

# Chapter 27

## Edible Coatings to Improve Food Quality and Safety

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### 27.1 Introduction

Edible films and coatings are useful materials mainly produced from edible biopolymers and food-grade additives (GRAS). Films are usually made from polymers that are able to provide mechanical strength to a stand-alone thin structure (Han 2005; Han and Gennadios 2005). Coatings are a particular form of films directly applied to the surface of materials. An edible coating has a close and continuous association with the food until consumption and it is regarded as a part of the final product. Edible coatings enhance the quality of food products, protecting them from physical, chemical, and biological deterioration (Kester and Fennema 1986; Han and Gennadios 2005).

The application of edible coatings can improve the physical strength of food products, reduce particle clustering, and improve visual and tactile features on product surfaces (Cuq et al. 1995; Cisneros-Zevallos et al. 1997). The coatings can also protect food products from moisture migration, microbial growth on the surface, light-induced chemical changes, oxidation of nutrients, etc. Edible coatings can act as barriers against oils, gases, or vapors and as carriers of active substances (antioxidants, antimicrobials, colors, and flavors) (Kester and Fennema 1986; Gennadios and Weller 1990; Guilbert and Gontard 1995; Krochta and De Mulder-Johnston 1997; Miller et al. 1998). These functions enhance the quality of food products, resulting in shelf-life extension and safety improvement. Further, edible coatings can be utilized as active films when applied to modify the atmosphere of food surface conditions (Cuq et al. 1995; Guilbert and Gontard 2005).

As an example of edible films, yuba (soy-milk skin) has been traditionally used in Asian countries since the fifteenth century (Wu and Bates 1972; Park et al. 2002). Wax coatings were applied to citrus fruits in the twelfth and thirteenth centuries, but only

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commercially utilized on apples and pears since 1930 (Baldwin 1994; Debeaufort et al. 1998; Park 1999). Lipid coatings (larding) on meats and cheeses have been used since the Middle Ages for shrinkage prevention (Donhowe and Fennema 1994).

The objectives of this presentation are to: (1) discuss the basic concepts of food coating applications, (2) review the most significant publications in this area, and (3) describe the different experiments carried out in our laboratory studying the performance of edible coatings to improve food quality and safety.

## 27.2 Edible Coating Materials

Coating materials can be either hydrophilic or hydrophobic; the main film-forming materials are biopolymers, such as proteins and polysaccharides (carbohydrates and gums); lipids and resins are also used, however they are not biopolymers (Gennadios et al. 1997). Coating materials can be used alone or in combinations (Krochta et al. 1994; Baldwin et al. 1997). The formulation of bioplastics or edible films implies the use of at least one component able to form a matrix, having sufficient cohesion and continuity (Nussinovitch 2003).

**Polysaccharides:** The most common polysaccharides used for edible coatings are methyl cellulose (MC), carboxymethyl cellulose (CMC), hydroxypropyl cellulose (HPC), hydroxypropyl methyl cellulose (HPMC), starch, modified starches, amylose, hydroxypropyl amylose (HPA), alginate, carrageenan, pectin, chitosan, gellan gum, xanthan gum, etc. The occurrence of relatively large numbers of hydroxyl groups in the structure of polysaccharides indicates that hydrogen bonds may play significant roles in film formation and characteristics. Starch is the most common agricultural raw material used for biodegradable and edible films formulation, and is an appropriate matrix-forming material with lower cost compared to other alternatives. Starch is also widely available and relatively easy to handle; amylose is responsible for the film-forming capacity of starch (Guilbert and Gontard 2005). Cellulose derivatives are composed of linear chains of  $\beta$  (1–4) glucosidic units with methyl, hydroxypropyl, or carboxyl substituents. MC has excellent film-making properties, as well as high solubility and efficient O<sub>2</sub> and lipid-barrier properties (Donhowe and Fennema 1994; Nisperos-Carriedo 1994). Two other examples, chitin and its deacetylated product, chitosan, have received much interest regarding application in the food industry due to their biocompatibility, biodegradability, and bioactivity.

**Proteins:** Different proteins such as whey protein, casein, collagen, gelatin, egg white protein, keratin, fish, myofibrillar protein, corn zein, wheat gluten, soy protein, pea protein, peanut protein, cottonseed protein, rice bran protein, etc., have been used in edible coatings. The most distinctive characteristics of proteins, compared to other film-forming materials, would be the conformational denaturation, electrostatic charges, and amphiphilic nature of proteins. Many factors can affect the conformation of proteins, such as charge density and hydrophilic-hydrophobic balance, which in turn can affect the physical and mechanical properties of films and coatings.

Protein film-forming materials are derived from animal tissues, milk, eggs, grains, and oil seeds (Krochta 1997, 2002). Among the proteins considered, milk proteins (casein, whey proteins), soy proteins, and cereal proteins (wheat gluten, zein) have been more extensively studied (Gennadios et al. 1994; Guilbert et al. 2001). Since several edible food coatings are made with proteins that can cause allergic responses in some consumers and problems in people affected by Celiac disease (gluten intolerance), the use of a food coating with a known allergen or the presence of gluten in a food must be appropriately declared and clearly labeled on the product.

**Waxes/Lipids/Resins:** In this group, beeswax, candelilla wax, carnauba wax, cocoa butter, milk fat fractions, acetylated monoglycerides, fatty acids, and resins (shellac, terpene) are used in edible coatings. It should be noted that lipids and resins are not biopolymers; nevertheless, they are edible, biodegradable, and considered to be cohesive water barrier biomaterials. Most lipids and edible resins are soft-solids at room temperature and show characteristic phase transition temperatures. Films or coatings made from lipids have very high water resistance and low surface energy, due to their hydrophobic nature. Lipids are generally applied in thin layers or as a composite with a polymeric matrix. Lipids can be combined with other film-forming materials, such as proteins or polysaccharides, as emulsion particles or as multilayer coatings in order to increase the resistance to water penetration (Greener and Fennema 1989; Greener and Fennema 1992; Baldwin et al. 1997; Gennadios et al. 1997; Perez-Gago and Krochta 2002; Perez-Gago and Krochta 2005).

**Plasticizers:** The structures of films containing polysaccharides and proteins are often brittle and stiff due to extensive interactions between the polymer molecules (Krochta 2002). The film-forming mechanisms of biopolymers include intermolecular forces such as covalent bonds (e.g., disulfide bonds and cross-linking) and/or electrostatic, hydrophobic, or ionic interactions. The addition of plasticizers overcomes this film brittleness and improves flexibility and extensibility. Plasticizers are low molecular weight agents incorporated into the polymeric film-forming material; the plasticizer positions itself between the polymer molecules and interferes with the polymer-polymer interaction, increasing the flexibility and processability of the material (Guilbert and Gontard 1995). Plasticizers must be compatible with the film-forming polymer; they reduce the intermolecular forces and increase the mobility of polymer chains. Plasticizers increase the free volume of polymer structures or the molecular mobility of polymer molecules (Sothornvit and Krochta 2000, 2001), decreasing the ratio between the crystalline and the amorphous regions, and decreasing the glass transition temperature ( $T_g$ ) of the polymer (Krochta 2002; Guilbert and Gontard 2005). Most plasticizers are very hydrophilic and hygroscopic; they attract water molecules and form a large hydrodynamic plasticizer-water complex. Hydrophilic compounds, such as polyols (glycerol, sorbitol, and polyethylene glycol) are commonly used as plasticizers in hydrophilic film formulations.

**Functional additives:** This group includes antioxidants, antimicrobials, nutrients, nutraceuticals, flavors, and colorants, which can be combined with film-forming biopolymers to modify the functionality of films or coatings. The most beneficial characteristics of edible films and coatings are their edibility and inherent

biodegradability (Guilbert et al. 1996). To maintain edibility, all film components (i.e., biopolymers, plasticizers, and other additives) should be food-grade ingredients and all process facilities should be acceptable for food processing; solvents used should be restricted to water and ethanol.

### 27.3 Application and Distribution of Edible Coatings on the Food Surface

The application and distribution of the film-coating material in a liquid form can be achieved by hand spreading with a paint brush, spraying, falling film enrobing, dipping and subsequent dripping, and distribution in a revolving pan (pan coating), etc. (Guilbert 1986). Edible coating formulations must be wet when spread on the food's surface; upon drying the formulation must form a film coating with adequate adhesion, cohesion, and durability to function properly.

