Arabinoxylan–Lipid-Based Edible Films and Coatings. 3. Influence of Drying Temperature on Film Structure and Functional Properties

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This work is a contribution to better knowledge of the influence of the structure of films obtained from emulsions based on arabinoxylans, hydrogenated palm kernel oil, and emulsifiers on their functional properties. The sucrose esters (emulsifiers) have a great effect on the stabilization of the emulsified film structure containing arabinoxylans and hydrogenated palm kernel oil. The structure and stability of the emulsion during drying strongly affect barrier and mechanical properties of films. The higher are creaming and coalescence phenomena in films, the lower is the water vapor permeability. Emulsion destabilization is favored by high drying temperature and tends to give films having a "bilayer-like" structure, which tends to improve the functional properties of arabinoxylans-based edible films.

KEYWORDS: Water vapor permeability; water absorption rate; surface hydrophobicity; barrier layer

INTRODUCTION

Arabinoxylans from maize bran are glycosidic polymer chains included in the hemicellulosic chemical group. These are nonsoluble fibers when in their native form and are characterized by a very high moisture retention capacity. However, after alkaline extraction, arabinoxylans become totally soluble in water and give high viscosity to aqueous solutions (*1, 2*). Arabinoxylans are used to form films because of their good solubility, their ability to form a continuous and cohesive matrix, and their neutral taste and odor (*3*). A preliminary study of their properties as edible films was done by Chanliaud (*3*), who showed that their mechanical and barrier properties are of the same order of magnitude as those of gluten films (*4*), whey protein isolate films (*5*), hydroxypropyl methylcellulose films (*6*), methylcellulose films (*7*), or starch films (*8*). Addition of lipids to protein-based or polysaccharide-based edible films often allows a significant reduction in their moisture sensitivity and permeability (*9*). Indeed, for arabinoxylan films, Péroval et al. (*10*) observed better barrier properties when some lipids such as fatty acids, triglycerides, or hydrogenated oils were added to films as emulsion or bilayer structures. The barrier performances of films containing hydrogenated palm kernel oil seem to depend strongly on the film structure. Indeed, bilayer films

exhibit much better moisture barrier properties than those obtained from emulsions (*4, 11–14*). However, bilayer films require at least three steps to manufacture instead of only one or two in the case of edible films prepared from emulsions. For this, the food industry has focused its research toward emulsion-based edible barriers.

The distribution of the lipid particles within an emulsion-based film affects the moisture permeability. Park et al. (*15*), Debeaufort and Voilley (*16*), and Perez-Gaco and Krochta (*17*) showed the lower the lipid globule size is, and the more homogeneous the distribution, the lower the water vapor permeability is. However, heat and solvent evaporation during the drying of the film-forming emulsion induces changes in the emulsion structure because of destabilization phenomena such as creaming, aggregation, and/or coalescence. Krochta (*18*) Martin-Polo et al. (*11*), Debeaufort and Voilley (*16*), Gontard et al. (*19*), and Phan The et al. (*20*) showed that the globule diameter and distribution in the film depend on the nature and concentration of the emulsifier but very little on its hydrophilic–lipophilic balance (HLB). Scant work has been done on the effect of drying conditions on the functional properties of edible films prepared from emulsions. Gontard et al. (*19*) compared the barrier performances of some edible films based on wheat gluten and various lipids. They discussed the observed differences in permeability as a function of the effect of the drying on the fat. They concluded that the higher the melting point of the lipid, the lower the water vapor permeability is and the poorer the mechanical properties are. However, they did not prepare films at different drying temperatures. On the contrary,

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Debeaufort and Voilley (*16*) showed that films prepared from emulsions composed of methylcellulose solution and paraffin wax are destabilized when drying air speed and temperature increase. They tend to correlate the change in emulsion structure of the dried films to their functional properties such as water vapor permeability. Perez-Gaco and Krochta (*17*) made edible films from emulsions composed of whey protein isolate, dried at temperatures ranging from room temperature to 80 °C. For both 20 and 60% beeswax contents, the water vapor permeability of these films decreased when the drying temperature increased, mainly because of emulsion structure changes. Phase separation was observed only for emulsions having the largest mean diameter of lipid particles (>1.5 μm).

The objective of this work was to better understand the influence of drying temperature on the structure and functional properties of arabinoxylan-based films.

