WVP works as a function of temperature and vapour pressure gradient. The WVP measures the diffusion through the surface of the film and helps in the estimation of barrier property. The coated food materials such as fruits and vegetables, due to their irregular shapes and sizes make it difficult for the determination of WVP. In order to overcome these issues, García et al. in his previously reported works developed a biological replica of coated sliced carrots, where the determination of surface-area calculations were taken into account for WVP measurements. The WVPR measurements are greatly dependent on the barrier thickness, since it affects the water vapour transport through the film matrix. In case of amylase films and coatings, a lower WVP values can be obtained due to the higher amylose content, dense matrix structure, and higher degree of crystallinity than those of standard corn starch. Similarly, the addition of sunflower oil into the starch film matrix reduce the WVP of films and coatings [62]. The low rate WVP of edible films helps in the prevention of dehydration of foods in terms of packaging and coatings. The hydrophilic fraction of the film and rate of permeability depending on its hydrophilic-lipophilic ratio controls the transport of water vapour. The EOs are known to reduce the WVP of polysaccharide-based edible films due to their hydrophobic nature. Although, fewer differences could be evidenced between ALG and EOs containing films for consisting low oil content. Further, films prepared from sage oil-EOs showed a prominent reduction in WVP compared to ALG films ($p < 0.05$). Also, lipid components used in the films enhances the water vapour barrier property [64]. The water vapour barrier properties for any biopolymer films decrease upon the inclusion of greater amount of plasticizers such as glycerol, water in case of hydrophilic coatings, etc. The humidity difference also serves as a reason for altered WVP, where the increased humidity difference in the films provide improved WVP rate [64].

The water vapour transmission rate (WVTR) through the film is described as:

$$\mathrm{WVTR} = m_1/A \tag{15.1}$$

$$\mathrm{WVP} = L * \mathrm{WVTR}/(\rho_i - \rho_a) \tag{15.2}$$

where, A (m^2) is the exposed film area, ρ_i (Pa) and ρ_a (Pa) are the vapour pressures of saturated air and with 33% RH, respectively at a temperature of 25 °C. L is the average film thickness (m), $m_1$is the slope of the curve from weight loss of the film versus time.

15.3.2.2 Oxygen Transmission Rate (OTR)

The oxygen permeability (OP) is an essential property of the edible films and coatings. It is a crucial parameter for keeping quality and physiological aspects of coated fruit products during storage. The OP is checked in case of films, rather than coated products. The oxygen and carbon-dioxide barrier of the film leads to a reduction in the respiration rate of the film. This is achieved by limiting exposure to surrounding oxygen, increase carbon dioxide content, which in turn provide delayed ripening and aging with extended storage life of coated fruits [62]. Rojas-Graü and colleagues (2007), reported the incorporation of antimicrobials agents (oregano, carvacrol and lemongrass oil) into ALG based edible films, where, the antimicrobial agents has less effectiveness to the OP of the ALG based edible films [65]. Also, it has been reported that the incorporation of EOs also increases the OP due to its hydrophobic character. McHugh et al. studied the integrated property for whey-protein edible films where they found that the presence of sorbitol is much more effective as a plasticizer than glycerol for the films which showed lower oxygen permabilities. Additionally, the protein films are considered as poor moisture barrier, mostly because of its hydrophilic nature but serves as a good barrier for oxygen and carbon-dioxide [66].

15.3.3 Mechanical Properties

The food packaging industries always put an effort towards protecting the packaged food from environmental issues and mechanical stresses. The mechanical properties of edible films and coatings are generally dependent on filler-matrix interactions, physical, chemical and temperature conditions which influence the film stability and flexibility. According to the ASTM D882-91 method, the mechanical properties of the films are characterized in terms of (a) deformation at break (extension at the moment of rupture, mm), (b) percent elongation at break (EAB) (deformation divided by initial probe length and multiplying by 100%), (c) tensile strength (TS) (force at rupture divided by film cross section, MPa) and (d) elastic modulus (slope of force-deformation curve, N/mm) [62]. The tensile properties of the edible films are dependent upon the film's mechanical resistance due to cohesion, relative proportion and preparation conditions, whereas, the EAB is related to the plasticity of the film. The edible film composites, particularly in case of incorporation of citral EO into the films revealed improved mechanical resistance with respect to the neat films, making it more feasible for food wrapping applications [63]. Additionally, various kinds of sustainable bionanostructured materials are used such as nanocellulose, nanochitosan, nanostrach and inorganic nanofiller materials to obtain improved packaging properties such as mechanical property [67] In the study of mgCNF on CS based edible coating, the mechanical properties related to this revealed remarkable improvement in the TS (57.86 ± 14 MPa) and Young's modulus (2348.52 ± 276 MPa) as compared to neat CS (6.27 ± 0.7 and

462.36 ± 64 MPa, respectively) [4]. Further, the puncture force (PF) is the maximum force required to break a film with the help of a penetrating tip, which describes the film rigidity. In this regards, the films which are made from nanoemulsions showed significant lower values of PF as compared to the ALG films (11.47 ± 1.05 N) [64]. The CS also proves as a partial replacement for methylcellulose (MC) in terms of better puncture resistance and economic benefit [62].

The TS and EAB of the packaging materials are calculated as:

$$TS = F_{max}/A \tag{15.3}$$

$$EAB = L/L_0 * 100 \tag{15.4}$$

where, F_{max} is the maximum load for breaking films (N), A is the cross-sectional area of the sample (thickness * width), L_o represents the initial gage length (50 mm) of the sample, L is the final length of the film before the moment of rupture.

15.3.4 Optical Properties

The colour and the optical properties for edible films are very important in terms of food packaging applications. The optical properties include transparency and colour coordinates as L^*(lightness), a^* (red-green) and b^* (yellow-blue) values among hot buffer and alkaline soluble solid fractions. The optical properties of the edible films help in beautifying the overall appearance of the packaged food product which in turn affects the consumer acceptance. The EO based edible films ($p < 0.05$) affected L*, a*, b* parameters and difference of color (DE*) in edible films. The L* showed higher values in case of lemongrass (LG-EO) and sago (SG-EO) nanoemulsions, whereas ALG and thyme (TH-EO) showed lower values. The a^* indicated negative values with green color which significantly reduced to 0.83 ± 0.05 in the edible films of TH-EO. The coordinate b^* showed positive values with yellow colour and the most positive value (6.9 ± 0.6) was observed in TH-EO films, thus pertaining that these films showed light greenish-yellowish tone [64]. The edible films are fabricated through solution casting processes showed improved optical properties, in terms of food packaging. In general, the transparency of the packaged food materials is determined in different regions in case of ultraviolet (200–400 nm) and visible (400–700 nm) ranges.