Different tests can be performed on the filmogenic suspensions before the coating is applied on a foodstuff. Rheological analysis and viscoelastic properties of the suspensions help to determine the suitability of the food coating method (immersion, spraying, etc.). Rheological behaviors (flow curves) are normally evaluated using viscometers; oscillatory rheometers allow measurement of the viscoelastic parameters of the film-forming suspension (García et al. 2004b, 2006; Lopez et al. 2008).

Some negatively charged gums, such as alginate, pectin, and CMC, show significantly different rheological properties in acidic conditions as opposed to neutral or alkaline conditions. Rheological behavior and surface tension of the film-forming suspension are important factors linked to suspension spreadability and coating adhesion capacity. A decrease in the surface tension of the filmogenic suspension leads to a better coating adhesion to foodstuffs. The peel or surface of many vegetables has low surface tension for protection purposes; however, this natural advantage is a drawback in the case of aqueous coating applications. The properties of the edible coating depend on the type of film-forming materials and especially on their structural characteristics (Han and Gennadios 2005).

Cohesion is the attractive force between the molecules of the same substance; it depends on the structure of the polymer, the molecular length, geometry, and molecular weight distribution, and on the type and position of the lateral groups. Cohesion of film-forming materials influences the mechanical strength of the films (Guilbert et al. 1996). If the film-forming material contains different components that are not compatible with the main biopolymers, the cohesion decreases and film strength weakens. Plasticizers reduce the cohesion of film-forming polymers.

Adhesion of film-forming materials is an important parameter for film casting and coating processes (Guilbert and Gontard 2005). Adhesion is an attractive force between the surface molecules of coating materials and food surfaces. A low

adhesion force leads to an incomplete coating on the food surface. A large difference between the surface energy of a coating material, and the uncoated product surface, results in a poor coating performance. Surface active agents, such as emulsifiers in the film-forming solution, reduce the surface tension of the coating solution; then the difference between the solid surface energy and the surface tension of the coating solution decreases, increasing the work of adhesion (Cuq et al. 1995; Guilbert and Gontard 2005).

## 27.4 Edible Coating Characteristics as Related to Polymer Structure and Physico-chemical Properties

The physical and chemical characteristics of the biopolymers greatly influence the properties of resulting films and coatings. An edible film is essentially a dried and extensively interacting polymer network of a three-dimensional structure. The film-forming mechanisms of biopolymers include intermolecular forces such as covalent bonds (e.g., disulfide bonds and cross-linking) and/or electrostatic, hydrophobic, or ionic interactions. It is important to determine the relationship between the polymer structural chemistry and the physico-chemical and mechanical properties of the film or coating. Several techniques can be used to characterize the structure of the biopolymers forming the coatings, such as scanning electron microscopy (SEM), X-ray diffraction, differential scanning calorimetry (DSC), thermal mechanical analysis (TMA), dynamic mechanical thermal analysis (DMTA), and Fourier transform infrared spectroscopy (FTIR), etc. The main properties of a coating are as follows: moisture and gas permeation (barrier properties), water vapor sorption isotherms, optical attributes (light transmittance, color), thermoplastic characteristics, crystallinity, mechanical properties of the film (strength, elasticity), and film solubility in water and oil.

The optical properties impact the surface appearance of coated foods. Surface film color is generally measured using a colorimeter and opacity is determined by spectrophotometry. Film opacity is a critical property of a food coating; it can be measured using the method proposed by Gontard et al. (1992), in which the absorption spectrum is recorded in the visible range. The opacity is estimated as the area below the absorption curve; this area has a very low value for transparent films. The color, shininess, and transparency of edible films and coating layers vary significantly depending on the chemical composition and the structure of the polymer used. For example, the color stability of whey protein isolate (WPI) coatings is affected by Maillard reactions (produced by milk proteins during storage), causing yellowing.

The water sorption isotherms of the coatings can be measured on films formed under standard conditions. They are useful to estimate stability at different ambient conditions, mainly because the hydrocolloid film properties are highly dependent

on relative humidity (RH). The presence of plasticizers, such as glycerol or sorbitol increases the hygroscopic characteristics of the films.

Microstructure characterization of the coatings helps to understand the performance of the different formulations. Light microscopy observation (stereomicroscopy) is used to analyze coating thickness and uniformity; SEM permits evaluation of film homogeneity, the presence of different layers, pores, and cracks, and the smoothness of the coated surface. FTIR is a useful technique for composite film microstructure characterization since it allows evaluation of interactions and compatibility between the components (Lacroix and Le Tien 2005), and permits identification of the functional groups in the polymers after modifications.

The spectral regions  $3,300\text{--}3,000\text{ cm}^{-1}$  are generally used for elucidation of the free or interacting hydroxyl groups;  $2,950\text{--}2,850\text{ cm}^{-1}$  for alkyl ( $-\text{CH}_2-$ ) chains; at  $1,750\text{--}1,650\text{ cm}^{-1}$  the bands represent carbonyl groups and amides bands. Pinotti et al. (2007) applied FTIR spectrometry to determine the possible interactions between chitosan and MC composite systems.

X-ray diffraction patterns of the films are characterized by sharp peaks associated with the crystalline fraction and amorphous zones. The amorphous fraction of the sample can be estimated by the area between the smooth curve, drawn to follow the scattering hump, and the baseline joining the background within the low- and high-angle points; the crystalline fraction can be estimated by the upper region above the smooth curve. X-rays can also detect the recrystallization process during storage, such as retrogradation in starch-based films and coatings (García et al. 2000b; Mali et al. 2002, 2006)

Glass transition is a reversible change that takes place in the polymer between the rubbery and glassy states; the temperature at which it occurs is the  $T_g$ . The properties of amorphous or semicrystalline materials are seriously modified when the temperature of the compounds rises above  $T_g$ . Generally, fully amorphous bioplastic applications are limited by the fact that  $T_g$  of a polymer is highly affected by the RH (especially for hydrophilic polymers). Below  $T_g$  the material is rigid, and above  $T_g$ , it becomes rubbery and visco-elastic (Guilbert and Gontard 2005). Below  $T_g$ , weak and non-cooperative local vibrations and rotation movements of the molecules are possible; above  $T_g$ , strong and cooperative movements of whole molecules and polymer segments can be observed. The knowledge of  $T_g$  is important since it determines both the mechanical and barrier properties (Mali et al. 2005, 2006). DSC and DMTA techniques can be used to determine  $T_g$ . DMTA is considered the most sensitive method for measuring the  $T_g$  of a material. In DMTA, an oscillating strain is applied to a sample and the resulting stress developed in the sample is measured. The output signals are analyzed, and, using established mathematical methods, the rheological parameters are also computed. The ratio of viscous modulus ( $E''$ ) to elastic modulus ( $E'$ ) is the tangent of the phase angle shift between the stress and strain vectors ( $E''/E' = \tan \delta$ ); it measures the damping ability of the material. The glass transition of a film is detected as a sudden and marked decrease in the elastic modulus  $E'$  and a peak in the  $\tan \delta$  curve as temperature increases. Plasticization decreases the intermolecular forces between polymer chains, and reduces  $T_g$ .

## 27.5 Barrier Properties

The quality of most food products deteriorates from moisture absorption, O<sub>2</sub> invasion, flavor loss, undesirable odor absorption, and migration of packaging components into the food (Kester and Fennema 1986; Debeaufort et al. 1998; Miller et al. 1998; Krochta 2002). These mass transfer phenomena can occur between the food and the environment, the food and packaging materials, or among heterogeneous ingredients in the food product itself (Krochta 1997). The diffusion of atmospheric O<sub>2</sub> into foods causes oxidation of food ingredients; dried foods can absorb moisture from the environment or from other wet zones in the food, leading to the loss of crispness. Edible films and coatings may be used to wrap these food products or located between the heterogeneous parts of food products to prevent migration phenomena and to preserve quality (Guilbert et al. 1997; Krochta 2002).

To characterize the barrier properties of edible films and coatings, the transmission rates of specific migrants should be determined using stand-alone edible films (McHugh and Krochta 1994). Most of the research on this subject reports the water vapor, O<sub>2</sub>, CO<sub>2</sub> and flavor permeabilities, and oil resistance of edible films. However, barrier properties of films and coatings determine their applicability to improving the storage life of different products. Permeability depends not only on the diffusion coefficient but also on the solubility of permeants in the system. Barrier properties are strongly related to film structure since permeants are moved through the amorphous zone. Film formulation, especially with the addition of plasticizers, affects both the barrier and mechanical properties because these properties modify film structure, chain mobility, and diffusion coefficients of permeants.

