The surface properties of biopolymer-coated fruit: A review

Propiedades de superficie en frutas recubiertas con biopolímeros: revisión

D. Moncayo[1], G. Buitrago[2] and N. Algecira[3]

ABSTRACT

Environmental conservation concerns have led to research and development regarding biodegradable materials from biopolymers, leading to new formulations for edible films and coatings for preserving the quality of fresh fruit and vegetables. Determining fruit skin surface properties for a given coating solution has led to predicting coating efficiency. Wetting was studied by considering spreading, adhesion and cohesion and measuring the contact angle, thus optimising the coating formulation in terms of biopolymer, plasticiser, surfactant, antimicrobial and antioxidant concentration. This work reviews the equations for determining fruit surface properties by using polar and dispersive interaction calculations and by determining the contact angle.

Keywords: Edible coating, edible film, contact angle, wettability.

RESUMEN

La elaboración de películas y recubrimientos con materiales naturales y biodegradables, disminuyen los daños ambientales comparados con los ocasionados por materiales comunes de plásticos sintéticos. Las formulaciones con biopolímeros como celulosa, gomas, almidones o proteínas; permiten conservar la calidad de las frutas y los vegetales frescos. La determinación de las propiedades que tiene la superficie de la piel de la fruta y la solución de recubrimiento, permiten predecir la eficiencia del proceso. El estudio del fenómeno de humectabilidad, considerando el trabajo de expansión, cohesión, adhesión y medición del ángulo de contacto, logra optimizar la formulación de recubrimiento en cuanto a concentraciones de biopolímeros, plastificantes, surfactantes, antioxidantes, etc. Este trabajo es una revisión de las ecuaciones que permiten determinar las propiedades superficiales de las frutas, considerando el cálculo de las interacciones polares, dispersivas y la determinación del ángulo de contacto.

Palabras clave: recubrimiento comestible, película comestible, ángulo de contacto y humectabilidad.

Introduction

Fruit and vegetables are live tissues having high moisture content (60%-95%) and which lose water and continue respiration thereby producing heat and water at the expense of food reserves (Mishra and Gamage, 2007). Fresh products cannot continue replenishing carbohydrates or water after harvesting. Plants use stored starch or sugar in respiration and will stop when such reserves become exhausted. Consequently, ageing begins, culminating in product death and decay (FAO, 1993).

Decay is primarily caused by weight-loss, not only through direct quantitative loss but also through the deterioration of appearance, textural quality (softness, loss of turgidity and juiciness), and nutritional quality. Transpiration rate (evaporation of water from plant tissues) is influenced by internal or intrinsic factors (morphological and anatomical characteristics, surface lesions, and maturity stage) (Barreiro and Sandoval, 2006) and environmental factors (temperature, relative humidity, air movement and atmospheric pressure) (Kader, 2002; Ulloa, 2007).

Packaging is widely used for preserving, distributing and marketing fruit and vegetables and is often used in combination with other preservation methods (Hoover, 1997). However, the disposal of packaging materials leads to ecological problems and additional recycling costs (Tzoumaki et al., 2009; Viña et al., 2007). Edible coatings are one of the most innovative strategies for extending fruit and vegetable shelf-life life; such coatings act as barriers to gas transport and produce similar effects to stor-age in a controlled atmosphere (Park, 1999).

Edible coatings and edible films are terms which are frequently interchangeably regarding food packaging; however, a distinction must be made between the two terms. A film is a thin skin which has been formed, for example, by casting a biopolymer solution separately from the food; this film can be later applied to the food.

[1] Diana Cristina Moncayo Martinez. Maestría en Ciencia y Tecnología de alimentos. Affiliation: BSc Chemical Engineering. Universidad Nacional de Colombia, Colombia. E-mail: dcmoncayoma@unal.edu.co

[2] Gustavo Buitrago Hurtado. BSc Chemical Engineering. Affiliation: Biotechnology Institute. Universidad Nacional de Colombia, Colombia. E-mail: gbuitragoh@unal.edu.co

[3] Néstor Ariel Algecira Enciso. BSc Chemical Engineering. Universidad Nacional de Colombia, Colombia. Affiliation: Facultad de Ingeniería Química y Ambiental, Universidad Nacional de Colombia, Colombia. E-mail: nalgecirae@unal.edu.co

By contrast, a coating is a suspension or an emulsion which is applied directly to the food surface, and later becomes transformed into a film *(Souza et al.,* 2010).