Film Transparency. The film transparency (T) is considered to be important property for food packaging films and coatings. The whey protein isolate showed highest transparency value of 1.07 ± 0.15%, which is greater than any of the polystyrene or blended films (<0.05). The pure MC, hydroxypropyl methylcellulose (HPMC), sodium ALG films showed higher percentage of transparency values as compared to low density polyethylene, high density polyethylene, and polypropylene (PP) films (<0.05), which are commonly used in case of food packaging [68]. There are different food products which could be degraded by exposure to UV and visible range radiations. The percentage light transmission of the films is generally measured by using UV–Vis spectrophotometry. The % light transmission rate of the films is calculated by multiplying with the thickness of films (in cm) measured. In some of the food components such as proteins, lipids and fats, there may occur oxidation on exposure to light so reducing the transparency can also prove to be advantageous for packaging materials.

Color Property. The colour properties of the packaging materials in case of food products is measure generally, using coordinates including L, a*, b* and hue value. It is used to describe the quality, property and competence of the food products. Additionally, the colour parameters provide a safety tool for determining the product adulteration, the stability of pigment materials, the browning effects, etc. These factors are mostly affected by the incorporation of filler materials into the polymer matrix [4]. The colour parameters range from L = 0 (black) to L = 100 (white), −a (greenness) to +a (redness) and −b (blueness) to +b (yellowness). The total colour difference, ΔE is defined as [69]:

$$\Delta \mathrm{E} = \sqrt{(L^{*} - L)^{2} + (a^{*} - a)^{2} + (b^{*} - b)^{2}} \quad (15.5)$$

and,

$$\mathrm{YI}\ (\text{yellowness index}) = \frac{142.86\, b}{L} \quad (15.6)$$

$$\mathrm{WI}\ (\text{whiteness index}) = 100 - \sqrt{(100 - L)^{2} + a^{2} + b^{2}} \quad (15.7)$$

where, L^*, a^*, and b^* are the standard colour parameter values and L, a and b are the colour parameter values for the sample.

15.3.5 Morphological Properties

The morphological properties of the food packaging materials are necessary to gather information about the size and shape of the framework of the polymer and the filler used for the edible films and coatings. It is generally characterized by FESEM analysis. This analysis also supports various other properties such as tensile and other physical properties. Sohail et al. (2006) investigated the morphological properties of edible casein films containing zein hydrolysate. The incorporation of wax into the casein films revealed similar smooth and uniform textures as in case without wax application. It ascribed towards the hydrophobicity of zein hydrolysate which is incorporated as a filler into the casein films [70]. The morphological analysis of CS-CNF and CS-mgCNF was carried out by FESEM analysis and the different kinds of morphological distributions were observed as a fiber like dispersion and aligned particles (mgCNF) dispersion of nanofillers [4]. The FESEM studies in case of neat pea starch (PS) showed swollen starch granules structures and for neat peanut protein isolate (PPI) films showed presence of cavities and the blends of these with PPI at 40% level, revealed a flexible network type morphology of the edible films [71]. Thus, the morphology and percholation networks can be studied using FESEM micrographs.

15.3.6 Thermal Properties

The thermal property of edible film applications is also considered as one of the main attributes in wide utilization of developed edible film materials. The thermal property of the films is generally characterized by the differential scanning calorimetry technique (DSC) and thermogravimetric analysis (TGA). The DSC is a thermos analytical technique in which the heat difference is calculated between the sample and the reference. The TGA analysis details about the mass and composition of the edible films. In this technique, a sample is heated with different degradation temperature, where the weight fraction and change of mass are calculated. The analysis depends on the starting sample mass, the type and amount of NPs [6]. In the study of DSC analysis of the kefiran films with different glycerol contents showed the interaction between the polymer and the plasticizer which is necessary for food packaging applications [72]. The thermal stability of mgCNF improved considerably, where ∼17% of reduction in weight is observed, whereas the CNF degrades completely under the temperature range of 30–700 °C. The TGA analysis shows that there is an improvement in thermal stability for both CNF- and mgCNF-reinforced CS nanocoatings, where mgCNF provided more heat dimensional stability as compared to CNF- dispersed CS nanocoated films [4]. Yoo et al. (2011) studies the thermal properties of whey protein and polysaccharide edible films, where the thermal transitions of the biopolymers can be reviewed using DSC [68]. Martins et al. (2013) studied the synergistic effects between k-carrageenan (k-carr) and locust bean gum (LBG) edible films. The TGA curves depicted all events of k-carr/LBG films and found the films are stable up to 60 °C and a loss of 55% up to 319.2 °C occurs due to LBG thermal decomposition [73].

15.3.7 Physicochemical Property

The physiochemical properties of edible films and coatings include the measurement of water solubility, viscosity, moisture content, and stability of film-forming dispersion.

Water Solubility. The water solubility is an important parameter to be analysed in terms of possible applications of biopolymer edible films. The film solubility in water is defined as the ratio of water-soluble dry matter of film to that of the dissolved after dipping in the distilled water. The percentage of the total soluble matter (% TSM) of the films is calculated using the below equation.

$$\%\mathrm{TSM} = [(\text{Initial dry weight} - \text{Final dry weight})/\text{Initial dry weight}] \times 100 \quad (15.8)$$

Ghasemlou et al. (2011) found that the solubility of the films generally increases gradually with increased glycerol content. In case of glycerol-plasticized films, the addition of glycerol can diminish interactions between biopolymeric molecules, but, increase the solubility due to its hydrophilic nature, making the polymer more attracted towards water molecules [72]. Also, films developed from MC are completely soluble in water, whereas CS films provide lower water-solubility values. The composite of MC and CS provide reduced water-solubility values with increased CS concentration [62].

Viscosity. The viscosity measurement of the edible films is quite important in terms of food packaging applications. The spray coating process requires a low viscosity solution, whereas the immersion coating solution requires higher viscosity solution. In the case of nanoemulsions, viscosity is considered as one of the most relevant parameters since it showed a significant effect on EO loaded nanoemulsions' stability, which is based on the rheological study of the emulsion phases. The viscosity of pure sodium ALG solutions was found as 800 mPa s, whereas, EO type significantly ($p < 0.05$) affected viscosity of nanoemulsions (with lemon-grass EO, LG-EO incorporated sodium ALG solution has viscosity of 616 ± 62 mPa s). Since, EOs are complex in their mixing with different components, this might lead to different adsorption kinetics between ALG molecules and the oil droplets. Also, the high viscosity of nanoemulsions could affect the effectiveness of the droplets disruption because of the microfluidizer, which could lead to inclusion of sufficiently intense disruptive forces at the required pressure [64].