MATERIALS AND METHODS

Materials. Arabinoxylans (AX, quality D) were freely provided and characterized by Ulice Co. They were used as the film-forming component of the hydrophilic continuous phase of emulsion-based edible films. Arabinoxylans constitute the soluble fraction of maize bran fibers and are used for their nutritional and water retention properties (*3*). Arabinoxylans are $\beta 1-4$ d-xylopyranosyl chains branched by L-arabinosyl residues with a molecular weight ranging from 100000 to 250000 Da. Anhydrous glycerol (98% purity, Fluka Chemical) was used as a plasticizer and was added to arabinoxylans to improve the mechanical properties of dried films. The hydrophobic dispersed phase was hydrogenated palm kernel oil (HPKO) (OK 35, Société Industrielle des Oléagineux), which has a melting point of $T_m = 34.5-36.5$ °C. To improve the stability of film-forming emulsions, two sucrose esters were tested: SP10 and SP 70 (Sisterna-Unipex), the physicochemical properties of which are follows: for SP10, HLB = 2, $T_m = 58$ °C, containing 10% monoesters and 90% di- and triesters composed of 70% stearate and 30% palmitate, soluble in oils and not soluble in water and glycerol; and for SP70, HLB = 15, $T_m = 50$ °C, 70% monoesters content (composed of 70% stearate and 30% palmitate), soluble in water, glycerol, and ethyl alcohol and not soluble in oils for SP70.

Methods. *Preparation of Homogeneous Edible Films.* An aqueous solution of 16% (w/v) of arabinoxylans was heated at 75 °C for 5 min with 600 rpm magnetic stirring prior to the addition of glycerol at a concentration of 15% on dry weight. The film-forming solution was held for 10 min at these same conditions and was then spread onto glass plates previously covered with a poly(vinyl chloride) adhesive sheet to prevent sticking of the dried films. A uniform thickness of 750 μm was obtained for the casting solution using a thin-layer chromatography spreader. All of the films were dried for 8 h in a ventilated incubator (KBF 240 binder, Odil) with temperatures fixed at 30 ± 1, 40 ± 1, or 80 ± 2 °C at 40 ± 2% relative humidity (RH). The temperatures were chosen to be lower, very close to, or higher than that of the melting point of the lipid used in the following emulsions.

After drying, the thickness of homogeneous edible films (arabinoxylan—glycerol) ranged between 65 and 80 μm.

The hydroxypropylmethylcellulose (HPMC) films, used as reference, were prepared with the same procedure except that the film-forming solution was composed of 9 g of HPMC dissolved in 100 mL of a water/ethyl alcohol mixture (75:25, w/w). The thickness of the spread solution was 1000 μm, and drying conditions were the same as for homogeneous AX films.

Preparation of Emulsion-Based Films. Three films were prepared from emulsions: one without emulsifier and two with different sucrose esters.

Emulsion-based films were made according to a process similar to that used for homogeneous films. The lipid phase represents 30% of the dry basis in either emulsions or dried films. The addition of the lipid (HPKO) and emulsifier (SP10 or SP70) was done differently according to their HLB. The emulsifier concentration was determined in a previous study (*20*) and was equal to 2.5% of the lipid phase. For

SP70 (hydrophilic), lipid and the sucrose ester were successively added to the arabinoxylan—glycerol solution. For SP10 (hydrophobic), the sucrose ester was first solubilized in the molten lipid prior to being added to the arabinoxylan—glycerol solution.

The mixture was then predispersed under magnetic stirring for 5 min at 600 rpm and 75 °C before homogenization using an Ultra-Turrax (T25-IKA, Labortechnik, Odil) for 2 min at 24000 rpm.

Film-forming emulsions were then spread at a 625 μm thickness prior to being dried using the same technique as that used for homogeneous arabinoxylan—glycerol films. After drying, film thickness ranged between 65 and 75 μm.

Characterization of Dried Films. The structure of emulsion-based films was characterized by laser light scattering granulometry. Globule size distribution profiles were determined using a Malvern Mastersizer Hydro 2000G (Malvern Instruments). The mean diameter of lipid globules and specific surface area were calculated from an optical model involving the absorbance of a 633 nm laser light by the lipid phase and the ratio of the refractive indices of the dispersed phase (lipid) and water (ratio = 1.11). Before structure analysis, 5 g of dried film was solubilized in 45 mL of deionized water at a temperature lower than the melting point of the lipid in an aim to prevent lipid melting or coalescence during sample preparation. Aggregation was displayed by adding 0.5% (w/v) of sodium dodecyl sulfate (SDS) prior to analysis.

Film microstructure was also observed by environmental scanning electron microscopy (ESEM, Phillips XL 30 ESEM). Using double-sided adhesive tape, a 5×10 mm^2 film sample was fixed on the support at a 90° angle to the surface, which allowed observation of the film cross section.

Mechanical properties of edible films (strength at breaking, *S*, and percentage of elongation, %*E*) were measured with a universal testing instrument (Instron UTTI 1122, Instron Ltd.). Samples were stored at 57% RH over a sodium bromide saturated solution (NaBr, Merck) for 10 days at 25 °C prior to analysis. Film samples were stretched at a constant rate of 100 mm·min^{-1} at the same temperature (25 °C) and RH (57%). The effective dimensions of the film before stretching were 20×60 mm^2.