### 27.5.1 Water Vapor Permeability

Water vapor permeability (WVP) determinations can be performed using commercial instruments; in addition, many researchers applied the ASTM (1995) method E96 with the modifications introduced by Gennadios et al. (1994). In this method the film sample is sealed over a circular opening of known area in a permeation cell that is stored at constant temperature in a desiccator. Anhydrous calcium chloride or silica gel (0% RH) is placed inside the cell and a saturated solution (constant RH) is used in the desiccator; thus water vapor transport is determined from the weight gain of the permeation cell (García et al. 1999, 2000a, 2004b, 2006; Romero-Bastida et al. 2005).

Synthetic moisture-barrier films such as low-density polyethylene (LDPE) show low values of  $WVP = 9.14 \times 10^{-13} \text{ g m}^{-1} \text{ s}^{-1} \text{ Pa}^{-1}$  in comparison with polysaccharides and proteins, which are polymeric and hydrophilic by nature and poor moisture barriers. The WVP permeabilities of proteins, such as gluten and zein plastified with glycerol, range between  $7.00 \times 10^{-10}$  and  $8.90 \times 10^{-10} \text{ g m}^{-1} \text{ s}^{-1} \text{ Pa}^{-1}$ .

Lower WVP values were reported for several polysaccharides; sorbitol plastified yam starch (3%) and corn starch (2%) films have WVP values of  $1.50 \times 10^{-10}$  and  $1.75 \times 10^{-10} \text{ g m}^{-1} \text{ s}^{-1} \text{ Pa}^{-1}$ , respectively (Mali et al. 2002, 2004, 2006). Films of MC (1%) and Chitosan (1%) have WVP values similar to that of cellophane ranging between  $7.55 \times 10^{-11}$  and  $9.03 \times 10^{-11} \text{ g m}^{-1} \text{ s}^{-1} \text{ Pa}^{-1}$  (Pinotti et al. 2007).

It is important to remark that edible films made of proteins and polysaccharides have low water-vapor barrier properties, with WVP values at one and two orders of magnitude higher than that of LDPE; therefore, they can only be used as protective barriers to limit moisture exchange in short-term applications. However, these edible films can be of considerable value for numerous processes, as in the case of modified atmosphere packaging of fresh, minimally processed or fermented foods (fish, meat, fruits, vegetables, and cheeses).

Lipids or waxes for edible applications show better water-vapor barrier properties than starch or protein-based materials do, but they still have significantly lower water-vapor barrier properties than most synthetic plastic films; WVP values ranging between  $2.75$  and  $3.1 \times 10^{-12} \text{ g m}^{-1} \text{ s}^{-1} \text{ Pa}^{-1}$  were reported for beeswax and carnauba wax (Shellhammer and Krochta 1997; Perez-Gago and Krochta 2005).

Lipid compounds, such as animal and vegetable fats (natural waxes and derivatives, acetoglycerides, surfactants, etc.), could be proposed as components of edible coatings given their excellent moisture-barrier properties; however, they can cause textural and sensory problems due to oxidation and a waxy taste; further, their nonpolymeric nature limits the ability to form cohesive films (Perez-Gago and Krochta 2005). The addition of lipids to polysaccharide- and protein-based films decreases WVP due to hydrophobicity; this is an important property in the case of fresh fruits and vegetables because composite coatings retard moisture loss and shriveling in fresh products.

### 27.5.2 Gas Permeabilities

$\text{CO}_2$  and  $\text{O}_2$  barrier properties are important in understanding the quality and physiological aspects of coated food during storage and determine food coatings applications. Gas permeability is commonly measured on isolated films.  $\text{O}_2$  permeability can be measured using available commercial equipment; moreover,  $\text{CO}_2$  and  $\text{O}_2$  permeability of films can be assessed by the accumulation method in a specially designed cell (García et al. 1999, 2000b; Bifani et al. 2007). This quasistatic method was based on measuring the amount of gas diffusing through a film, quantified by gas chromatography.

García et al. (1999) measured gaseous permeabilities to  $\text{O}_2$  ( $\text{PO}_2$ ) and  $\text{CO}_2$  ( $\text{PCO}_2$ ) for different starch-based films; values of  $\text{PO}_2 = 5.69 \times 10^{-9} \text{ cm}^3 \text{ m}^{-1} \text{ s}^{-1} \text{ Pa}^{-1}$  and  $\text{PCO}_2 = 0.248 \times 10^{-9} \text{ cm}^3 \text{ m}^{-1} \text{ s}^{-1} \text{ Pa}^{-1}$  were reported for corn starch films plasticized with glycerol. All tested starch-based films, plasticized with either glycerol or sorbitol, showed higher gas permeability to  $\text{CO}_2$  than  $\text{O}_2$ , with a ratio of permeability ( $\text{PCO}_2/\text{PO}_2$ ) ranging between 8 and 10 (selectivity



coefficient). These results can be attributed to a higher solubility of CO<sub>2</sub> than O<sub>2</sub> in the coatings (McHugh and Krochta 1994). Synthetic materials such as LDPE have lower selectivity coefficients between CO<sub>2</sub> and O<sub>2</sub> (around 4) compared to an average ratio of 9 in the case of starch-based films (Cuq et al. 1998; García et al. 1998ab; Park 1999). Cuq et al. (1995) compared the CO<sub>2</sub> to O<sub>2</sub> permeability ratio for several synthetic and edible films and reported that edible films show higher selectivity than synthetic ones, with ratios in the range of 8–30. Very high gas selectivity is particularly interesting for modified atmosphere packaging of cheeses (to control proliferation of microflora), and for fresh fruits and vegetables (to control respiration rates).

Gas permeability is strongly related to crystallinity of polymeric chains, where in general, the higher the degree of crystallinity, the lower the permeability. As for synthetic materials, gas and vapor permeabilities depend on several factors, such as the ratio between crystalline and amorphous zones, polymeric chain mobility, and specific interactions between the functional groups in the polymers and gases in the amorphous zones. Permeability increases with a decreasing crystalline-amorphous ratio because permeation occurs through the amorphous zones of the film.

Starch and proteins have gas-barrier properties in dry conditions, especially against O<sub>2</sub>. For example, the O<sub>2</sub> permeability of a wheat gluten film was 800 times lower than that of LDPE. However, O<sub>2</sub> permeability is very sensitive to RH (Guilbert et al. 1997). At higher RH conditions, O<sub>2</sub> permeability increases significantly; thus, it is important to maintain low RH in the environment to maximize the effectiveness of edible films as gas barriers. Values of the selectivity coefficient between CO<sub>2</sub> and O<sub>2</sub> ranging between 4 and 35 were reported by Gontard et al. (1996); this selectivity coefficient is very sensitive to moisture and temperature for gluten films, whereas for synthetic polymers it remains relatively constant ranging between 4 and 6 (Guilbert and Gontard 2005).

## 27.6 Composite Film Formation

Composite films and coatings can be formulated to combine the advantages of each component. Biopolymer composites can modify film properties and create film structures for specific applications (Wu et al. 2002). A composite film that combines lipids and hydrocolloids can be produced as either a bi-layer or a stable emulsion. In bi-layer composite films, the lipid forms a second layer over the polysaccharide or protein layer. In emulsion composite films, the lipid is dispersed and entrapped in the supporting matrix of protein or polysaccharide (Perez-Gago and Krochta 2005). The emulsifying character of proteins makes them appropriate for this technique; however, in the case of polysaccharides, the addition of an emulsifier is required to improve emulsion stability. Emulsion preparation parameters, such as stirring velocity and emulsifier addition are important factors in obtaining homogenous films and coatings, since they determine emulsion stability.

## 27.7 Examples of Coating Applications

In general, an ideal coating should be safe, unperceived by the consumer, have no off-flavor and taste, be a desirable moisture and gas barrier, and have a certain mechanical strength. Many factors determine the success of edible coatings in improving the quality and extending the shelf-life of foods. Factors such as chemical composition, structure, methods used to form the films or coatings, storage conditions, and properties of the food itself (maturity stage in the case of fruits and vegetables, moisture and lipid content, etc.) are all important. When applying edible films and coatings, these factors have to be carefully considered (Zhao and McDaniel 2005).