Moisture Content. It is defined as the weight of the moisture gained by the film per unit time (g/s). The knowledge of the relative importance of different available mechanisms controlling the moisture transfer through hygroscopic films is also very important for designing new edible films with improved and selective barrier properties. The hydrophobic nature of the films is also an important parameter to control the sensitivity of the films to moisture and it is commonly analysed by the contact angle measurements. If films are formed from raw polymeric ingredients, then it might be brittle even under low moisture conditions. So, plasticizers are used in order to increase the flexibility of the films and to reduce moisture sensitivity. In addition, a high amount of plasticizer may reduce the mechanical and barrier properties of the films. Thus, the ratio of polymer to plasticizer is important for determining the functional properties of the film. The analysis of moisture sorption is an indication of its storage stability in reduced moisture environments. The moisture content of most of the foods increases sigmoidally with respect to water, where the relationship is measured by a_w, which is the moisture sorption (or desorption) isotherm of the food. The moisture content plays an important role in order to reduce the exchange of water between the food and the environment [74].

Stability of Dispersion and its Rheological Property. The film-forming dispersions provide tailored property in the film structure, and improved film attributes. The SEM analysis plays an important role in film homogeneity, film structure, surface smoothness, and thickness. The surface of plasticized films (can be analysed by SEM) containing lipid molecules exhibited smooth surfaces and compact structure, which indicated a homogeneous dispersion of lipids within the film matrix [62]. Additionally, the drying time of films, plays a vital role in determining the arrangement of the components during the film-forming process of the edible films. In the study of magnetic cellulose nanofiber (mgCNF) dispersed CS-based edible nanocoating, the inclusion of filler material mgCNF provide the possible intercalation within the polymer matrix for increased interactions and better dispersion due to negligible agglomeration [4]. The film forming dispersions generally follows two mechanisms such as dry process and wet drying mechanism. In dry process, such as in extrusion, the polymers are heated above their glass transition temperature, Tg, under low water-content conditions. The edible film forming process generally follows the wet forming mechanism, where the polymers at first are dispersed in liquid phase and then dried. In this, the edible preformed films and coatings are formed by dipping, brushing or spraying. The rheological properties mostly depend on the design characteristics and processing conditions of film forming solutions or dispersion. Peressini et al. (2003) studied the rheological properties of edible film-forming dispersions containing corn starch, MC and glycerol using oscillatory and steady shear flow tests [75]. The combined effects of both the glycerol content and blending levels of MC with starch are evaluated in order to obtain the information relevant to the food coating. The film-forming dispersions depicted shear-thinning behaviour under steady state shear flow. The flow property evaluation can be carried out by the Herschel–Bulkley model (as shown below), in order to fit the flow data of dispersions with MC lower than 31%.

$$\tau = \tau o + K_{\gamma}^{\cdot n} \tag{15.9}$$

where,

τ(Pa) is the shear stress, $\gamma^{\cdot}(s^{-1})$ is the shear rate, τo is the apparent yield stress, K (Pa s^{-n}) and n (dimensionless)are the consistency and flow indexes, respectively.

Further, a systematic study of the rheological behaviour of the film-forming dispersions containing starch and MC have provide the effects of film formulations on the non-Newtonian behaviour and viscoelastic properties of the film-forming dispersions [75].The rheological properties of several emulsions are mainly associated with the characteristics of both the dispersed phase (volume concentration, distribution, size, shape and electrical charge of the particles), the continuous phase (viscosity, chemical composition, and concentration of electrolytes) and use of different types of surfactants and thickening agents. The use of microfluidization treatments in these emulsion appeared to be a suitable approach in the production of ALG based film-forming dispersions for targeted application [64].

15.4 Shelf Life Analysis of Food Products

As shown in Fig. 15.5, the shelf life analysis of food products is evaluated based on various properties such as physicochemical properties, respiration rates of fruits and vegetables, sensory evaluation, texture properties, microbiological study, color properties, texture properties, etc. Among available properties of food products, the sensory properties play a key role in the commercialization of any developed food products, as consumer acceptance is necessary for successful marketing of the product. However, the various food products undergo changes in nutritional quality during storage life, where the nutritional composition in food products vary from sources to sources. The fruit products with a fresh smell and having a proper size, consistency, and shape are more acceptable in comparison to other non-uniform and foul-smelling fruit products (which occurs due to environment degrading factors). In this regards, the food products should maintain the consistency, food property, and microbiologically safe for the entire product life. Additionally, the packaging materials in terms of primary and secondary packaging materials are used to carry the fruit products, where the packaging materials or food contact materials (FCMs) should be safe for food products. Additionally, the FCMs such as food manufacturing equipment, food packaging materials, food preparation/dining wares, and others are tested with different food simulants including acetic acid 3% v/v, ethanol 10% v/v, ethanol 50% v/v, vegetable oil, distilled water, and others to ensure safe consumption. Interestingly, the level of migration from FCMs to foodstuffs generally depends on several factors such as storage temperature, storage time, type of food products, type of packaging, etc., where, the migration of several components from the printing inks, varnishes, adhesives, inner bags, foodstuffs, and others may occur. In this regards, the fruits and vegetables, fish products, meat and meat products, dairy products, and others are analysed for various components to ensure safe storage and quality analysis. The materials and methods that are accompanied for analysis of shelf life of food products have been discussed in the following sections.

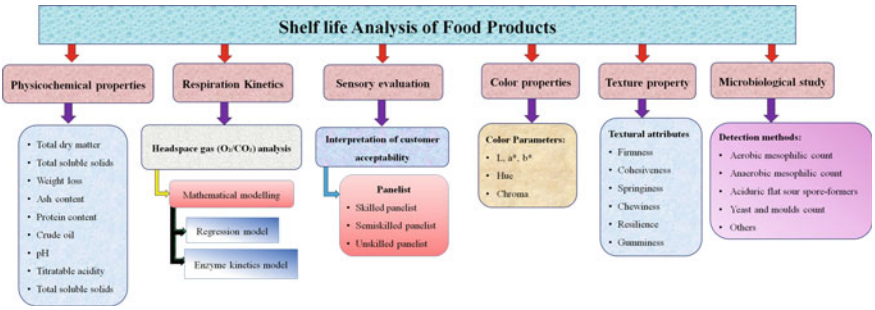

Fig. 15.5 Several property analysis of stored food products

15.4.1 Physicochemical Properties

The several physicochemical properties of food products include the analysis of moisture content, total dry matter, total soluble solids (TSS), weight loss analysis, and other properties (ash content, protein content, dietary fiber content, fat content, pH, titratable acidity ([TA], etc.). The nutritional composition of food products such as moisture content, ash, carbohydrate content, crude protein, and crude fiber is determined according to the standard methods of AOAC, 2010 [76] [**AOAC, 2010]**. The moisture content is determined following the gravimetric method, where an amount of 5 gm (W) is weighted and kept in an oven at 105 °C for 24 h, where the moisture content is determined from the percentage weight loss of the products. The dry matter of food products is generally calculated by measuring the weight difference of food products before and after drying (at 70 °C). The endpoint of after drying is selected till the constant weight of product is obtained. The pH of fruit products is determined using a pH meter. The pH of fruit products generally increases during storage due to ripening. Further, the TSS of fruit products is determined using a portable digital refractometer. The TSS content of fruit products increases during storage due to ripening. The TA of fruit products are determined by taking 250 g of well mixed juice, which is titrated against 0.1 M NaOH and the results are expressed as a percentage of malic acid. Additionally, the weight loss analysis of fruit products are measured by calculating the initial (weight at 0 days) and final (weights at sampling day) weights of the food products using a weighing balance [4, 77, 78]. The percentage weight loss of the stored food products is generally measured using the below Eq. (15.10):