Edible coatings are usually made from materials such as proteins, lipids and polysaccharides; the main polysaccharides used in this are starches and modified starches, cellulose derivatives, chitosan, pectin, alginate and other gums (Hernandez-Izquierdo and Krochta, 2008; Tzoumaki et al., 2009). Thin edible films act as barriers to external elements (such as moisture, lipids and gasses) and improve mechanical properties during handling, transportation and may also serve as food additive carrier. Films also prevent the loss of and even increase volatile flavour production, thus extending product postharvest shelf-life (Azeredo et al., 2012; Durango et al., 2011; Guilbert et al., 1996; Oliva and Barbosa-Cánovas, 2005; Quintero et al., 2010; Ramos et al., 2013).

Most edible films and coatings contain at least one high molecular weight polymer. Large chain polymeric structures are required for creating matrices having suitable cohesive strength. The films so obtained usually have reduced flexibility and are porous and permeable to gasses, vapour and solutes. A uniform distribution of polar groups throughout the polymer chain increases coating material ability to form hydrogen bonds and participate in ionic interactions (Kester and Fennema, 1986).

Typical coating-forming methods would include spray-coating, and dipping (Dangaran et al., 2009). The choice of method de-pends on coating solution concentration and a coating's ability to form a thin layer which develops into a protective film on fruit surface, upon drying (Pavlath and Orts, 2009).

Edible coatings represent a novel approach to preserving the quality of characteristics such as fresh or minimally-processed products' colour, texture, antioxidant properties and freshness, thus extending product shelf-life (Ali et al., 2010; Chiumarelli and Hubinger, 2012; Das et al., 2013; Fan et al., 2009; Gounga et al., 2007; Qi et al, 2011; Robles-Sánchez et al, 2013). It has been reported that chitosan (a chitin derivative) can form excellent films having antimicrobial properties; it has been widely used in controlling weight-loss in fresh strawberries (*Fragaria x ananassa*) and raspberries (*Rubus idaeus*) (Han et al., 2004; Park, 1999; Ribeiro et al., 2007; Vargas et al., 2006), mango (*Mangifera indica*), litchi (Martínez-Castellanos, 2009), blueberries (Duan et al., 2011) and other fruit and vegetables (Lin and Zhao, 2007). Coat-ings consisting of caseinates and milk proteins provide an excel-lent barrier to oxygen and have been studied for controlling postharvest respiration in apples (*Malus sylvestris*) (Letien et al., 2001) and strawberries (Vachon et al., 2003).

The effectiveness of fruit and vegetables' edible coatings primarily depends on controlling coating solution wettability which affects a film's coating thickness (Park, 1999). The coating should be able to wet and spread uniformly over product surface and, upon drying, a coating should have suitable adhesion, cohesion and durability to function properly. Coating involves the wetting of the product to be coated by the coating solution, which may penetrate into the fruit skin (Hershko et al., 1996, Krochta and Mulder-Johnston, 1997), followed by possible adhesion between these two commodities.

The aim of this review is to describe the concepts for characterising the surface properties of fruit coated by biopolymers and to describe the effects of plasticiser type and concentration, as well as surfactant and polymer concentration, on these surfaces' coating wettability.

Surface properties

Optimum wettability requires the greatest possible area for contact between a solution and the surface to be coated, thus avoiding the disruption of air between the solution and the surface. The film is expected to be heavy and continuous on a non-porous surface; therefore, the film must have good cohesion. The adhesion between the final dried film and the food is extremely important; a layer of the dried, solid film should attach to the food, particularly in surface regions where discontinuities may exist. Wettability can be characterised in terms of two physical parameters: interfacial tension and contact angle (Marzo-Rojas, 2010).

Wettability

Wettability determines the spreading coefficient (Ws), the work of adhesion (Wa) and the work of cohesion (Wc). Adhesion strength promotes liquid expansion on a solid surface, and cohesive hardness promotes liquid contraction on a solid surface. Wettability behaviour is determined by the equilibrium between these forces. The surface tension of a coating solution is typically determined using a pendant drop technique, together with the Young-Laplace equation (Song and Springer, 1996).