Quality maintenance and product enhancement are significant functions of edible films and coatings (Krochta 1997). They can retard surface dehydration, moisture absorption, oxidation of ingredients, aroma loss, frying oil absorption, ripening/aging, and microbial deterioration of food products. In addition to the physical and chemical quality enhancements, edible films and coatings contribute to visual quality, surface smoothness, flavor carriage, edible color print, and other marketing-related quality factors. Edible films and coatings may be used to preserve the quality of several food commodities. The O<sub>2</sub>-barrier property of films and coating layers can prevent oxidation of lipid ingredients, colorants, and flavors of food products such as nuts, confectionary, and fried foods (Baldwin et al. 1997). This property is also used to retard the respiration rate of fruits and vegetables (Baldwin et al. 1995). Many climacteric fruits and vegetables can be coated with edible film-forming materials to slow down their respiration rate (Park 1999; Amarante and Banks 2001).

High-fat meat and fish products, such as sausages and fillets, can be protected from oxidation using O<sub>2</sub> barrier edible coatings. Moisture loss is the most critical quality degradation factor in fresh produce (Guilbert et al. 1997). Moisture-barrier properties of edible films and coatings can protect fresh fruits and vegetables from dehydration. The moisture-barrier property can also be utilized to prevent moisture migration between heterogeneous food product ingredients, for example between raisins and breakfast cereals (Kester and Fennema 1986), pie fillings, and crusts, etc. Different materials used in edible-coating formulation have been reported in literature (Kester and Fennema 1986; Gennadios and Weller 1990; Krochta et al. 1994; Krochta and De Moulder Johnston 1997; Debeaufort et al. 1998; Zhao and McDaniel 2005; Lin and Zhao 2007).

Some selected examples are listed below:

- Wheat gluten, whey proteins, corn zein, waxes (beeswax, carnauba, candelilla), cellulose derivatives and pectins for fruits, grains, and vegetables as O<sub>2</sub> and moisture barriers
- Waxes or fatty acids on fruits and vegetables to delay spoilage and to reduce water loss and, on cheeses, to prevent mold growth

- Gelatin, seaweed, and pectinate to eliminate dripping, and as moisture barriers on cheese, ice cream, and yogurt
- Corn zein, milk and whey proteins, waxes, and methyl cellulose in confections
- Cellulose derivatives on fried foods to reduce oil absorption
- Gelatin, carrageenan and alginate, whey protein, collagen, casein, and cellulose derivatives on poultry, beef, fish and seafood
- Acetylated monoglyceride and whey protein on frozen salmon to reduce moisture loss and lipid oxidation
- Casein, xanthan gum on peeled carrots to reduce dehydration and white blush formation
- Chitosan on strawberries to delay spoilage, and on tomatoes, to extend shelf-life
- Cellulose, chitosan, and sodium caseinate on bell peppers to reduce O<sub>2</sub> and CO<sub>2</sub> permeability
- Starch in coated prunes, strawberries, and nuts to prolong storage life
- Dextrin on apples to delay oxidative browning
- Corn zein on tomatoes to delay color changes and loss of firmness
- Wheat gluten, cellulose, soy protein, and whey protein applied individually on egg shells to improve shell strength and to reduce microbial contamination
- Starch, corn zein, whey protein, and acetylated monoglyceride on nuts to delay rancidity
- Soy protein on apples to retard changes in firmness and color
- Chitosan-based coatings to reduce drip loss on frozen-thawed raspberries
- Alginate on fresh meat, poultry, and precooked ground pork to reduce shrinkage, oxidative rancidity, moisture migration, and oil absorption
- Mixtures of stearic-palmitic acids and hydroxypropyl cellulose, and MC and palmitic acid coatings, as moisture barriers for heterogeneous foods (puree, cake, ice cream cones)

## 27.8 Incorporating Functional Ingredients into Edible Films and Coatings

A unique feature of edible films and coatings is their capacity to carry many functional ingredients, including antioxidants, antimicrobial agents, flavorings, and colorants. Integration of these ingredients can enhance food stability, quality, functionality, and safety. For instance, food microbial stability can be improved using edible-active layers, which act as surface-retention agents to limit the diffusion of food additives into the food core; these layers can be used with simultaneous treatments such as controlled atmosphere and refrigeration. In another example, high concentration of a chemical preservative incorporated into an edible coating may allow a decrease in the total amount of the preservative in the food, attaining similar performance.

### 27.8.1 *Antioxidant Edible Coatings*

Antioxidants, such as tocopherol and butylated hydroxytoluene (BHT) were integrated into edible coatings to inhibit lipid oxidation in precooked beef patties and fish muscle, respectively (Zhao and McDaniel 2005). The antioxidant effects of ascorbic acid in whey protein film coatings applied on roasted peanuts for the control of lipid oxidation were also studied by Min and Krochta. (2007). By measuring the peroxide values and the thiobarbituric acid reactive substances, it was shown that the addition of ascorbic acid to the coating retarded lipid oxidation in peanuts.

### 27.8.2 *Antimicrobial Edible Films*

The concept of active packaging was introduced with antimicrobial films and coatings. Edible films can serve as carriers of antimicrobials to extend product shelf-life and to reduce the risk of pathogen growth on food surfaces. Edible films have been developed that can reduce, inhibit, or delay the growth of microorganisms on the surface of coated foods. Excellent reviews on antimicrobial edible films have been published by different authors (Han 2000, 2002, 2003a, b; Franssen and Krochta 2003; Cagri et al. 2004).

**Chitosan:** A polysaccharide obtained by deacetylation of chitin, chitosan originates from crustacean exoskeleton and fungal cell walls. It has been widely used in antimicrobial films and coatings due to its property of inhibiting the growth of many pathogenic bacteria and fungi. In some fungi, chitosan can alter membrane functions by interacting with the strongly electronegative microbial surface, leading to changes in permeability, metabolic disturbances, and eventually death.

**Natural preservatives:** There has been a tendency in the last several years to use natural preservatives in edible-coating formulations. Some of the most commonly used preservatives and antimicrobials are:

- Organic acids (acetic, benzoic, lactic, propionic, sorbic)
- Parabens (methyl paraben)
- Fatty acid esters (glyceryl monolaurate)
- Polypeptides (lysozyme, peroxidase, lactoferrin, nisin)
- Nitrites (potassium nitrite, sodium nitrite)
- Sulfites (potassium sulfite, sodium sulfite)
- Natural preservatives (essential oils, spices, extracts such as cinnamon, sage, allicin, and liquid smoke)
- Other preservatives (atamycin, EDTA)

These agents can be used in protein-, polysaccharide-, and lipid-based edible coatings and films.

**Benzoic acid and sodium benzoate:** These two are among the first chemical preservatives permitted in foods by the Food and Drug Administration. Benzoic acid ( $pK_a = 4.20$ ) and sodium benzoate are generally regarded as safe preservatives at levels up to 0.1%. Sodium benzoate and benzoic acid are inhibitory to mold, yeast, and pathogenic and psychrotrophic spoilage bacteria. Sodium benzoate is one of the most commonly used antimicrobials in edible films because it is soluble in most film solutions and remains active after film preparation. Organic acids are more effective in the undissociated form and, therefore, the antimicrobial activity is related to pH. At  $pH = 4.0$ , 60% of benzoic acid is undissociated (Cagri et al. 2004). This antimicrobial can be incorporated into MC, collagen, and chitosan films, all of which have a relatively low pH. Edible films containing benzoic acid and its sodium salt are adequate for acidic foods like cheeses and fermented meat products.

**Sorbic acid:** This is a straight chain unsaturated monocarboxylic acid; the carboxyl group reacts to form calcium, sodium, or potassium salts. Potassium sorbate is highly soluble in water (58.2% at 20°C). Edible films containing potassium sorbate are most effective at  $pH < 6.0$ . Sorbic acid salts were widely used as antimicrobial agents in carbohydrate- and protein-based edible films formulated with MC, WPI, and chitosan. Films containing sorbates have been tested against spoilage bacteria, pathogenic bacteria, yeasts, and molds in laboratory media.

**Propionic acid:** This acid is commonly used as a food preservative because of its wide spectrum of activity. Propionic acid, which is a monocarboxylic acid, is produced by *Propionibacterium freudenreichii subsp. shermanii*. Antimicrobial activity of propionates is also pH dependent. Although this acid is primarily active against molds, some yeasts and bacteria are also inhibited. The amount of propionate used in foods is generally less than 0.4% (Cagri et al. 2004).

**Parabens:** Produced by esterification of the carboxyl group of benzoic acid, most parabens are active at  $pH 3.0$ – $8.0$ . The methyl, propyl, and heptyl parabens can be used as food preservatives in most countries. Parabens can also be used effectively in a wide range of foods; they are generally more active against molds and yeasts than bacteria. For complete inhibition of bacteria and fungi, concentrations of esters of p-hydroxybenzoic acid ranging between 0.033 and 1.0 mg/L are necessary (Cagri et al. 2004).