$$Weight\,loss\,(\%) = \frac{m_0 - m_s}{m_0} \times 100 \tag{15.10}$$

where, m_0 and m_s are the sample weight at 0 day and sampling day, respectively.

15.4.2 Study on Respiration Kinetics

The fruits and vegetables continue respiring even after getting detached from the mother plant. The respiration in living plant products occur continuously by consuming O_2 from the surrounding and releasing CO_2 to the environment [79]. The respiration rates of plant materials have an inverse relation with the shelf life of plant materials, where increased respiration rates decrease the shelf life of commodities [80]. The respiration of living food commodities is considered as a crucial factor responsible for postharvest wastes of perishable fruits and vegetables. Further, the design of modified atmospheric packaging (MAP) and controlled atmospheric packaging (CAP) of food products depend on the respiration rate of fruit products. However, the respiration of food products is influenced by the

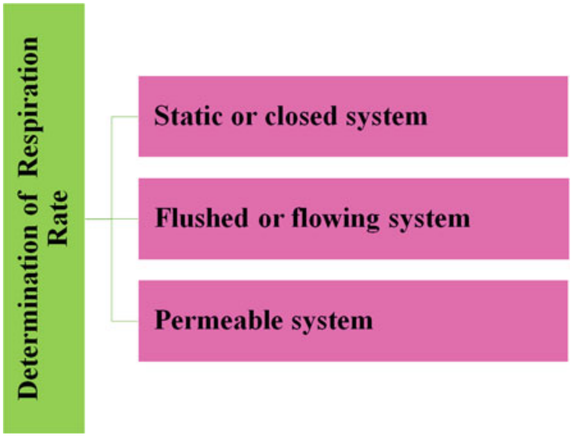

Fig. 15.6 Available methods for respiration rate measurement

storage temperature, gaseous composition, storage time, commodity type, etc. Besides respiration, respiration quotient is another factor associated with respiration of commodities, which is defined as the ratio of produced CO_2 to consumed O_2 during respiration cycles. However, as represented in Fig. 15.6, the common methods of measuring the respiration rates include static or closed system, flushed or flowing system, and permeable system [81].

Closed or Static Method for Respiration Rate Measurement. The closed or static system is a non-destructive method, time consuming, and labor consuming process, however, it can test different gaseous combinations, and the method is suitable for low respiring products [81]. In a static or closed system, the respiration study is generally conducted at various storage temperatures for a various time period (storage time depends on the storage temperatures). For this kind of respiration rate measurements, a specific amount of fruit products is stored in airtight close chamber, with ambient air as the initial gas composition as shown in Fig. 15.7. The gas chamber should have some headspace after loading of selected commodity for measuring the respiration rates. The practice of applying Vaseline white between the cover and the open top side of the container to avoid any gas leakage. Further, there is available gas septum on the top of the airtight chamber, where syringe is inserted to obtain the data set of gas concentrations in the headspace of gas chamber. A gas analyzer is generally used to analyze the O_2/CO_2 gas concentration in the headspace of the chamber. Besides, the gas chromatographs or O_2 probes are also used to determine the respiration rate of fruit products. In the closed system method, there is a need to estimate the free/gaseous volume of the closed chamber, which is difficult to estimate the same. The close chamber is generally kept inside the incubator to maintain the storage temperature of the selected food products. Additionally, the use of temperature sensors also helps to indicate the temperature of the airtight chamber.

The respiration rate of fruits and vegetables can be determined by the ratio of gas exchange amount to the amount of sample taken and storage periods. The various

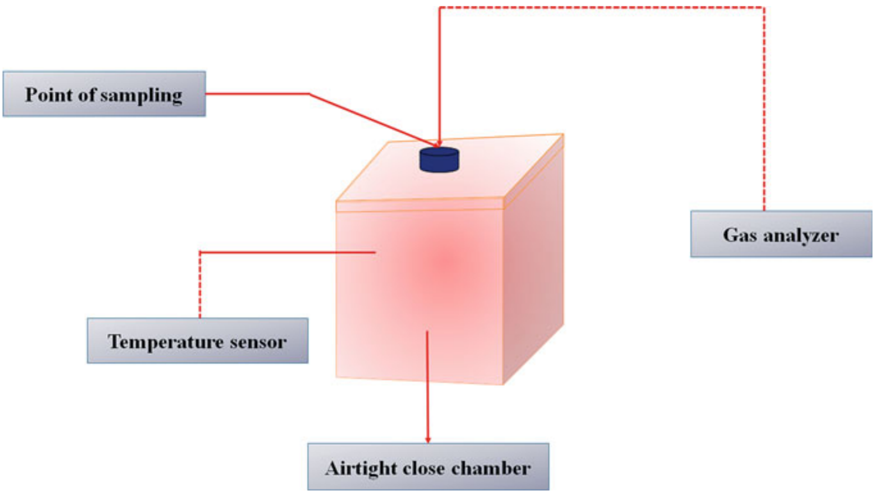

Fig. 15.7 Determination of respiration rate of fresh fruit products using airtight close chamber

dataset is collected and respiration rate in terms of O_2 and CO_2 gases are determined using the Eqs. (15.11) and (15.12).

$$RR_{O_2} = \frac{\left(GC^{i}_{O_2} - GC^{f}_{O_2}\right) \times V_f}{100 \times W \times (\Delta t)} \tag{15.11}$$

$$RR_{CO_2} = \frac{\left(GC^{f}_{CO_2} - GC^{i}_{CO_2}\right) \times V_f}{100 \times W \times (\Delta t)} \tag{15.12}$$

The linearization of Eqs. (15.11) and (15.12) are given in the Eqs. (15.13) and (15.14).