Contact angle (θ)

Wettability property determination is essential in characterising the surface of packaging materials. The wettability of a solid surface can be determined in a relatively simple manner by contact angle measurement (Kiely and Olson, 2000). When a liquid drop is placed on a smooth, flat solid surface, the liquid forms a thin film or droplet (sessile drop) on the surface. The droplet makes a finite contact angle with the surface (Figure 1). The magnitude of the contact angle depends on the attractive force between the solid and the liquid and on the surface tension of the liquid (Tracton, 2005).
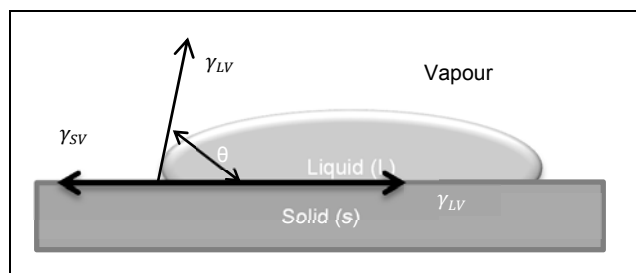

Figure 1. Surface contact angle and surface tension

The contact angle (θ) of a liquid droplet on a solid surface is determined by the mechanical equilibrium of the droplet regarding the action of three interfacial tensions: solid-vapour (γ_{SV}), solid-liquid (γ_{SL}) and liquid-vapour (γ_{LV}). The equilibrium spreading coefficient may be defined by equation (1-1) and can only have negative or zero values (Rulon and Robert,1995):

$$W_S = W_S = W_a - W_c = \gamma_{SV} - \gamma_{LV} - \gamma_{SL} \qquad (1\text{-}1)$$

where Wa and Wc are the work of adhesion and cohesion, as defined by equations (1-2) and (1-3), respectively:

$$W_a = \gamma_{LV} + \gamma_{SV} - \gamma_{SL} \qquad (1\text{-}2)$$
$$W_c = 2 * \gamma_{LV} \qquad (1\text{-}3)$$

For θ < 90 ° (or equivalently, for $\gamma_{SV} > \gamma_{SL}$), the liquid is said to wet the solid or the system is said to be wetting. For θ > 90°, the system is non-wetting; in this case, the liquid does not wet the

solid. An extreme case occurs when θ approaches zero: the liquid expands on the solid and shows perfect wettability (Palacios, 1999; Skurtys et al., 2010).

Contact angle value is affected by the following factors:

- Droplet size. Some studies have stated that the contact angle should be measured on two sides of a drop (Drelich, 1996; Greiveldinger and Shanahan, 1997). As the surface of a specimen is heterogeneous, the arithmetic average of at least nine different drop measurements should be used as the test result. Furthermore, readings must be taken quickly to avoid volume becoming reduced by evaporation. Kaelble and Cirlin (1971) recommended that:

- Drop volume should be 28 to 0.5 mm³;

- Temperature should be up to 8°C. Temperature changes have a small effect on surface free energy. Normal temperature fluctuations which may occur during tests do not have a strong effect on contact angle value (Zouvelou et al., 2007);

- Regarding surface impurity, the appearance of hysteresis is usually attributed to roughness, its chemical heterogeneity, and/or surface active impurities present in the liquid (Chibowski et al., 2002); and

- Concerning surface roughness (thermodynamic hysteresis), it is generally accepted that if Ra< 0.5 μm, the effect that roughness has on the contact angle is insignificant (Rudawska, 2009).

Surface free energy

Surface free energy is a thermodynamic quantity associated with the equilibrium state of the atoms in a surface layer of matter. This quantity is characteristic for each substance and represents the specific state of non-equilibrium intermolecular interactions in the boundary phase between two media. There are many methods for determining a liquid's surface free energy and various indirect methods are used for determining the surface free energy of solids. Examples of indirect methods would include Fowkes, Owens-Wendt and Wu's methods. Other methods include the Zisman method, the Newman method and van Oss-Chaudhury-Good methods: the latter has become increasingly popular (Rudawska and Jacniacka, 2009). The Owens-Wendt method consists of determining dispersion and polar surface free energy components on the basis of the Bethelot hypothesis (Rudawska and Jacniacka, 2009). The Fowkes' method divides surface energy into dispersive and polar components and uses a geometric mean approach for combining their contributions (Lu et al., 2012). The Neumann method is based on the assumption that there is a relationship between surface energy free of a solid, surface energy free of the liquid wetting the solid surface and surface energy free of the solid–liquid interface (Żenkiewicz, 2007).