Film thickness was measured with an electronic gauge (Multicheck FE, Sodexim) with precision ranging between 0.1 and 1% as a function of the thickness value (0—100 or 0—1000 μm). Twenty replicates were performed on each type of film.

Water vapor permeability (WVP) at a relative humidity differential of 84—22% was measured using a modified French standard method, AFNOR NF H00-030 (*21*), analogous to the ASTM Method E96-80 (*22*) and adapted to edible materials by Debeaufort et al. (*7*) and McHugh et al. (*23*). Films were fixed between two Teflon rings on the top of a glass cell containing a saturated salt solution of potassium chloride (KCl, Merck), which has a water activity of 0.84 at 25 °C. The surface exposed to air (outer) during drying was always exposed to the higher RH (84%) compartment of the permeation cell. Test cells were placed in a stirred air incubator (KBF 240, Binder, Odil) in which the temperature and relative humidity were fixed, respectively, at 25 ± 0.5 °C and 22 ± 1%. Test cells were periodically weighed until a constant weight variation rate was attained. Prior to measurement of the WVP, all films were stored at 25 °C in a desiccator over potassium acetate saturated solution (CH$_3$COOK, Merck) for 48 h, which fixes the RH at 22%. WVP (g·m^{-1}·s^{-1}·Pa^{-1}) was calculated using the equation

$$\text{WVP} = \frac{\Delta m x}{A \Delta t \Delta p}$$

where Δm is the weight loss by the permeation cell (g), x the film thickness (m), and A the exposed area (9.11×10^{-4} m^2) during the Δt duration (s) with a Δp partial water vapor pressure differential (Pa). The transfer rate, and thus the WVP, could be corrected by taking into account the diffusion of moisture in the air gap between the film and the saturated salt solution as suggested by Gennadios et al. (*24*). However, in our case, the air gap was always held constant to 1 cm, so all transfer measurements can be directly compared.

Surface hydrophobicity and wettability of films were estimated from the contact angle measurement of a 20 μL water droplet deposited on

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Table 1. Mean Diameter D[3,2] (Micrometers) and Phenomena Occurring during Drying of Film-Forming Emulsions at Three Temperatures[a]

emulsions: HPKO + AX + Gly	before drying		drying at 30 °C		drying at 40 °C		drying at 80 °C	
	water	SDS	water	SDS	water	SDS	water	SDS
without sucrose ester	4.4	2.1	8.7	10.0	nd	nd	27.1	18.6
	aggregation		aggregation + coalescence		aggregation + coalescence		coalescence	
with 2.5% SP70	1.3	1.3	9.4	1.0	23.4	22.2	23.9	22.0
	stable		aggregation		coalescence		coalescence	
with 2.5% SP10	2.8	2.5	0.7	0.6	11.4	0.7	15.7	18.2
	stable		stable		aggregation		coalescence	

[a] AX, arabinoxylans; Gly, glycerol; HPKO, hydrogenated palm kernel oil; nd, not determined.

the film surface using a G1 Kruss goniometer (Kruss GmbH) equipped with image analysis software (Drop Shape Analysis, Kruss GmbH). Kinetics of contact angle and liquid moisture absorption rate were determined at 25 °C.

Significance. Each film attribute was measured at least in triplicate, and differences between means were tested at the $p = 0.05$ level using the Student−Newmans−Keuls t test of SAS-ANOVA (Statistical Analysis System, version 6.02).

RESULTS AND DISCUSSION

Influence of Drying Temperature on Film Structure. Drying temperatures were chosen as a function of the melting point of HPKO. Temperatures were 30 °C, that is, lower than the melting point, 40 °C, slightly higher than the melting point, or 80 °C, which is much higher than the melting point of HPKO. The mean diameters D(3,2) of HPKO globules in dried films after dispersion in cold water or cold SDS solution (dissociating medium) are given in **Table 1**. The aveage standard deviation on the D(3,2) is lower than 4.5%. All measurements were at least duplicated. These results show that at temperatures above the HPKO melting point, the formation of large particles is favored. Moreover, globule sizes in water or SDS solution are not significantly different at the $p = 0.05$ level, which means that HPKO globules merge because coalescence is easier with molten lipids. For the films dried at 30 °C, some coalescence occurred when no emulsifier was used, whereas only aggregation is involved when SP70 is added to the emulsion. Aggregation is evident when the mean particle diameter is smaller when granulometry analysis is done in the dissociating medium (0.1% SDS solution). At 40 °C, coalescence occurs for the 2.5% SP7o, whereas only aggregation occurs for the 2.5% SP10. This can be explained by the higher melting point of SP10 (58 °C), which increases the melting point of the HPKO + SP10 mixture to 38.7 °C, very close to the drying temperature (40 °C).