$$GC^{f}_{O_2} = GC^{i}_{O_2} + \frac{RR_{O_2} \times W}{V_f}(t - t_i) \times 100 \tag{15.13}$$

$$GC^{f}_{CO_2} = GC^{i}_{CO_2} + \frac{RR_{CO_2} \times W}{V_f}(t - t_i) \times 100 \tag{15.14}$$

where, RR_{O_2} (ml $kg^{-1}h^{-1}$) and RR_{CO_2} (ml $kg^{-1}h^{-1}$) are the respiration rates of O_2 and CO_2 gases, respectively; V_f (mL) is the free headspace volume of the jar; W (kg) is recognized as the sample weight taken for the study; $\Delta t = t - t_i(h)$ is the storage time between two consecutive readings; $GC^{i}_{O_2}$ is the initial O_2 concentration, cm^3/cm^3 of space; $GC^{f}_{O_2}$ is the O_2 concentration at time t, cm^3/cm^3 of space; $GC^{f}_{CO_2}$ is the CO_2 concentration at time t, cm^3/cm^3 of space; and $GC^{i}_{CO_2}$ is the initial CO_2 concentration, cm^3/cm^3 of space.

Mathematical Modelling of Respiration Rate of Food Products. The mathematical modelling of respiration rate of fruit products can be studied based on enzyme kinetics and the Arrhenius equation as detailed in this section [79]. The several research reports based on the respiration study and mathematical modelling of several fruit products are available such as banana [82, 83], tomato [84], litchi [85], green mature mango [80], fig [86], bhimkol [87], apple [88], fresh cut pineapple [89], guava [90], blueberry [91], etc. The above mentioned equations are utilized to determine the respiration rates of specific food products in terms of O_2 and CO_2, which are further used for the mathematical modelling of respiration rates such as regression function model and enzyme kinetics model. The mathematical models are developed to correlate the respiration rate of fruit products with different storage parameters including storage temperature, and gaseous composition. In this regards, Mahajan & Goswami have done the enzyme kinetics based mathematical modelling of apple, where the respiration rates of apple in terms of O_2 and CO_2 follow uncompetitive inhibitions [79]. Further, a detail of the available regression function model and enzyme kinetics model for respiration kinetics study have been described below.

Regression Model. The regression function is used to fit the gas concentration dataset of commodities versus storage time, where the respiration rates of the commodities are determined from the first order derivatives of the regression function [82]. The respiratory behaviour of perishable food products is studied using regression function model, where the temperature dependence of model co-efficient can be determined by curve fitting method. The respiration data sets are used to fit gaseous concentrations at various time periods using the Eqs. (15.15) and (15.16).

$$\mathrm{GC}^{f}_{\mathrm{O_2}} = \mathrm{GC}^{i}_{\mathrm{O_2}} - \left[\frac{t}{(at+b)}\right] \tag{15.15}$$

$$\mathrm{GC}^{f}_{\mathrm{CO_2}} = \mathrm{GC}^{i}_{\mathrm{CO_2}} - \left[\frac{t}{(at+b)}\right] \tag{15.16}$$

where, a, and b are the regression coefficients. The first order derivatives of Eqs. (15.15) and (15.16) are represented in Eqs. (15.17) and (15.18) and can be used to measure the change in gaseous concentrations with storage time.

$$\frac{\mathrm{dGC_{O_2}}}{\mathrm{dt}} = at(at+b)^{-2} - (at+b)^{-1} \tag{15.17}$$

$$\frac{\mathrm{dGC_{CO_2}}}{\mathrm{dt}} = -at(at+b)^{-2} + (at+b)^{-1} \tag{15.18}$$

where, $\frac{\mathrm{dGC_{O_2}}}{\mathrm{dt}}$ and $\frac{\mathrm{dGC_{CO_2}}}{\mathrm{dt}}$ are the first order derivatives of gaseous concentrations in terms of O_2 and CO_2 gasses, respectively.

Enzyme Kinetics Model. The enzyme kinetics model is based on the principles of enzyme kinetics for the respiration rate predictions of fresh produces [82]. The enzyme kinetics model generally follows uncompetitive inhibitions. As represented in Eqs. (15.19) and (15.20), the model parameters are V_m, K_m, and K_i for O_2 consumption rate and CO_2 evolution rates.

$$\mathrm{RR}_{\mathrm{O}_2} = \frac{V_{mo}\mathrm{GC}_{\mathrm{O}_2}}{K_{mo} + \left[1 + \frac{\mathrm{GC}_{\mathrm{CO}_2}}{K_{io}}\right]\mathrm{GC}_{\mathrm{O}_2}} \tag{15.19}$$

$$\mathrm{RR}_{\mathrm{CO}_2} = \frac{V_{mo}\mathrm{GC}_{\mathrm{O}_2}}{K_{mo} + \left[1 + \frac{\mathrm{GC}_{\mathrm{CO}_2}}{K_{io}}\right]\mathrm{GC}_{\mathrm{O}_2}} \tag{15.20}$$

where, V_m (ml kg^{-1}h^{-1})) is the maximum respiration rate; K_m is the dissociation constant (% O_2); K_i is the inhibition constant (%CO_2). Further, the Arrhenius relationship, which is a function of temperature, is chosen to describe the enzyme kinetics model of fresh produces. The parameters of enzyme kinetics model are determined using Arrhenius relations as represented in Eqs. (15.21) and (15.22).

$$\mathrm{RR}_{\mathrm{O}_{2,m}} = \mathrm{RR}_{\mathrm{O}_{2,p}} \times e^{\left[\left(-E_{a,\mathrm{O}_2}/R\right)\left(\frac{1}{T}-\frac{1}{T_r}\right)\right]} \tag{15.21}$$

$$\mathrm{RR}_{\mathrm{CO}_{2,m}} = \mathrm{RR}_{\mathrm{CO}_{2,p}} \times \mathrm{e}^{\left[\left(-E_{a,\mathrm{CO}_2}/R\right)\left(\frac{1}{T}-\frac{1}{T_r}\right)\right]} \tag{15.22}$$

$\mathrm{RR}_{\mathrm{O}_{2,m}}$ is model parameter of Michaelis-Menten equation; $\mathrm{RR}_{\mathrm{O}_{2,p}}$ is respiration pre-exponential factor;$\mathrm{RR}_{\mathrm{CO}_{2,m}}$ is model parameter of Michaelis-Menten equation; $\mathrm{RR}_{\mathrm{CO}_{2,p}}$ is respiration pre-exponential factor; E_{a,O_2} activation energy, kJ g^{-1} mol^{-1}; E_{a,CO_2} activation energy, kJ g^{-1} mol^{-1}; R is the universal gas constant, 8.314 kJ g^{-1} mol^{-1} K^{-1}; T is recognized as the storage temperature, K; T_r is the reference temperature, K. The linearized form of the above Eqs. (15.12) and (15.23) are shown in Eqs. (15.24) and (15.25).

