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.

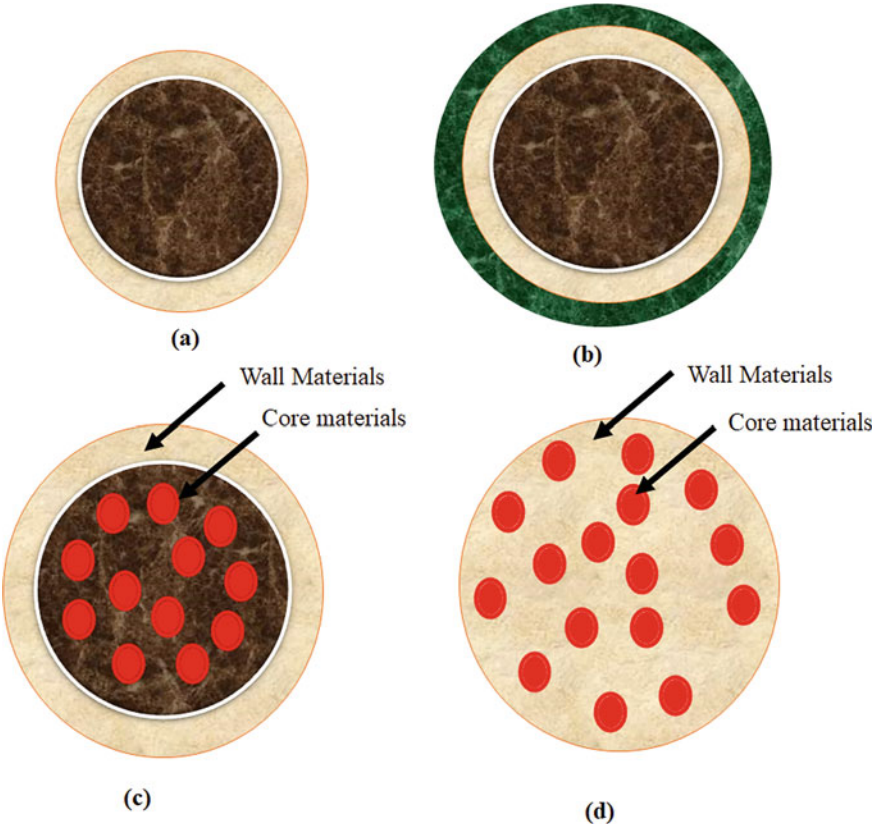

Fig. 10.1 Various structures of nanoencapsulates **a** mononuclear, **b** multishell, **c** multicore, and **d** matrix

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.

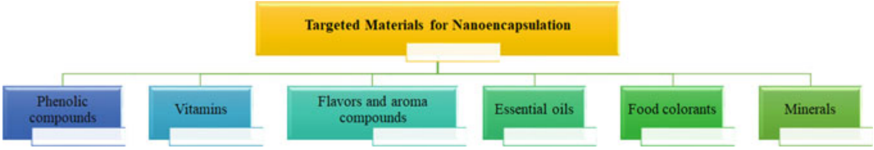

Fig. 10.2 Several targeted materials for nanoencapsulations

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,

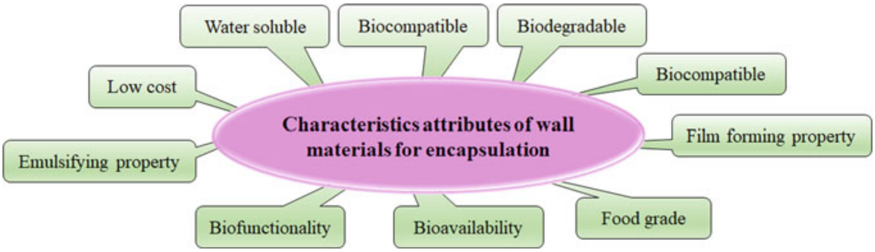

Fig. 10.3 Characteristics attributes of carrier or wall materials for fabrication of nanoencapsulates of bioactive food components

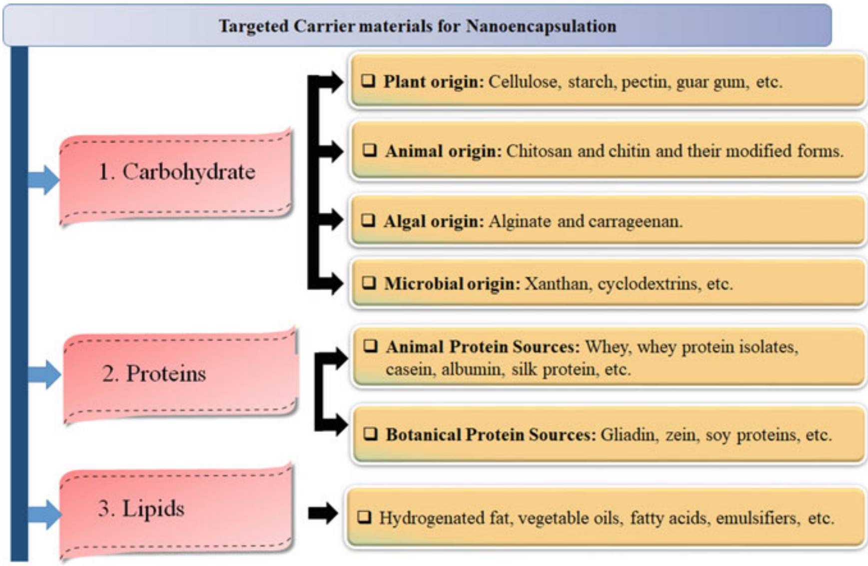

Fig. 10.4 Several targeted carrier or wall materials for nanoencapsulation techniques