From **Table 1**, as drying temperature increased, emulsion stability decreased. Dickinson and Stainsby (*25*) showed that high temperature induces a decrease of the viscosity of the continuous phase, which favors creaming and coalescence of oil-in-water emulsions. In this case, migration of lipid globules toward the evaporation surface (creaming) during drying results in aggregation of HPKO globules when the temperature is lower than 40 °C. At 40 and 80 °C, HPKO melts and allows the coalescence phenomenon to occur. Perez-Gaco and Krochta (*17*), however, did not see a change of the mean diameter of beeswax droplets in whey protein isolate (WPI)-based films when the drying temperature was increased. They explained the greater stability of these film-forming emulsions by both the higher viscosity of the WPI solution and the emulsifying character of the WPI.

These destabilization processes affect the HPKO distribution within the cross section of the film as shown in **Figure 1**. As observed by Phan The et al. (*20*) in previous work, film structure

depends on the nature and concentration of the sucrose esters used as emulsifier, and also on the drying conditions, particularly on the temperature, which can lead to an apparent bilayer structure from a dried emulsion. Indeed, panels c and f of **Figure 1** show that most of the HPKO has migrated toward the evaporation surface. Increasing temperature provokes the formation of nonspherical globules instead of those obtained when the emulsion is dried at 30 °C. Therefore, high drying temperature favors creaming and thus aggregation and coalescence as observed by Debeaufort and Voilley (*16*) for paraffin wax globules dispersed in a methylcellulose network. These authors also showed that air speed contributes to the destabilization of film-forming emulsion.

Influence of Drying Temperature on the Water Vapor Permeability and Moisture Sensitivity of Films. The WVP was determined at 25 °C on dried films exposed to a 22−84% RH differential. WVP values are given in **Table 2**. When films are dried at 30 or 40 °C, permeabilities are not significantly different at the $p = 0.05$ level, but when the temperature increases to 80 °C, moisture transfers through emulsion-based films containing SP70 or SP10 decrease from 8 to 23% respectively. This could be due to the apparent bilayer structure as observed in **Figure 1**. In the case of the emulsion-based film that does not contain emulsifier, WVP was not determined when these films were dried at 80 °C because they could not be removed from the support without breaking.

Water vapor transfer through arabinoxylan-based films depends on the nature of both the lipid and the continuous matrix as observed by Koelsch and Labuza (*26*), Gontard et al. (*19*), Péroval et al. (*10*), and Perez-Gaco and Krochta (*17*), but it also depends on the structure of the dried emulsion, that is, the globule distribution or their creaming and coalescence as shown by Debeaufort and Voilley (*16*). Indeed, Kamper and Fennema (*27*) observed that emulsion-based films composed of HPMC and a mixture of stearic and palmitic acids, dried at 90 °C for 15 min, exhibit 40 times lower permeability than that of bilayer films of the same composition. This is due to a change of the emulsion structure toward a bilayer one, but the lipid phase remains entrapped in the matrix which protects the outer layer enriched in lipid against cracking, which often occurs in bilayer films. It seems that drying at high temperature leads to destabilization of the emulsion, which increases the resistance to moisture migration. In contrast, for arabinoxylan or HPMC films without lipids, the drying temperature does not affect significantly the WVP. This confirms that the main effect of the drying temperature concerns the emulsion structure, that is, the lipid particle distribution. Indeed, the continuous hydrocolloid-based matrix seems not to be affected by the temperature in the range studied.