$$\mathrm{In\ RR}_{\mathrm{O}_{2,m}} = -\frac{E_{a,\mathrm{O}_2}}{R}\left(\frac{1}{T} - \frac{1}{T_r}\right) + \mathrm{In\ RR}_{\mathrm{O}_{2,P}} \tag{15.23}$$

$$\mathrm{In\ RR}_{\mathrm{CO}_{2,m}} = -\frac{E_{a,\mathrm{CO}_2}}{R}\left(\frac{1}{T} - \frac{1}{T_r}\right) + \mathrm{In\ RR}_{\mathrm{CO}_{2,P}} \tag{15.24}$$

Additionally, the Eq. 15.25 is used to study the temperature dependence of model parameters of Michaelis- Menten equation.

$$R_m = R_P \exp\left[-\frac{E_a}{RT_{abs}}\right] \tag{15.25}$$

R_m is the model parametric co-efficient for Michaelis-Menten equation; R_p is the pre-exponential frequency factor; T_{abs} is the absolute temperature, K

15.4.3 Sensory Evaluation

The sensory evaluation of a product involves the analysis and interpretation of the customer acceptability of a product using the human senses. The sensory analysis is applicable for executing experimental design and statistical analysis for the evaluation of consumer acceptance for a product. The customer acceptance of a product involves the smell, taste, sight, touch and others of the products. This sensory evaluation is generally done by skilled, semi-skilled and unskilled persons and the evaluation can be classified into three sub-sections such as analytical testing, affective testing, and perception as represented in Fig. 15.8. The analytical method of sensory evaluation includes the difference and descriptive analysis. The analysis of a difference test details/measures whether a product difference is acceptable from the existing product or not. On the otherhand, the affective method of sensory evaluation includes the hedonic and preference test, where the use of hedonic test measures the degree of liking of products. The hedonic scale can detail which product is most liked among the various products. The best timings for sensory testing are 10 AM–12 noon and 3 PM–5 PM.

Additionally, as represented in Fig. 15.9, the several types of sensory testing are difference test (Paired comparison test, Duo-Trio test, Triangle test), rating test (ranking test, single sample test, two-sample difference test, multiple sample difference test, Hedonic rating test, Numerical scoring test, composite scoring test), sensitivity test (Sensitivity threshold test/dilution test) and descriptive test. The paired comparison test includes the comparison of two different samples of food product based on one attribute (such as sensory evaluation for checking the

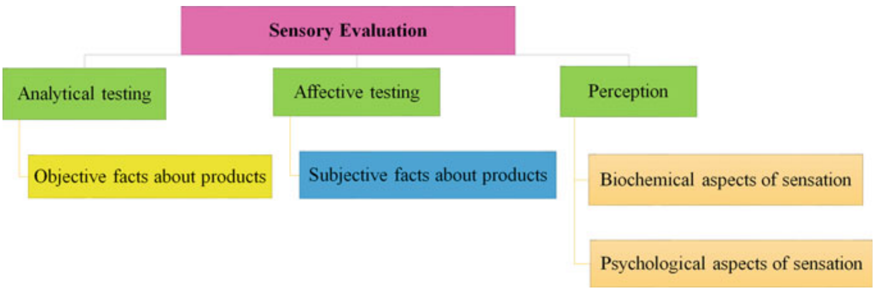

Fig. 15.8 Classification of sensory evaluation for food product testing

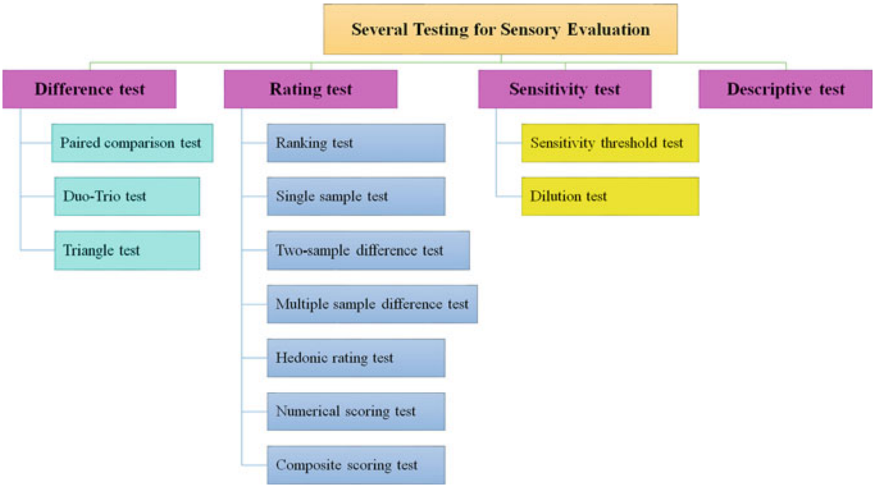

Fig. 15.9 Several Types of Sensory evaluation testing

smoothness of a sample). The duo-trio test includes the testing of three samples, where two samples are identical and one of the identical samples is taken as the control one. The panelist is supposed to find the identical sample (control) from the remaining two samples. In a triangle test, three samples are arranged in a triangle, where two of the three samples are identical. In this analysis, the panelist is asked to find the different one from the three samples. If the samples are named as "a" and "b", then the combinations of the samples arranged in a triangle are attained as either "aab" and "bba".

Moreover, the ranking test is a type of rating test, where several samples are given to the panelist with only code numbers and the panelist are asked to rank the samples based on a single attribute of a product. In the single sample test, the presence of particular quality attributes or the intensity of a quality attribute is determined by the panelist. For example, to detect the intensity (strong/moderate/strong) of off-flavor of a product and state the reason for off-flavor such as off-odor, off-taste, etc. The hedonic test based sensory evaluation is based on the 5-point (1: Dislike very much to 5: Like very much) or 9-point scale (1: Dislike very much to 9: Like very much) based testing, where the panelist put the remarks for taste, odor, color, texture, flavor, appearance. and overall acceptability of the product. Moreover, the Hedonic scale based on 9 scores is generally used for ranking food products and the scores provide the comment on the products, where 1: dislike extremely; 2: dislike very much; 3: dislike moderately; 4: dislike slightly; 5: Neither like or dislike; 6: Like slightly: 7: Like Moderately; 8: Like Very Much: 9: Like extremely. The sensory analysis of food products is generally evaluated for several attributes such as color, flavor, texture, taste, overall acceptability, and others using

Hedonic scale to find the most suitable and acceptable product among available. In this way, the sensory evaluation of food products is done to perceive, describe, and quantify the customer acceptability before commercialization.