**Free fatty acids:** Low concentrations of long-chain fatty acids are inhibitory to microorganisms, especially gram-positive bacteria and yeasts. Saturated fatty acids with chain lengths of C12 to C16 and C10 to C12 have the most antimicrobial activity against bacteria and yeasts, respectively (Cagri et al. 2004). Fatty acids are more active at low pH (5.0), and both fatty acids and monoglycerides are inhibitory to many bacterial species. Monoesters of glycerols and esters of sucrose are more antimicrobial than their corresponding free acids.

**Acetic acid and sodium diacetate:** Both are active against various spoilage and pathogenic bacteria and have been used in many foods. Like other organic acids, acetic acid can be used to acidify edible coatings containing chitosan, alginate, collagen, and WPI.

**Lactic acid:** This acid is used for improving and controlling the quality and microbial stability of foods. Lactic acid sprays (1–3% solutions) have been widely

used to sanitize meat carcass surfaces, with gram-negative psychrotrophs generally being more sensitive to treatment than gram-positive organisms. Lactic acid can be used as an acidulant in chitosan and collagen films.

**Nisin:** The first bacteriocin used in the food industry, nisin is one of the most investigated bacteriocins in antimicrobial edible films. It is produced by fermentation using *Lactococcus lactis* subsp. *Lactis*. It was recognized as a safe biological food preservative by a joint FAO/World Health Organization commission on food additives in 1968, and accepted by the Food and Drug Administration in 1988. Nisin is a polycyclic peptide antibacterial with 34 amino acids residues. It is obtained commercially from natural substrates, including milk, and is not chemically synthesized. It inhibits gram-positive spoilage and pathogenic bacteria (*Listeria monocytogenes*, *Staphylococcus aureus*, *Clostridium botulinum*). Incorporation of nisin into packaging materials inhibits the growth of gram-positive bacteria but does not inhibit gram-negative bacteria such as *E. coli*.

Nisin can be incorporated into the film suspension or applied directly to the film surface after casting. Various nisin-containing protein-based films (e.g., whey protein, corn zein, wheat protein, soy protein) have been assessed for antimicrobial activity against gram-positive bacteria such as *L. monocytogenes* and lactic acid bacteria (Cagri et al. 2004).

**Pediocins:** Produced by *Pediococcus acidilactici*, this group of bacteriocins can be used in edible films and coatings because of their wide spectrum of antimicrobial activity and effectiveness over a wide range of pH values and temperatures. Antimicrobial activity of pediocin is retained at 100°C, decreasing at 121°C, and is most evident at pH values between 4 and 7, with substantial losses at pH 3 and 9. Pediocin remains active following treatment with lipase, phospholipase C, lysozyme, DNase, or RNase, but its activity is destroyed by protease, papain, and a-chymotrypsin (Cagri et al. 2004).

**Lysozyme:** This enzyme is found in egg whites, human tears, and other secretions. It is responsible for breaking down the polysaccharide walls of many kinds of bacteria and thus it provides some protection against infection. The enzyme is formed by 129 amino acids cross-linked by four disulfide bonds. Dried egg white, the commercial source for lysozyme, contains about 3.5% lysozyme. It is heat stable (100°C) at pH 5.3 but inactivated at lower temperatures when pH is increased. Lysozyme is most active against gram-positive bacteria (Cagri et al. 2004).

**Spices, herbs, and essential oils:** Essential oils are responsible for the odor, aroma, and flavor of spices and herbs. These compounds can be added to edible films to modify flavor, aroma, and odor and to introduce antimicrobial properties. Films containing these ethanol-soluble compounds show activity against both gram-negative and gram-positive bacteria. Essential oils (cardamom, cinnamon, cloves, coriander, garlic, nutmeg, oregano, parsley, rosemary, sage, thyme, etc.) are inhibitory to various spoilage or pathogenic bacteria, molds, and yeasts. However, the use of these oils as food additives is limited due to their strong flavor.

**Lactoferrin (lactotransferrin):** This iron-binding glycoprotein is present in bovine milk. It inhibits the growth of some bacteria; however, other bacteria with

low iron requirements, such as lactic acid bacteria, are not inhibited by lactoferrin. *Pseudomonas fluorescens*, *Enterococcus faecalis*, and *Bifidobacterium bifidum* strains are highly resistant to this peptide. The mode of action of lactoferrin has not been fully elucidated, but presumably it alters membrane permeability because of its cationic nature.

**Liquid smoke:** Used in processed meats, sausages, and cheeses, liquid smoke contains phenols and acetic acid, which are bactericidal at relatively low concentrations. It can inactivate common food-borne pathogens, including *E. coli*, *Salmonella*, *Staphylococcus aureus*, and *L. monocytogenes*. It is generally very acidic and has antimicrobial, antioxidant, and flavor properties, making it a potentially attractive edible coating additive. However, incorporation of liquid smoke has only been studied for edible collagen casings.

Cha and Chinnan (2004) reviewed important aspects of biopolymer-based antimicrobial packaging. The incorporation of several antimicrobial agents (EDTA) and grapefruit seed extract into films, made of Na-alginate and K-carrageenan, produced a strong inhibition against *Micrococcus luteus*, *L. innocua*, *Salmonella enteritidis*, *E. coli*, and *S. aureus*; sodium alginate-based films, with the addition of nisin, lysozyme, and EDTA, showed the strongest inhibition against the same bacterial strains. Chitosan films containing nisin showed a high antimicrobial activity against *M. luteus*. Whey protein film coating was used as a vehicle for combining antimicrobials (grape seed extract, nisin, malic acid, and EDTA) (Cha and Chinnan 2004).

The primary advantage of antimicrobial edible films is that the inhibitory agents in these films can be specifically targeted to postprocessing contaminants on the food surface. It is important to predict and control the preservative release (Torres and Karel 1985; Torres et al. 1985; Redl et al. 1996). The diffusion rate of the antimicrobial into the product is partially controlled by agents incorporated into the film. The edible film matrix entraps the antimicrobial and decreases its diffusion during storage; thus, lower levels of preservative addition would be needed in an edible coating to achieve a targeted shelf-life, as compared to spraying the antimicrobial on the surface.

Controlling the antimicrobial release from edible films is very important; therefore, measurement of the diffusion coefficients of the antimicrobials in edible films should be carried out under controlled conditions (Vojdani and Torres 1990; Brody et al. 2001; Min et al. 2008). While antimicrobial coatings may extend the shelf-life and protect food products, it must be emphasized that such advantages are never substitutes for proper handling, storage, and good manufacturing practices.

### 27.8.3 Edible Coatings as Carriers of Nutraceutical Ingredients

There is an increasing consumer interest in the health-enhancing roles of specific foods and physiologically active food components (nutraceuticals or functional foods). Edible coatings could provide an excellent way to enhance the nutritional

value of foods by carrying nutrients or nutraceuticals that are lacking, or present in low quantities (Mei and Zhao 2003; Park and Zhao 2004; Park 2004). Zhao and Mc Daniel (2005) integrated high concentrations of minerals and vitamins into edible films and coatings.

Mei et al. (2002) developed xanthan gum coatings containing a high concentration of calcium and vitamin E, and applied such coatings to fresh baby carrots. Han et al. (2004) applied chitosan coatings with high concentrations of calcium or vitamin E on fresh and frozen strawberries and red raspberries.

## **27.9 Edible Coatings to Improve Quality and Extend Shelf Life of Foods - Case Studies**

### ***27.9.1 Edible Coatings Acting as Oil Barriers in Fried Products***

One of the applications of coatings is to reduce the OU of products during deep-fat frying (Balasubramaniam et al. 1997). The excess of fat in the diet has been linked to coronary heart disease; thus, coatings applied to food before frying can help reduce health problems associated with fat consumption. Frying occurs during the immersion of the product in oil at temperatures higher than the boiling temperature of water (150–200°C). Deep-fat frying is a complex process involving simultaneous heat and mass transfer (Bertolini Suarez et al. 2008)

Various hydrocolloids, especially cellulose derivatives, can be used as edible coatings to decrease oil content during frying. Williams and Mittal (1999) found that MC films showed the best barrier properties, reducing fat uptake more than hydroxypropylcellulose and gellan gum films. Cellulose derivatives, including MC and HPMC exhibit thermal gelation. When suspensions are heated a gel is formed that reverts back below the gelation temperature, and the original suspension viscosity is recovered. MC forms a gel at high temperatures; hydrophobic polymer chain interactions are involved in the thermal gelation process with a predominance of intermolecular hydrogen bonding over intramolecular hydrogen bonding within the cellulose ether. Extensive research on edible coatings from cellulose derivatives was carried out in our laboratories (García et al. 2002, 2004a; Bertolini Suarez et al. 2008). The objectives of the experiments were to analyze the coating performance in reducing OU in French fries and fried pastry.