Because of the destabilization of the emulsion, an irregular and nonsmooth surface is observed on the micrographs (**Figure**
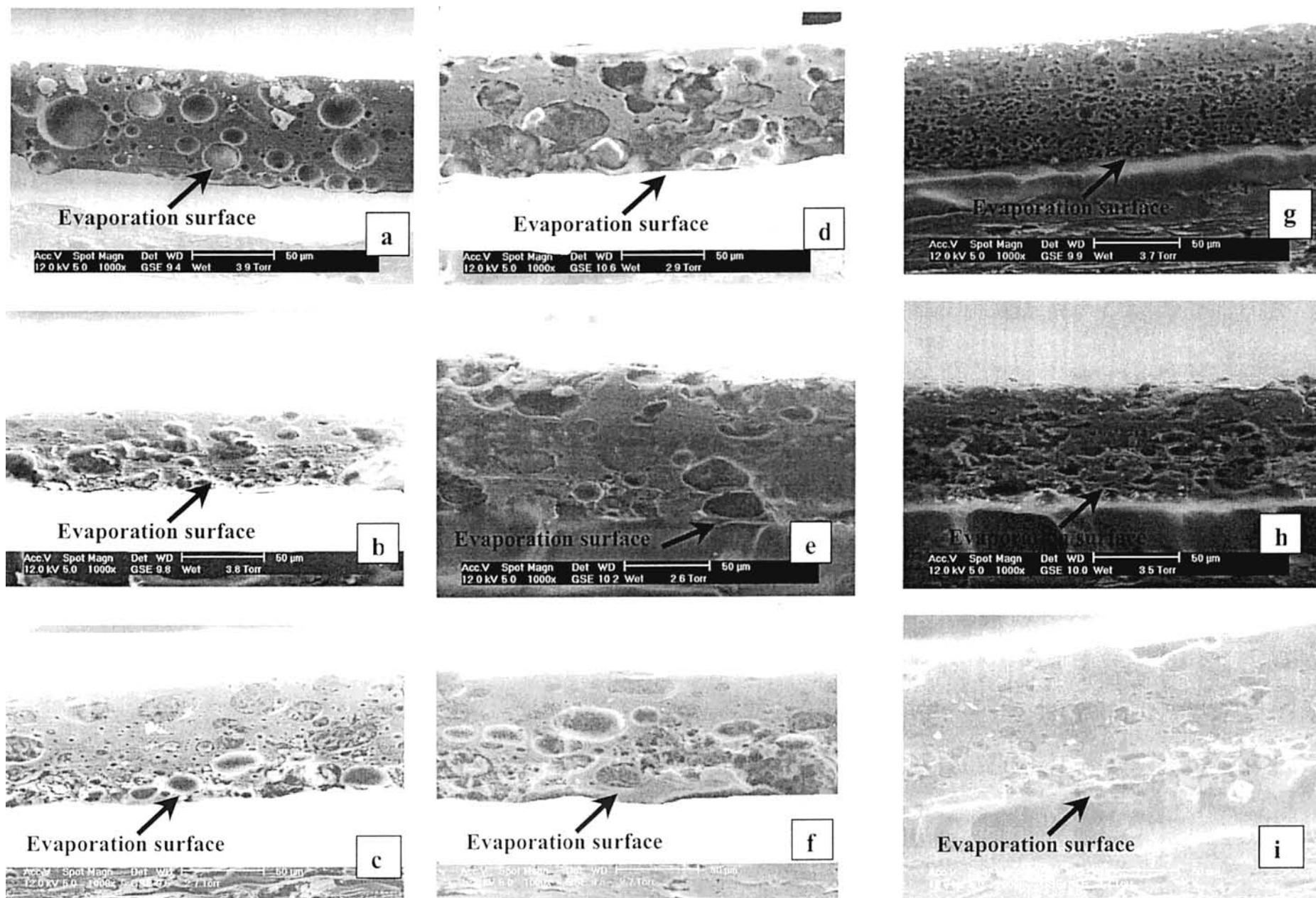
Figure 1. ESEM cross-section micrographs of arabinoxylan–HPKO–glycerol films without emulsifier dried at (a) 30, (b) 40, or (c) 80 °C or containing 2.5% SP10 dried at (d) 30, (e) 40, or (f) 80 °C or containing 2.5% SP70 dried at (g) 30, (h) 40, or (i) 80 °C.

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Table 2. Water Vapor Permeability, Contact Angle, and Water Absorption Rate on Films as a Function of the Drying Temperature[a]

drying temp:	WVP (10^{-11} g·m^{-1}·s^{-1}·Pa^{-1})			contact angle (deg)			water absorption rate (μL·s^{-1})		
	30 °C	40 °C	80 °C	30 °C	40 °C	80 °C	30 °C	40 °C	80 °C
HPMC + 15% Gly	19.73 ± 0.56 a	15.54 ± 0.91 a,b	19.68 ± 0.39 a	65.4 ± 1.6 f	60.6 ± 1.8 f	69.6 ± 1.1 f	0.44 ± 0.03 k	0.37 ± 0.08 k	0.25 ± 0.04 m
AX + 15% Gly	13.70 ± 0.72 b	11.70 ± 0.44 b,c	12.70 ± 0.39 b	65.5 ± 3.8 f	62.1 ± 18.0 f	53.5 ± 0.7 e	0.19 ± 0.10 p	0.39 ± 0.13 k	0.30 ± 0.10 l
AX + 15% Gly + HPKO	9.25 ± 0.45 d	10.45 ± 0.28 c,d	nd	79.5 ± 0.6 h	108.1 ± 4.5 j	nd	0.17 ± 0.06 p	0.08 ± 0.01 a	nd
AX + 15% Gly + HPKO + 2.5% SP10	9.51 ± 0.79 c,d	9.63 ± 0.40 c,d	7.72 ± 0.66 e	61.3 ± 4.9 f	81.4 ± 5.6 i	78.8 ± 7.8 h	0.19 ± 0.04 o	0.30 ± 0.06 l	0.22 ± 0.03 m,n
AX + 15% Gly + HPKO + 2.5% SP70	11.13 ± 0.55 c	11.33 ± 0.28 c	10.70 ± 0.11 c,d	57.6 ± 2.2 e	71.7 ± 4.7 g	77.0 ± 4.4 h	0.24 ± 0.02 m	0.26 ± 0.08 l,m	0.17 ± 0.01 o