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]

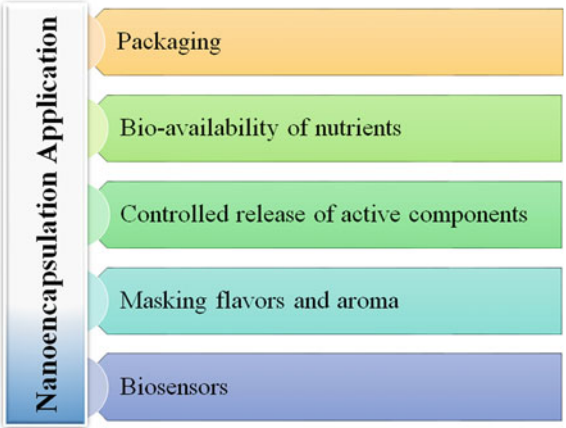

Fig. 10.5 Various applications of nanoencapsulation

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

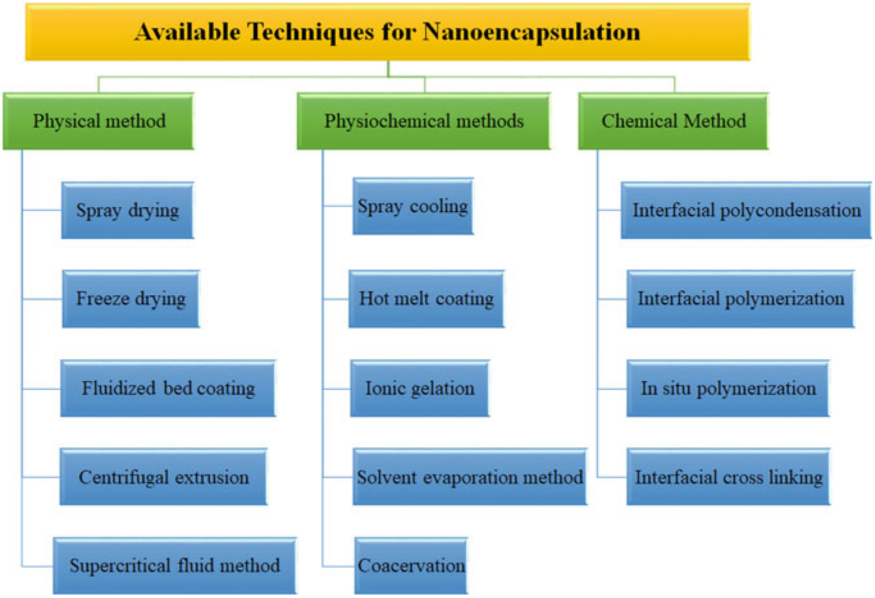

Fig. 10.6 Several techniques for developing nanoencapsulates of bioactive components

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

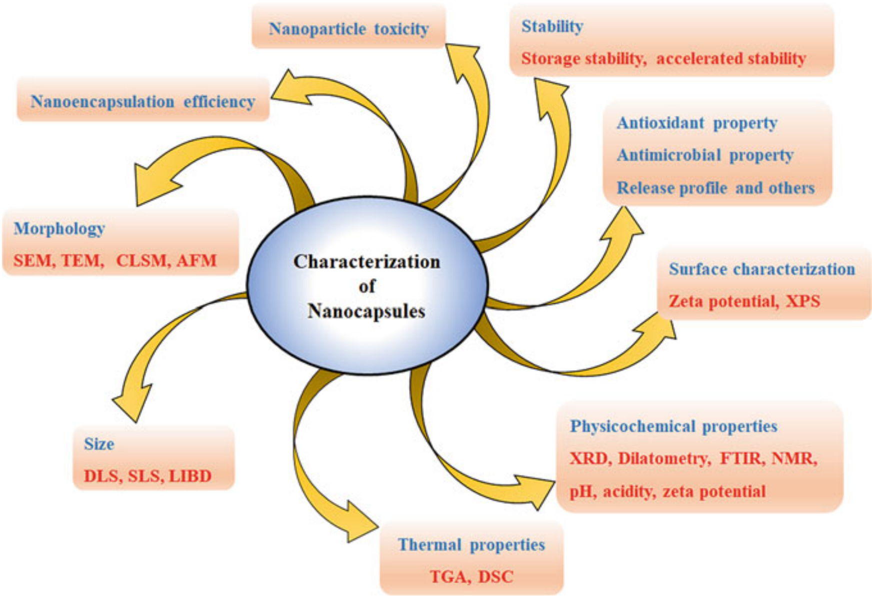

Fig. 10.7 Characterization techniques of nanoencapsulated powders (SEM: scanning electron microscopy; TEM: transmission electron microscopy, CLSM: confocal laser scanning microscopy; AFM: atomic force microscopy; DLS: dynamic light scattering, SLS: static light scattering; LIBD: laser-induced breakdown 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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