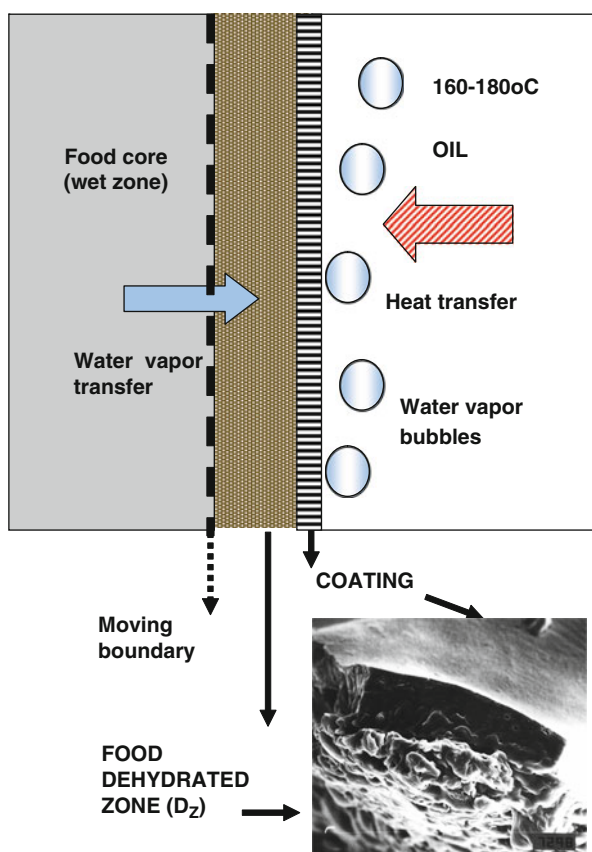
Aqueous suspensions of 1% Methocel A4M (based on MC), 2% K100LV, and 2% E15LV (based on MC and HPMC), with and without sorbitol (0.25–1%), were used for coating applications. The rheological behavior of the suspensions and the viscoelastic moduli were measured with an oscillatory rheometer. MC suspensions were selected for coating application because they showed the highest elastic ( $G'$ ) and complex ( $G^*$ ) moduli.

Model dough discs (60 mm dia. and 7 mm thick) were prepared with refined wheat flour and distilled water. Samples were dipped in coating suspensions for 30 s



and drained. Coated and uncoated samples were fried in sunflower oil under controlled temperature conditions ( $160 \pm 0.5^\circ\text{C}$ , previously determined by a sensory panel). Surface color (lightness and chromaticity parameters), firmness (instrumental texture analysis), and OU of the control and coated fried samples were measured at different frying times.

During frying of both the coated and uncoated samples a moving boundary was produced within the product, separating the dehydrated zone from the wet core (Fig. 27.1). A protective layer (approximately  $15\ \mu\text{m}$  thick) was formed on the surface of the coated samples during the initial stages of frying, due to MC thermal gelation above  $60^\circ\text{C}$ ; the coating was becoming dehydrated and remained attached to the surface of the product. SEM observations showed the integrity of the MC layer and good adhesion of this coating to the fried product (Fig. 27.1); the addition of a plasticizer (sorbitol) in the coating formulation was necessary to achieve coating integrity and uniformity.



**Fig. 27.1** Schematic description of frying process in a coated product, and scanning electron microscopy (SEM) micrograph of the methylcellulose coating applied on a fried dough sample

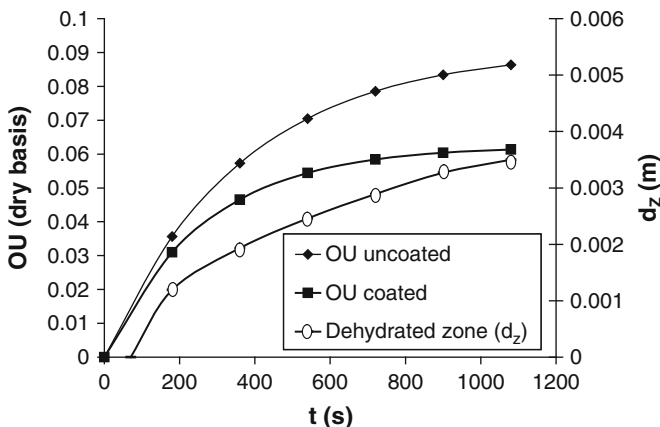
OU was determined by combining a preliminary batch extraction process with a semicontinuous extraction in a Soxhlet; the extracted lipid phases were analyzed by HPLC (García et al. 2002, 2004a). MC coatings (1% A4M and 0.75% sorbitol) were effective in decreasing oil content without affecting the water content of the samples (Bertolini Suarez et al. 2008); the OU in coated samples was 30% lower than in the uncoated ones at the final frying time (Fig. 27.2).

Concerning the quality attributes of the fried dough, differences between the instrumental color parameters and firmness values of coated and uncoated samples were not significant, during the frying process ( $t < 720$  s); besides, the panelists could not distinguish the differences between the control and coated samples. These results demonstrate the effectiveness of the MC coating to reduce OU without affecting the sensory attributes of the samples (García et al. 2002).

The frying process was mathematically modeled (Bertolini Suarez et al. 2008) and experimentally validated; temperature profiles and water-content distributions were predicted by the model. Nonlinear-coupled energy and mass transfer partial differential equations, under unsteady state conditions, considering a moving boundary, were numerically solved considering both thermal and physical properties change with temperature and moisture content. The model simulated appropriately the experimental data of temperature and water content during the different frying stages.

Microstructural changes are produced during the frying process; the heat and mass transfer model predicted the position of the vaporization front and the thickness of the dehydrated zone ( $d_z$ ) as a function of frying time (Fig. 27.2). In addition, water content correlated linearly with the vaporization front position corresponding to the thickness of the humidity core (Bertolini Suarez et al. 2008).

During frying the vapor leaves voids in the product and allows for the oil to enter later; OU is mainly produced when the product is removed from the frying medium.



**Fig. 27.2** Effect of methylcellulose coating on oil uptake in fried dough. The presence of coating decreases oil uptake. Oil uptake curve can be closely related to increase of dehydrated zone thickness ( $d_z$ )

OU is affected by the microstructure of the dehydrated zone and is dependent on the balance between the oil retained on the surface and the oil drained after retrieval of the product from the oil bath; the oil retained by the surface penetrates into the pores left by the water evaporation. The amount of oil retained at the sample surface is limited, due to the oil surface wetting, the property related to the interfacial tension that governs these phenomena. Figure 27.2 shows similar shapes for the OU curves (experimental data) and the thickness of the dehydrated zone (predicted values). From these results, OU was linearly correlated with the thickness of the dehydrated zone during the initial frying period (Bertolini-Suarez et al. 2008).

Other results obtained in our laboratory on using edible coatings to reduce OU during frying (4 min/180°C) showed a reduction of 40.6% in oil content of French fries (0.7 × 0.7 × 5 cm) using MC (1%) and sorbitol, without causing detrimental effects on the quality attributes (surface color and texture).

### ***27.9.2 Starch-Based Edible Coatings to Prolong Storage Life of Refrigerated Highly Perishable Fruits***

The strategies explored to extend postharvest life of fruits should consider several challenges, such as extending maturation and senescence periods, reducing dehydration, and reducing onset and rate of microbial growth. Edible films and coatings could offer solutions to all such challenges simultaneously, the use of which has been reported by several researchers for different vegetables and fruits (Drake and Nelson 1990; El Gaouth et al. 1991; Park 1999, 2003, Park et al. 2005). The use of coatings with selective permeabilities on fruits has also been shown to have an influence on fruit physiology in retarding ripening and postharvest metabolism, thus, extending the fruit's storage life (Baldwin 1994).

Edible coatings can be made from food materials regarded as GRAS, such as proteins, lipids, cellulose derivatives, starch, and other polysaccharides. Composite films can be formulated to combine the advantages of both lipid and hydrocolloid components (Guilbert 1986). In the case of fruit and vegetable conservation, the lipid component in coating formulation can serve as a good barrier against water vapor, while the hydrocolloid component can provide a selective barrier against O<sub>2</sub> and CO<sub>2</sub> and the necessary supporting matrix. The functional, organoleptic, nutritional, and mechanical properties of an edible film can be modified with the addition of various chemicals in minor amounts, such as plasticizers, antimicrobial agents, and lipids.