[a] AX, arabinoxylans; Gly, glycerol; HPKO, hydrogenated palm kernel oil; nd, not determined. Values having the same letter are not significantly different at the $p < 0.05$ level.
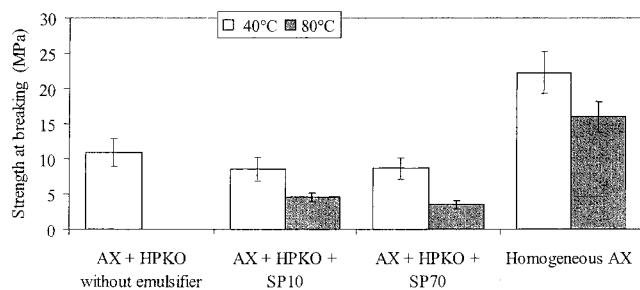
Figure 2. Strength at breaking of arabinoxylan-based edible films as a function of the drying temperature.

Figure 3. Elongation at breaking of arabinoxylan-based edible films as a function of the drying temperature.

1). Therefore, the contact angles measured on films dried at 30 and 80 °C tend to increase with the drying temperature. Indeed, the contact angle is at least 25% greater for films dried at 40 and 80 °C than for films dried at 30 °C. The destabilization of the emulsion results in creaming during drying and tends to increase the surface hydrophobicity of films. This explains higher contact angles because of lipid enrichment at the outer surface. However, from contact angle kinetics, the liquid water absorption rates in the films were determined and are given in **Table 2**. In the absence of sucrose ester, the water absorption rate tends to decrease when the drying temperature increases. This is also correlated to the structure. Indeed, the increasing lipid concentration at the evaporation surface of the film tends to slow water penetration. Nevertheless, the addition of sucrose esters significantly increases the water absorption rate, particularly for hydrophilic sucrose esters such as SP70. This is due to the polar part of the sucrose ester, which decreases the surface tension and favors surface wetting and penetration of the water droplet in the film. However, the water absorption rate of emulsion-based film is always lower than that of arabinoxylans + glycerol or HPMC + glycerol films. Therefore, from **Table 2**, emulsion-based films show reduced liquid and vapor transfers as drying temperature increased. However, several authors showed that when the hydrocolloid continuous matrix is rapidly dried, interactions between biopolymer chains are less numerous, which induces a loss of the mechanical resistance and deformation of the film (*19, 28−30*).

Mechanical Properties as a Function of the Drying Temperature of Films. Strength at breaking (*S*) and percentage of elongation (%*E*) of stretched samples were determined on films dried at 40 and 80 °C (**Figures 2** and **3**). Whatever the drying temperature, the mechanical properties of films containing lipids are from 0.8 to 3 times lower than those of homogeneous arabinoxylan films. Gontard et al. (*19*) showed that the addition of lipids to protein-based films decreases significantly both strength and elongation. The higher the solid fat content is, the poorer the mechanical properties of films obtained from emulsions are (*5, 19*). Moreover, Krochta (*18*)
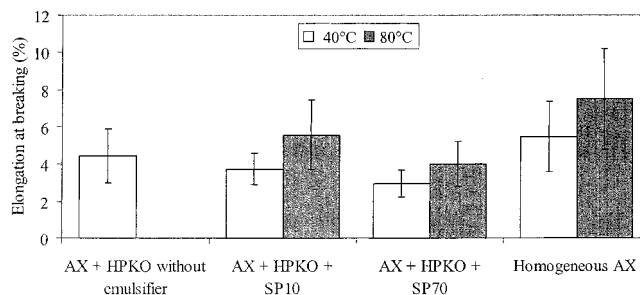
explained that liquid fats could act as a plasticizer or as a lubricant in hydrocolloid networks, which could improve their mechanical properties, although solid fat inclusions in polysaccharidic or protein matrices induce disruptions and preferential breaking zones. This is confirmed by Perez-Gaco and Krochta (*17*), who showed that increasing the amount of solid beeswax particles in WPI films strongly reduces tensile strength and elongation. Then solid lipids tend to increase the weakness of edible films prepared from emulsions. In our case, the presence of HPKO globules decreases significantly the mechanical performances of arabinoxylan-based films.