Surface tension

The surface tension of a liquid is defined as the amount of energy needed to increase the surface per area unit (J• m⁻²). Surface tension may be equivalently defined as the normal force acting per unit length (N• m⁻¹) of the surface. Surface tension results from the action of intermolecular forces on the interface (i.e., the separation plane between the two phases) and depends on the nature of the liquid, the surrounding medium and temperature. The surface tension of a particle has a non-polar component (i.e. the Lifshitz-van der Waals (LW) force), a polar component (the Lewis acid-base (AB) force) and an electrostatic component (Han et al.,

2005). Surface tension is also used as a measure of adhesion properties and is determined by measuring the contact angle of a standard liquid on the surface. This method requires calculating the critical surface tension of the solids being studied (Casariego et al., 2008).

Cerqueira et al., (2009) derived a set of equations for calculating the surface energy of fruit in contact with a pure liquid, for known polar (γ_L^P) and dispersive (γ_L^D) interactions. If θ is the contact angle between the liquid and solid, the interactions can be expressed in terms of the reversible work of adhesion, W_a, as:

$$W_a = W_a^d + W_a^p \leftrightarrow W_a = 2\left(\sqrt{\gamma_S^d\gamma_L^d} + \sqrt{\gamma_S^p\gamma_L^p}\right) \quad (1\text{-}4)$$

Rearranging (1-1) yields:

$$\frac{1+\cos\theta}{2} * \frac{\gamma_L}{\sqrt{\gamma_L^d}} = \sqrt{\gamma_S^p} * \sqrt{\frac{\gamma_L^p}{\gamma_L^d}} + \sqrt{\gamma_S^d} \quad (1\text{-}5)$$

where $\sqrt{\frac{\gamma_L^p}{\gamma_L^d}}$ is an independent variable and $\frac{1+\cos\theta}{2} * \frac{\gamma_L}{\sqrt{\gamma_L^d}}$ is the dependent variable in (1-5).

Table 1 shows the dispersion component of surface tension for pure liquids, which can be used for calculating the critical surface tension for fruit characterisation.

Table 1. Components of the surface tension of liquids used for characterising fruit surfaces

Compound	γ_L (mN/m)	γ_L^D (mN/m)	γ_L^P (mN/m)
Water [a]	72.10	19.90	52.20
Bromonaphthalene [a]	44.40	44.40	0.00
Formamide [a]	56.90	23.50	33.40
Toluene [b]	28.50	27.18	1.32
Ethylene glycol [c]	47.99	18.99	29.00
Glycerol [c]	63.40	30.00	33.40

a (Busscher et al., 1984)

b (Janczuk and Bialopiotrowicz, 1989)

c (Ramírez et al., 2012)

Critical surface tension (γ_c) is estimated by extrapolating a Zisman plot, obtained by plotting the cosine of the contact angle of pure liquids on a solid surface to be studied against the surface tension of the same series of liquid. The intercept of the curve at cos θ = 1 is known as critical surface tension. Critical surface tension is a fictitious value of γ_{SV} which is often used to describe surface wettability (Lima et al., 2010). Critical surface tension is thus defined as:

$$\gamma_C = \lim \gamma_{LV} \qquad \text{when} \qquad \theta \to 0 \qquad (1\text{-}6)$$

Choi et al., (2002) found that the surface tension of a chitosan solution decreased as the concentration of Tween 80 was increased. By contrast, interfacial tension () between the coating solution and an apple skin decreased from 21.1 to 3.7 dyne/cm by adding 1,000 ppm Tween 80; these value indicated that the apple skin was a low energy surface. Table 2 shows critical surface tension values for fruit coated with polymeric materials.