Strawberries (*Fragaria × ananassa*), a typical soft fruit variety, have high physiological postharvest activity that transforms them into a highly perishable fruit with a low period of commercialization, which is mainly limited by fungal infection. As a consequence, strawberries have short ripening and senescence periods, which makes coating this fruit a good alternative for extending storage life. In this case, the effect of adding plasticizer and lipid and antimicrobial agents to

starch-based coatings, on their performance, applied to strawberries is discussed (García et al. 1998a, b, 2001).

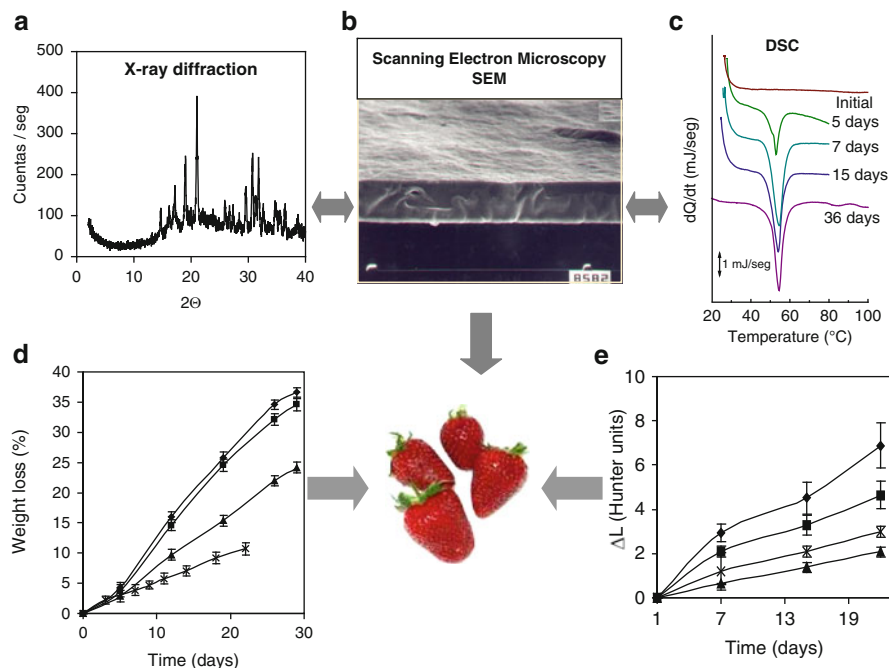
Strawberries (*Fragaria × ananassa*, cv Selva) at the commercial ripening stage (75% red color), grown on a local farm in greenhouses, were harvested and immediately treated. The strawberries were dipped in chlorinated water (0.25 g Cl<sub>2</sub>/L), dried with air, dipped in coating suspensions, and dried again with air (1.2 m/s, 20°C and 84.8% RH). Uncoated fruit (control) was treated similarly, replacing immersion in starch suspensions with distilled water. Samples were stored in a cold chamber at 0°C and 84.8% RH.

Two different starches were used for coating formulations: commercial corn starch with 25% amylose and a high amylose corn starch; and amylomaize (Amylomaize VII, Amaizo, USA) with 65% amylose. Starches were cold-gelatinized using sodium hydroxide; suspensions were neutralized with H<sub>3</sub>P<sub>0</sub>4. The gelatinization method was selected to comply with the dipping procedure for coating application, and the heat-sensitive characteristic of strawberries. Glycerol or sorbitol was added as plasticizer at a concentration of 20 g/L. Additionally, 2 g/L sunflower oil were added and emulsions were homogenized at 7,800 rpm. Potassium sorbate (2 g/L) and citric acid (to reach pH 4) were also included in the formulations as antimicrobial agents.

Rheological behavior of coating suspensions was analyzed. The suspensions showed pseudoplastic behavior ( $n < 1$ ) and the Power law rheological model satisfactorily fit experimental data ( $r^2 > 0.96$ ). Plasticizer and lipid addition to corn and amylomaize suspensions decreased the flow behavior index and increased the consistency index. Apparent viscosity of corn starch suspensions also decreased with plasticizer and lipid addition.

Microstructure characterization of the coatings contributed to understanding the performance of the different formulations. This characterization included light microscopy observation (stereomicroscopy) and SEM. The stereomicroscope was used to determine coating thickness (40–50 μm) and to check coating uniformity; coated samples were stained with iodine solution to improve coating visualization. Polarized light microscopy observations revealed that starch was totally gelatinized in the filmogenic suspensions (García et al. 1998a, b). SEM observations showed that plasticizer addition was necessary for film integrity, to avoid pores and cracks. Plasticized films containing lipid (Fig. 27.3b) exhibited smooth surfaces and a compact structure, thereby indicating the homogeneous dispersion of lipids in the film matrix (García et al. 1999, 2000a, b).

Modifications of the crystalline structure of films stored at 20°C and 63.8% RH were evaluated by DSC and x-ray diffraction, using K $\alpha$  Cu radiation ( $\lambda = 1.5418 \text{ \AA}$ ). X-rays detected the starch recrystallization process during storage. In the case of starch-based films, peak width slightly decreased and peak intensities increased with storage time, showing a tendency toward increase in crystallite size, which can be attributed to a slow recrystallization process (García et al. 2000b, 2001). In the case of starch coatings obtained by alkaline treatment, X-ray diffraction patterns showed that both corn and amylomaize films attained a common crystalline structure of higher stability with storage time, exhibiting an A-type starch pattern



**Fig. 27.3** Application of starch-based coatings to prolong storage life of refrigerated strawberries. (a, b, c) Coating characterization: (a) X-Ray diffraction pattern of amylo maize coating containing 20 g/L glycerol and 2 g/L sunflower oil; (b) SEM micrograph of corn starch-based films with 20 g/L sorbitol and 2 g/L sunflower oil. Magnification: 100 μm between marks; (c) DSC thermograms of amylo maize film stored at 20°C and 63.8% relative humidity. (d, e) Effect of coating formulation on quality parameters of refrigerated strawberries during storage at 0°C and 84% RH; (d) weight losses, (e) lightness differences. Control samples, uncoated fruits, (○); composition of coating formulation: (□) amylo maize without plasticizer; (Δ) amylo maize with 20 g/L sorbitol; and (◇) amylo maize with 20 g/L sorbitol and 2 g/L sunflower oil. Bars indicate standard errors

(Fig. 27.3a). Films without plasticizer showed higher crystallinity (higher peaks) than films containing plasticizer, which showed a larger amorphous zone and lower peaks.

Amylo maize films containing neither plasticizer nor lipid showed a higher crystallinity (higher numbers of peaks) than films containing plasticizer and lipid. These results agree with the DSC studies that showed endothermic transitions with lower peak temperatures and lower  $\Delta H$  in films with plasticizer and lipid than in control films. Presence of lipid and plasticizer in film formulations did not alter the x-ray pattern of the most stable structure developed in stored control films; peaks maintained their initial positions.

Crystalline evolution of the film matrix during storage was also evaluated by DSC; this technique also allowed determining the  $T_g$ . Starch coatings obtained by alkaline treatment, showed an endothermic transition with a peak temperature around 50°C during storage; this peak became narrower and its temperature and the corresponding enthalpy ( $\Delta H$ ) increased with storage time (Fig. 27.3c).