The nature of the emulsifier (SP10 or SP70) does not affect significantly the mechanical properties of films, but temperature greatly modifies both strength and elongation. Indeed, elongation increases, whereas strength decreases, when temperature increases from 40 to 80 °C (**Figures 2** and **3**). At higher temperature, solvent (water) evaporates more rapidly and films are dried more quickly. Therefore, arabinoxylan chains have less mobility (because of rapid concentration process) and less time to settle bonds between chains. Consequently, the number of interactions between arabinoxylan chains is reduced, which decreases the strength and increases elongation because the network is less organized and more stretchable (*31, 32*). Moreover, the increase of the drying temperature provokes a bilayer-like structure of films as observed on micrographs (**Figure 1**). This particular structure tends to form a continuous layer of homogeneous arabinoxylan, which improves the mechanical properties of films dried at 80 °C. Indeed, Debeaufort et al. (*13*) and Quezada-Gallo et al. (*15*) showed that mechanical properties of methylcellulose−HPKO or wheat gluten−HPKO bilayer films depend only on the mechanical properties of the hydrocolloid layer, that is, methylcellulose or wheat gluten, so it seems that the destabilization of the emulsion during drying at high temperature tends to favor both elongation and barrier properties of films prepared from arabinoxylan−HPKO emulsions.

Conclusion. Under moderated drying conditions lipophilic sucrose ester SP10 stabilizes the film-forming emulsion during

drying, which gives films having a lower WVP. Hydrophilic SP70 confers to films structure in which very small globules of HPKO are observed and homogeneously distributed. However, the films permeability is slightly greater than that obtained with SP10. At high drying temperature, the structure of the emulsion is greatly modified; creaming, aggregation, and coalescence are displayed, resulting in an apparent "bilayer-like" structure with the lipid concentrated at the surface, which results in reduction of moisture transfer. Moreover, increasing drying temperature tends to induce a gain of mechanical performances (elongation) of films containing lipids.

For industrial applications, high temperatures are recommended to decrease process time, so optimization of drying conditions has to be improved for upgrading barrier and mechanical performances of films prepared from emulsions.

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LITERATURE CITED

(1) Watson, S. A. Corn Hull Gum. In *Industrial Gums, Polysaccharides And Their Derivatives*; Whistler, R. L., BeMiller, J. N., Eds.; Academic Press: New York, 1959; pp 299−306.

(2) Sugawara, M.; Benno, Y.; Koyasu, E.; Takeuchi, M.; Mitsuoka, T. Digestion and Fermentation by Human Intestinal Bacteria of Corn Fiber and its Hemicellulose *in vitro. Agric. Biol. Chem.* **1991**, *55*, 565−567.

(3) Chanliaud, E. Extraction, Caractérisation et Propriétés Fonctionnelles des Hétéroxylanes de Son de Maïs. Ph.D, University of Paris VII and ENSIA, 1995; pp 1−198.

(4) Gennadios, A.; Weller, C. L.; Testin, R. F. Modification of Physical and Barrier Properties of Edible Wheat Gluten-based Films. *Cereal Chem.* **1993**, *70*, 426−429.

(5) McHugh, T. H.; Krochta, J. M. Water vapor permeability properties of edible whey protein−lipid emulsion films. *J. Am. Oil Chem. Soc.* **1994**, *71*, 307−311.

(6) Kester, J. J.; Fennema, O. Edible Films and Coatings: A Review. *J. Food Technol.* **1986**, *12*, 47−59.

(7) Debeaufort, F.; Martin-Polo, M.; Voilley, A. Polarity, Homogeneity and Structure affect Water Vapor Permeability of Model Edible Films. *J. Food Sci.* **1993**, *58*, 426−434.

(8) Arvanitoyannis, I.; Psomiadou, E.; Nakayama, A.; Aiba, S.; Yamamoto, N. Edible Films Made from Gelatin, Soluble Starch and Polyols, Part 3. *Food Chem.* **1997**, *60*, 593−604.

(9) Morillon, V.; Debeaufort, F.; Blond, G.; Capelle, M.; Voilley; A. Factors affecting the moisture permeability of lipid-based edible films: a review. *CRC Nutr. Food Sci.* **2002**, *42* (1), 67−89.

(10) Peroval, C.; Debeaufort, F.; Despre, D.; Voilley, A. Arabinoxylan-lipid-based edible films. 1. Water vapor permeance and other physical properties. *J. Agric. Food Chem.* **2002**, submitted for publication.

(11) Martin-Polo, M.; Mauguin, C.; Voilley, A. Hydrophobic Films and Their Efficiency against Moisture Transfer. 1. Influence of the Film Preparation Technique. *J. Agric. Food Chem.* **1992**, *40*, 407−412.