15.4.4 Texture Property

The textural properties of food products are considered as the physical properties of food products, resulting from the macro/micro-structural properties of several food components. As discussed in the earlier section, the textural properties of food products can be analyzed and interpreted by panelist. However, the instrumentation methods of texture analysis are getting much interest due to several advantages such as rapid, economic, ease of standardization, etc. The texture properties of solid, semi-solid and liquid products are measured using the Texture analyzer instruments. The texture analysis of food products is executed via applying controlled forces (tensile/compressive forces) to the food products and the responses of the applied forces are obtained in the form of deformations, forces and time. The food texture is a very critical factor in food products to analyze the quality of food products for its overall acceptability. Among the several textural properties, firmness or hardness is one of the most essential properties for food products for determining the food quality. The firmness is a key parameter for fruits and vegetables. On the otherhand, crunchy food products should maintain the required crispiness for consumer acceptance. The other textural attributes are cohesiveness, adhesiveness, springiness, etc. The attributes for texture are moistness, oiliness, flakiness, dryness, crunchiness, etc. However, the textural attributes of food products include several characteristics such as (i) Mechanical characteristics: Primary parameters (hardness, cohesiveness, elasticity, viscosity, adhesiveness), secondary parameters (brittleness, chewiness, gumminess); (ii) Geometrical characteristics: Particle size and shape, particle shape and orientations; (iii) other characteristics: MC, fat content, greasiness, etc. [92, 93]. In this regards, the several probes for texture analysis of food products include the use of cylinder, cone, ball, blade, wire, plate, knife, etc., where the selection of probes depends on the selected food product.

Texture Profile Analysis. Additionally, the texture profile analysis (TPA) is the measurement of food texture where the spatial textural events of samples are monitored and recorded. The mechanical measurements of TPA of food products are classified into destructive and non-destructive methods, where the destructive methods include the testing such as puncture and penetration tests, three-point bending test, single-edge notched bend test, etc., whereas the non-destructive methods include impact response, quasi-static force-deformation, bioyield detection, etc. In non-destruction method of texture measurement for TPA analysis, there is no visible damages are obtained. As represented in Fig. 15.10, the texture modeling of food products includes first order reaction/kinetic model,

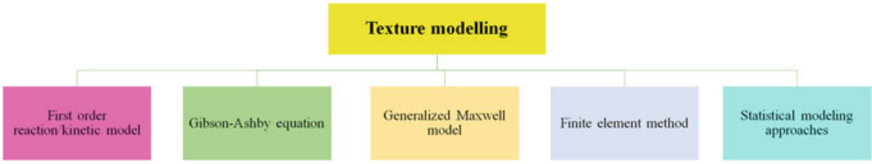

Fig. 15.10 Several Texture modelling of food products

Gibson-Ashby equation, Generalized Maxwell model, Finite element method (FEM), statistical modelling approaches, etc. [94]. Additionally, a model for mouth behavior can also be utilized to mimic the chewing behavior of food products [95]. The textural characteristics of food products including hardness, adhesiveness, springiness, cohesiveness, resilience, gumminess, chewiness and others can be analyzed using TPA to control the quality of the food products [96]. Moreover, the texture measurement for objective evaluation is classified into three types: fundamental, empirical, and imitative [97]. In this way, the textural attributes of stored food products with and without edible food packaging (films/coatings) are analyzed for customer acceptability and to maintain food texture.

15.4.5 Migration Study for Packaging Application

The NPs are widely utilized in the fabrication of composite based food packaging materials for obtaining the tailored-made properties. However, the NPs used in the packaging materials should have the migration values within the permissible limits. Thus, risk assessment of NPs and potential migration assays are required for maintaining the safety of the food products [98]. As discussed in earlier chapters, the nanotechnology has received a great deal of interest in developing FCMs, where the FCMs is classified into various sectors such as improved packaging properties with NPs reinforcements, active packaging due to reinforcement of active NPs, intelligent packaging with nanosensors, biocomposite based packaging materials, etc. Additionally, all packaging materials generally have the problems of migration of several chemical substances from FCMs into food stuffs such as food additives, monomers, food residues, etc. [99]. The migration study in food packaging is generally defined as a mass transfer phenomenon, where low molecular weight components are firstly diffused into the food products, and the mechanism of migration behavior of food components can be described by Fick's second law. Thus, in migration testing, the release of substances from the packaging materials into the food products or to the food simulants are determined. Further, the migration models are generally developed based on Fick's second law, where the diffusion coefficients are estimated for the modelling of partition migration [100]. The migration tests are conducted following the rules and regulations of Commission Regulation EU No. 10/2011 [101]. For the determination of overall migration, a specimen/film having area of 1 dm^2 of contact area per 100 mL of simulants are stored for 10 days at 40 °C. According to the European Regulation, the migration study is generally studied using overall migration tests and specific migration tests [102].

Interestingly, the migration of several compounds in food products occurs following several steps such as (1) the diffusion of chemical compounds due to Brownian movement such as unreacted monomers, monomers developed from packaging materials due to adverse conditions. However, the generation of chemical compounds during storage of food products or heating depends on several factors such as type of packaging materials, temperature, time period, and others. (2) desorption of the absorbed compounds from the polymeric surface; (3) solvation or migration of the compounds to food products from food-plastic interface, where solvation of compounds occurs when the migrants have the better solubility in food products than in FCMs; and (4) dispersion into bulk food materials. The migration of several plastic compounds may held by various ways such as (1) diffusion mechanism (direct contact migration): The compounds of packaging materials diffused from the packaging layer to the FCMs; (2) Gas phase migration is considered as indirect contact migration where the volatile compounds are generally generated due to high temperature exposure and can migrate from the packaging materials to the FCMs; and (3) Set off migration is generally caused due to migration of compounds during manufacturing and storage. Additionally, in packaging materials, the migration can occur through several ways such as penetration migration, contact/set off migration, evaporation migration, and temperature gradient migration. As shown in Fig. 15.11a, the penetration migration in packaging films occurs from printed side to the unprinted side (FCMs) through the substrate. The contact/set off migration (Fig. 15.11b) occurs from the printed side to

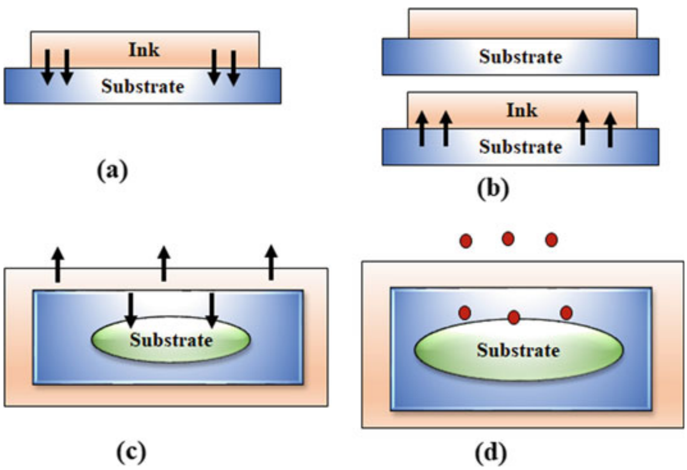

Fig. 15.11 General mechanisms of migration (a) Penetration migration, (b) Contact/Set off migration, (c) Evaporation migration, and (d) Temperature gradient migration

the unprinted side of the substrate, when remains in contact with each other. The evaporation migration (as shown in Fig. 15.11c) held due to the evaporation of several volatile compounds from the packaging materials during heating such as microwave heating of food products with the containers. The temperature gradient migration (Fig. 15.11d) causes due to the raising temperature such as sterilization. Further, the use of functional barrier may decrease the migration of packaging components to food simulating liquids [103].