**Table 27.1** Barrier properties of starch-based coatings: water vapor permeability (WVP) and gaseous permeabilities

Starch-based coating	Additives	WVP $\times 10^{10}$ ( $\text{g m}^{-1} \text{s}^{-1} \text{Pa}^{-1}$ )	CO <sub>2</sub> permeability <sup>a</sup> $\times 10^9$ ( $\text{g m}^{-1} \text{s}^{-1} \text{Pa}^{-1}$ )	O <sub>2</sub> permeability <sup>a</sup> $\times 10^9$ ( $\text{g m}^{-1} \text{s}^{-1} \text{Pa}^{-1}$ )
Corn	Without additives	3.68 $\pm$ 2.24 <sup>b</sup>	29.21 $\pm$ 13.89	1.59 $\pm$ 0.30
	G	2.57 $\pm$ 1.04	5.69 $\pm$ 0.97	0.46 $\pm$ 0.05
	G + SO	1.92 $\pm$ 0.47	5.87 $\pm$ 0.58	ND
	S	1.75 $\pm$ 0.14	4.19 $\pm$ 0.81	0.24 $\pm$ 0.03
	S + SO	1.22 $\pm$ 0.11	4.72 $\pm$ 0.65	ND
Amylo-maize	Without additives	2.62 $\pm$ 1.39	28.05 $\pm$ 7.37	2.64 $\pm$ 0.25
	G	2.14 $\pm$ 0.75	3.85 $\pm$ 1.28	0.321 $\pm$ 0.02
	G + SO	1.76 $\pm$ 0.37	4.39 $\pm$ 0.90	ND
	S	1.21 $\pm$ 0.15	2.96 $\pm$ 0.46	0.23 $\pm$ 0.03
	S + SO	0.97 $\pm$ 0.08	3.43 $\pm$ 0.21	ND

G glycerol (20 g/L), S sorbitol (20 g/L), SO sunflower oil (2 g/L), ND not determined

<sup>a</sup>At standard pressure and temperature conditions

<sup>b</sup>Value  $\pm$  standard deviation

Barrier properties, which include water vapor (WVP), and O<sub>2</sub> and CO<sub>2</sub> permeabilities of starch-based coating formulations, were determined on films obtained by casting (García et al. 2000b). The addition of lipids to starch-based films decreased WVP due to its hydrophobicity (Table 27.1). This is an important property in the case of fresh fruits and vegetables because composite coatings retard moisture loss and subsequent shriveling of fresh products. Table 27.1 shows that O<sub>2</sub> permeabilities were much lower than those of CO<sub>2</sub>, indicating a selective action of these films on gas permeabilities. Amylomaize films with higher amylose content showed higher crystallinity and therefore, lower permeabilities than corn starch films. The development of edible coatings with selective gas permeabilities is a good example of active packaging's role in controlling respiratory exchange and improving the conservation of fresh vegetables. Moreover, the addition of lipid, which is necessary to reduce WVP, maintained the selective gas permeability property since CO<sub>2</sub> and O<sub>2</sub> permeabilities did not differ from those of plasticized starch-based films.

The effect of coatings on the quality attributes of refrigerated strawberries was analyzed and the following results were obtained:

**Weight loss:** The same fruits were weighed at the beginning of the experiment and during storage; weight loss was expressed as percentage loss of initial weight. All the tested coatings showed a beneficial effect on weight loss (Fig. 27.3d). Weight loss in fruits coated with starch formulations, without plasticizer, was similar to those of control fruits due to pores and cracks. Coatings with sorbitol led to significantly lower fruit weight loss than glycerol, regardless of starch type. However, weight loss was unacceptable after 3 weeks of storage for coated fruits without lipid. The addition of sunflower oil was necessary to reduce weight loss

significantly ( $P < 0.05$ ) and to increase storage life, provided the microbial counts were below the established limit ( $10^6$  CFU/g fruit), even at 28 days of storage. The maximum weight loss reduction at 28 days of storage was 63.2%, obtained with coating formulations that included sunflower oil (García et al. 2001).

**Texture changes:** Important modifications in texture can occur in fruits and vegetables during storage, determining the postharvest storage life of the product. Fruit softening is generally attributed to degradation of the cell wall components, mainly pectins, due to the activity of specific enzymes such as polygalacturonase. The rate and extension of firmness loss during ripening of soft fruits, such as strawberries, is one of the main factors; for both control and coated fruits, the breaking force decreased as a function of storage time. The formulations that minimized weight loss maintained better firmness, since this attribute is also highly influenced by water content.

**Color changes:** The effect of coatings on surface color modifications in strawberries was also analyzed, because this quality attribute may determine consumer acceptability of the fruit. Lightness (L) and chromaticity parameters (a and b) were recorded at 1st, 8th, 15th and 22nd days of storage. Both plasticizers significantly delayed surface color development. Formulations containing glycerol gave better surface color results compared to those containing sorbitol. Oil addition did not modify surface color results significantly, regardless of plasticizer used in formulation (Fig. 27.3e).

Physiological parameters of fruit, such as titratable acids, pH, anthocyanin, and sugar content are good indicators of maturation and senescence. Coatings may alter natural physiological behavior, modifying the organoleptic characteristics of fruit such as color, taste, or flavor. In coated strawberries, these physiological parameters were slowed down but reached commercially acceptable values. The results indicate that starch-based coatings retard the metabolic reactions, and thus, senescence of coated refrigerated fruits is delayed. This result is attributed to the differential gaseous permeability of films. The  $O_2$  and  $CO_2$  barriers lead to a reduction in respiration rate by limiting the exposure to ambient  $O_2$ , increasing internal  $CO_2$ , delaying ripening, senescence, and extending storage life of treated fruits (Baldwin 1994; Avena-Bustillos et al. 1997; García et al. 1998a, b, 2001).

In microbiological analysis, surface microbial growth is the main cause of spoilage for many food products. Microorganisms growing on strawberries were mainly yeasts, molds, and sugar-fermenting bacteria. Aerobic mesophilic and psychrotrophic bacteria, molds and yeasts, and coliform microorganisms were analyzed at different storage times. Viable counts were expressed as log CFU/g fruit. Microbial counts in unplasticized, coated strawberries did not differ significantly from the control. Coatings with potassium sorbate, a well-known effective antifungal agent, significantly decreased ( $P < 0.05$ ) yeast and mold counts on coated strawberries. Since the undissociated form is the active antimicrobial agent of sorbic acid, citric acid at fruit pH was added to coating formulation to increase potassium sorbate effectiveness. The addition of 0.2 g/L potassium sorbate

and citric acid to the starch-based coating was the most effective formulation for decreasing microbial counts. This active coating allows using lower amounts of preservatives, maintaining the same antimicrobial efficacy as traditional techniques, with consequent health and economic benefits.

The storage life of a refrigerated fruit, defined as the time necessary to reach  $10^6$  CFU/g, was determined for the different formulations. At 0°C, the storage life of the uncoated fruit was 14 days; coatings with sorbitol extended fruit storage life to 21 days. At maximum storage time assayed (28 days), formulations with sorbitol and potassium sorbate showed microbial counts below  $10^6$  CFU/g in fruit. The addition of potassium sorbate enhanced the effectiveness of starch coatings; moreover, the addition of citric acid (to reach pH 4) increased the antimicrobial action of potassium sorbate, leading to a shelf-life of more than 28 days for coated strawberry.

The presence of antimicrobial agents in the coating provided a local high and effective concentration of the preservative. Edible films and coatings act as surface-retention agents and limit preservative diffusion in the food core. This allows reducing the total amount of preservative added to a food compared to traditional methods (e.g., dipping in chemical preservative solutions) (García et al. 1998a, b, 2001).

In conclusion, starch-based coatings with selective gaseous permeabilities proved to extend the storage life of refrigerated strawberries. Plasticizer and lipid addition improved coating performance by decreasing WVP. Weight losses were reduced, color changes delayed, and firmness of tissue and fruit appearance improved. Modifications of the physiological parameters of the fruit were slowed down but reached commercially acceptable values. Starch-based coatings containing potassium sorbate and citric acid helped decrease microbial growth; as a result, shelf-life of the fruit was extended to more than 28 days compared with 14 days for uncoated fruits.

## 27.10 Final Remarks

Edible films and coatings enhance the quality of food products, protecting them from physical, chemical, and biological deterioration, which results in extended shelf-life and improved safety. They can be used on fruits, vegetables, seafood, meats, and confectionery products. Edible coatings can help retain or improve food product quality by:

- Forming an efficient barrier to prevent moisture loss.
- Delaying ripening process in vegetables, through selective permeability to gases that affects postharvest metabolism, extending the storage life.
- Reducing OU in frying process.
- Adding vitamins or other functional ingredients to enhance quality.



- Incorporating active additives such as antimicrobial agents and antioxidants. Active components are located at the surface of the product, where their action is required. Coating matrix limits diffusion into the core of the food and thus, minor additive concentrations are needed compared to bulk addition.

The factors contributing to renewed interest in the development of edible coatings include:

- Consumer demand for safe and high quality foods, with longer shelf-life.
- Interest of food processors in new storage techniques.
- Opportunities for creating new markets for film-forming ingredients derived from under-utilized agricultural commodities.

Successful growth in the area of edible films will require strong interaction between food technologies and polymer science. Collaborative research work will allow edible coatings to be used in target applications. Moreover, the development of new technologies (functionalization, cross-linking, etc.) to improve the film properties (control release, bioactivity protection, resistance to water etc.) of active packaging and coatings is a major focus for future research.

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