(12) McHugh, T. H.; Krochta, J. M. Dispersed Phase Particle Size Effects on Water Vapour Permeability of Whey Protein-Beeswax Edible Emulsion Films. *J. Food Process. Preserv.* **1994**, *18*, 173−188.

(13) Debeaufort, F.; Quezada-Gallo, J. A.; Delporte, B.; Voilley, A. Lipid hydrophobicity and physical state effects on the properties of bilayer edible films. *J. Membr. Sci.* **2000**, *4679*, 1−8.

(14) Quezada-Gallo, J. A.; Debeaufort, F.; Callegarin, F.; Voilley, A. Lipid hydrophobicity, physical state and distribution effects on the properties of emulsion-based edible films. *J. Membr. Sci.* **2000**, *4678*, 1−10.

(15) Park, H. J.; Bunn, J. M.; Vergano, P. J.; Testin, R. F. Gas permeation and thickness of the sucrose polyesters, Semperfresh tm coatings on apples. *J. Food Process. Preserv.* **1994**, *18*, 349−358.

(16) Debeaufort, F.; Voilley A. Effect of Surfactant and Drying Rate on Barrier Properties of Emulsified Edible Films. *Int. J. Food Sci.* **1995**, *30*, 183−190.

(17) Perez-Gaco, M. B.; Krochta, J. M. Lipid particle size effect on the water vapor permeability and mechanical properties of whey protein/beeswax emulsion films. *J. Agric. Food Chem.* **2001**, *49*, 996−1002.

(18) Krochta, J. M. Emulsion Films on Food Products to Control Mass Transfer. *J. A.I.Ch.E.* **1990**, *86*, 57−61.

(19) Gontard, N.; Duchez, C.; Cuq, J. L.; Guilbert, S. Edible Composite Films of Gluten and Lipids: Water Vapour Permeability and other Physical Properties. *Int. J. Food Sci. Technol.* **1994**, *29*, 39−50.

(20) Phan The, D.; Debeaufort, F.; Peroval, C.; Despre, D.; Courthaudon, J. L.; Voilley, A. Arabinoxylan-lipid-based edible films. 2. Influence of the sucrose ester nature on the emulsion structure and film properties. *J. Agric. Food Chem.* **2002**, *50*, 266−272.

(21) AFNOR H-00-030 Matières en Feuille. Détermination du Coefficient de Transmission de la Vapeur d'Eau. In *Recueil des Normes Françaises*; Association Française de Normalisation: Paris, France, 1974.

(22) ASTM E96-80. Standard test method for water vapor transmission of materials. In *ASTM Book of Standards*; American Society for Testing and Materials: Philadelphia, PA, 1980.

(23) McHugh, T. H.; Avena-Bustillos, R.; Krochta, J. M. Hydrophilic Edible Films: Modified Procedure for Water Vapor Permeability and Explanation of Thickness Effects. *J. Food Sci.* **1993**, *58*, 889−903.

(24) Gennadios, A.; Weller, C. L.; Gooding, C. H. Measurement errors in water vapor permeability of highly permeable, hydrophilic edible films. *J. Food Eng.* **1994**, *21*, 395−409.

(25) Dickinson, E.; Stainsby, G. *Advances in Food Emulsions and Foams*; Elsevier Applied Science: London, U.K., 1988; pp 1−228.

(26) Koelsch, C. M.; Labuza, T. P. Functional, Physical and Morphological Properties of Methyl Cellulose and Fatty Acid-Based Edible Barriers. *J. Food Sci. Technol.* **1992**, *25*, 404−411.

(27) Kamper, S. L.; Fennema, O. Water Vapour Permeability of an Edible, Fatty Acid, Bilayer Film. *J. Food Sci.* **1984**, *49*, 1482−1485.

(28) Greener-Donhowe, I.; Fennema, O. The effects of solution composition and drying temperature on crystallinity, permeability and mechanical properties of methylcellulose films. *J. Food Process. Preserv.* **1993**, *17*, 231−246.

(29) Greener-Donhowe, I.; Fennema, O. The effects of plasticizers on crystallinity, permeability, and mechanical properties of methylcellulose films. *J. Food Process. Preserv.* **1993**, *17*, 247−257.

(30) Shastry, A. V.; Hartel, R. W. Crystallization and drying in thin sucrose films during panning. *J. Food Sci.* **1996**, *61*, 978−981.

(31) Sears, J. K.; Darby, J. R. *The Technology of Plasticizers*; Wiley Interscience Publications: New York, 1982.

(32) Ashby, M. F.; Gibson, L. J. *Cellular Solids: Structure and Properties*; Pergamon Press: Oxford, U.K., 1988.