Several Migrating Components from Food Contact Materials. The migrating components to food products include isopropylthioxanthone, epoxydised soy bean oil, diethylhexyl phthalate, primary aromatic amines, plasticizers, additives (plasticizers, coloring agents, heat stabilizers), etc. The potential migrants in food products are antimicrobials, colorants, UV absorbers, light stabilizers, plasticizers, anti-fogging additives, and others. The contaminants such as plasticizers, heavy metals, polyaromatic hydrocarbons, fluorescent whitening agents, residual solvents, etc. are generally analyzed for recycled paper and boardwhen used as a packaging materials [104]. The volatile compounds in pellets and packaging materials for food products are 2-Methyl-1-propene, 2-Propanone, dichloromethane, cyclohexane, ethylbenzene, xylene, styrene, decane, dodecane, heptane, 3-methyl pentene, cyclohexanone, tetradecane, pentadecane, octadecane, etc. [105]. Several plastic containers are used to carry food products in microwave ovens, where volatile compounds can release and migration of the compounds can occur from container to food products [105]. The several plastic containers used for microwave heating are polycarbonate, PP, PP-copolymer, and others. The plastic containers are not inert and diffusion of different plastic components held when food products are packed within it. The plastic components get diffused from the plastic to the food products, where increase in temperature may increase the migration of plastic compounds. In plastic based packaging, the monomers such as styrene, vinyl chloride, isocyanate, caprolactam, and others can create adverse health effect due to carcinogenic nature if get diffused or leached out to food products.

Food Simulants for Migration Testing. The food simulants for migration testing include water, acidic water (mimic acidic foods), 15% ethanol (mimic alcoholic beverages), and rectified olive oil (mimic fatty foods) [106]. Tenax® is another known dry food simulant, which consists of granules of modified polyphenylene oxide [107]. A research reports the study on migration of adhesives into Tenax®, where 57% of total compounds (55 different compounds) are found to migrate in the selected food simulants [107]. Further, porapak is also found to use as a solid food simulant for paper and board packaging and further, porapak is having stable property even at high temperature in comparison to Tenax [108]. For paper and board based packaging, several food products including pasta, milk powder, dry soup, flour and bakery products, icing sugar and others has been tested for migration study [104]. The perfluorochemicals are also used to prepare FCM such as polytetrafluoroethylene, and the development of this kind of materials involve the use of perfluorooctane, sulfonate, perfluorooctanic acid, which are biopersistent in nature and can cause several health issues [103].

The use of nanoclay to develop food packaging application is found to provide very low migration value into food products in comparison to other nanomaterials using food simulants such as 10% ethanol and 3% acetic acid and storage conditions (1) 40°C for 10 days, and (2) 70 °C for 2 h [109]. Further, a study shows that the level of migration into pork meat products (packaged in low density polyethylene films and storage condition: 10 days and 25 °C) increase with increased storage temperature and fat content. Additionally, the use of nanomaterials such as silver NPs for developing biocomposite based packaging is dependent on the migration of nanomaterials from FCM into food products for safety purpose [110]. The migration analysis is time consuming and required specific conditions to study the behavior of packaging components. The detection of chemical substances that releases from plastic substances due to migration can be detected using a mass spectrometry such as High resolution mass spectrometry with enhanced accuracy [111]. In this regards, a report suggests the detection of various compounds from vacuum packed meat samples using a mass-spectrometry based instrumentations. Thus, the migration testing of packaging materials are essential to avoid the adverse health effect which may be caused by the several packaging compounds.

15.4.6 Other Properties

Besides, the above mentioned properties, the microbiological study to test the microbial growth in edible coated or stored food products are undergone. In this regards, the mesophilic and psychrophilic counts are determined during the storage life to test the effectiveness of edible coating and films in inhibiting or reducing the microbial growth. Moreover, other studies such as color properties, food composition are also determined to check the quality of stored food products.

15.5 Conclusions

The nanoscience and nanotechnology have played a vital role in the upliftment of the usage of the food packaging materials across the globe. The food, which is one of our basic needs of life, need to be consumed in a healthy manner. The developments of nanomaterials have paved a path to introduce essential nutrients towards healthier and safer consumption of food. The introduction of biodegradable and sustainable polymer edible films into this, would not only protect the food products from outside but also can be consumed along with the food. The incorporation of nano-biomaterials onto the food packaging can improve the shelf-life and quality of the food. The edible films that are introduced can be easily accepted by the cells of the body because of their various surface and size related properties. The developed biobased nanomaterials for edible food packaging are characterized by different characterization techniques that are discussed which can give us a greater idea of different types of biomaterials used and their properties. The natural based biopolymers when incorporated with nano-biomaterials not only improves its surface properties but also mechanical properties and thermal properties to withstand various temperature conditions. Therefore, the introduction of the various biomaterials and their characterization helps in understanding and preventing the food from damage and also minimise the hazard from disposal of the packaging products. The sensory properties are considered as the most crucial element for attaining the consumer acceptability. The sensory properties of food products are taste, smell, appearance, texture, sound, mouthfeel and others, which can be well assayed by sensory evaluation process. The polymer based packaging materials developed through incorporating filler materials, cross linking agents, compatibilizers plasticizers and others, where some components or filler materials of packaging materials which are unable to react with matrix materials have a tendency to get trapped within the matrix materials. The trapped materials have a tendency to migrate from the packaging materials when attained a change in storage condition or get exposure to a similar kind of materials. However, the filler materials or other agents are generally incorporated in a small amount and the migration of the components should be within the permissible limits. Thus, the migratory components need to be controlled and regulated properly to avoid migration, and the migration of FCM to food products is found to obey the Fick's laws of diffusion. The migration of packaging components from FCM to food products generally depends on several factors such as nature of FCM, physical and chemical properties of food components, storage temperature, contact area, used adhesives for sealing the packaging materials, types of food products, etc.

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