Table 2. Critical fruit skin surface tension

Fruit	Critical surface tension (mN / m)	T (°C)	Reference
Hog plum	9.39±0.07	21.3	(Cerqueira et al., 2009)
Cajá (miniature mango-like fruit)	23.92±0.10	21.3	(Cerqueira et al., 2009)
Mango	26.68±0.09	21.3	(Cerqueira et al., 2009)
Redcurrant	13.42±0.09	21.3	(Cerqueira et al., 2009)
Plum	19.62±0.09	21.3	(Cerqueira et al., 2009)
Mango	19.5	20	(Lima et al., 2010)
Apple	25.4 / 18.56 / 18.70 / 25.4	20 / 25 / - / 21.1	(Lima et al., 2010) (Ramírez et al., 2012) (Choi et al., 2002) (Carneiro-da-Cunha et al., 2009)
Tomato	17.4	20	(Casariego et al., 2008)
Quince	18.71	25	(Ramírez et al., 2012)
Strawberry	18.84	19	(Ribeiro et al., 2007)
	18.8	19	(Castro, 2005)
Waxed cranberry	26.8	21	(Skurtys et al., 2011)
Unwaxed cranberry	34.1	21	(Skurtys et al., 2011)
Waxed apple – Granny Smith	39.7	21	(Velásquez et al., 2011)

Edible solution wettability

Cerqueira et al., (2009) found that Ws values depended on the source and concentration of the galactomannans and fruit being evaluated. The following solutions were determined to be the best edible formulations for fruit: acerola-0.5% A. pavonina and 1.0% glycerol, caja-1.0% A. pavonina and 1.0% glycerol, mango and pitanga-1.5% A. pavonina and 1.0% glycerol and seriguela-0.5% C. pulcherrima and 1.5% glycerol. Lima et al., (2010) showed that the best Ws values for coating apples were obtained for coating solutions which did not contain glycerol. This result may have been due to the surface characteristics of apple, which has a high dispersive component having predominantly non-polar forces; although glycerol is polar, its presence can decrease the spreading of a solution. A formulation with 0.5% A. pavonina galactomannan, 1.5% collagen and 1.5% glycerol had the best spreading coefficient value, while an optimal solution of 0.5% C. pulcherrima and 1.5% collagen was suitable for apples. Ramírez et al., (2012) reported that apple skin had a better spreading coefficient than quince skin, most likely due to the wax which is naturally present in quince skin.

Carneiro-da-Cunha et al., (2009) discussed the effect of adding Tween 80 to a coating solution thereby lowering the surface tension of a liquid by reducing the cohesive forces and thus increasing Ws and improving compatibility between a solution and fruit surface. Tween 80 did not show any marked influence on Wa value.

Casariego et al., (2008) found that increasing chitosan and glycerol concentrations in coating solutions decreased the spreading coefficient and adhesion, while increasing the cohesion coeffi-

cient. An inverse relationship was observed when sorbitol was used as a plasticiser. Low energy surfaces mainly interact with liquids through dispersion forces, which may explain the low work of adhesion for tomato surfaces used to characterise chitosan solutions (polar liquids), when glycerol and sorbitol were used as plasticisers.

The formulations that produced suitable wettability for strawberries, as follows: 2% starch and 2% sorbitol, 0.3% carrageenan, 0.75% glycerol and 0.02% Tween 80 and 1% chitosan and 0.1% Tween 80 (Castro, 2005).

Skurtys et al., (2011) found that Wc decreased with increasing concentrations of Tween 20 or glycerol. The chitosan concentration affected cohesion in glycerol solutions but had a lesser effect on Tween 20 solutions. The authors found that adding glycerol to a chitosan solution decreased cranberry surface wettability by the solution. Removing wax from fruit epicarp was also found to improve wettability. Chitosan and Tween 20 solutions were found to wet the cranberry epicarp more (with or without wax) than glycerol-chitosan solutions.

Conclusions

Edible coatings can be applied to fruit to extend product shelf-life (Das et al., 2013), protect the fruit, decrease water loss, slow colour change, pH and titratable acidity during storage (Han et al., 2004) and modify the atmosphere (Park, 1999). Edible coatings are also biodegradable and help to protect the environment (Rhim et al., 2007).

Knowledge of surface properties is essential for understanding film adhesion and optimising performance characteristics, such as water permeability. Good surface wettability does not always correspond to good adhesion because wettability is necessary but not sufficient for proper adhesion (Piergiovanni and Limbo, 2010).

Different fruits' surface tension varies, depending on the particular fruit texture and skin composition, such as wax coating (Ramírez et al., 2012; Skurtys et al., 2011; Velásquez et al., 2011).

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