

COLOUR, MECHANICAL PROPERTIES AND WATER VAPOUR
PERMEABILITY OF PECTIN FILMS

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Abstract. The aim of this work was to analyse the effect of pectin and glycerol concentration on colour, mechanical properties and water vapour permeability of pectin edible films. Pectin films were obtained from apple pectin film-forming solutions at concentrations of 1.5, 2.5 and 3.5%. Glycerol was added as a plasticizer at 30, 50 and 70% (w/w of pectin). Colour was measured in the $L^*a^*b^*$ system and total colour difference was calculated. Water vapour permeability at relative humidity differentials of 30-100% was measured at 25°C. Mechanical properties were presented as tensile force and elongation at break. Based on the results obtained, increasing content of pectin caused an increase in total colour difference, water vapour permeability and tensile force of analysed films. Glycerol caused increasing elongation at break and water vapour permeability.

Key words: edible films, pectin, colour, water vapour permeability mechanical properties

INTRODUCTION

An edible coating is a thin layer of material formed as a coating on a food product, while an edible film is a preformed thin layer made of edible material, which once formed can be placed on or between food components (McHugh, 2000, Falguera *et al.* 2011). Edible films are classified into three categories taking into account the nature of their components: hydrocolloids (proteins, polysaccharides or alginates), lipids (fatty acids, acylglycerols or waxes) and composites (made by combining substances from the two categories) (Mariniello *et al.* 2003).

The use of edible coatings or films in food applications, and especially highly perishable products such as horticultural ones, is conditioned by the achievement of diverse characteristics such as cost, availability, functional attributes, mechanical properties (flexibility, tension), optical properties (brightness and opacity), the

barrier effect against flow of gases, structural resistance to water and microorganisms, and sensory acceptability. These characteristics are influenced by parameters such as the kind of material implemented as structural matrix (composition, molecular weight distribution), the conditions under which films are preformed (type of solvent, pH, concentration of components and temperature) and the type and concentration of additives (plasticizers, cross-linking agents, antimicrobials, antioxidants or emulsifiers) (Rojas-Grau *et al.* 2009).

Pectin is a complex anionic polysaccharide composed of β -1,4-linked d-galacturonic acid residues, wherein carboxyl groups of uronic acid are either fully (HMP, high methoxyl pectin, DE > 50%) or partially (LMP, low methoxyl pectin, DE < 50%) methyl esterified. Like alginates, low methoxyl pectins form gel with divalent cations (Marudova *et al.* 2004). The most important environmental factors are pH, Ca^{2+} and soluble solids. They have different and partly opposite effects on the gelling process of different pectin types (Kastner *et al.* 2012). Apple pectin is able to form easily detachable and transparent edible films with promising use in food applications.

The choice of plasticizer to be added to the formulation depends on the compatibility with the polymer and solvent used, i.e., it must be miscible in the polymer and in the solvent in order to avoid premature separation during the drying process. The concentration of plasticizers used in film preparations generally ranges from 10 to 60 g·100 g⁻¹ of dry polymer, depending on the desired flexibility (Guilbert *et al.* 1996). The most effective plasticizers generally have a molecular structure close to the polymer which should be plasticized.

The literature records the usage of various types of plasticizers in the preparation of edible films. Most applicants are polyols, especially glycerol (Flores *et al.* 2007) and sorbitol (Martelli *et al.* 2006) or mixtures of glycerol, sorbitol and xylitol (Mali *et al.* 2005). Various polyethylene glycols (PEG from 200 to 3400), diethanolamine and triethanolamine, and many other compounds, were evaluated by Irissin-Mangata *et al.* (2001). Therefore, all these plasticizers have hydrophilic characters, which can contribute to the increase in water permeability and matrix susceptibility to humidity.

Glycerol is one of the most popular plasticizers used in the preparation of edible films and coatings due to its stability and compatibility with the hydrophilic nature of the biopolymer chains (Chillo *et al.* 2008). Numerous research studies indicate that glycerol has better activity than other substances used (Sothornvit and Krochta 2001, Vanin *et al.* 2005). The main advantages of this plasticizer are a high boiling point, lack of odour, water solubility and miscibility with those components (Jangchud and Chinnan 1999).

The aim of this work was to analyse the effect of pectin and glycerol concentrations on colour, water vapour permeability and mechanical properties of pectin edible films.

MATERIALS AND METHODS

Apple pectin (type NEJ-A2) was obtained from PEKTOWIN (Jaslo, Poland). Anhydrous glycerol and calcium chloride were purchased from POCH S.A. (Gliwice, Poland).

Film-forming solutions were prepared by slowly dissolving pectin in distilled water (1.5, 2.5 and 3.5%). Glycerol at 30, 50, and 70%, and calcium chloride at 1% (w/w of pectin) were then added. The solutions were heated to 70°C and poured onto a series of Petri dishes (diameter 15 cm). To control film thickness, the quantity of each film-forming solution poured onto a plate was always 15 mL. The solutions were dried for 3 days at 25±1°C and 40±2% relative humidity in a ventilated climate chamber. After this time the dried film solutions were easy to cast out. The dried films were peeled off and conditioned at 53±1% RH and 25±1°C for 7 days prior to testing. Composition and symbols of the films are given in Table 1.

Table 1. Composition of pectin (P) film-forming solutions with various level of glycerol (G)

Film	Pectin (%)	Glycerol (%)	Glycerol (g)	CaCl ₂ (g)	Water (g)
1.5P_50G	1.5	50	0.75	0.015	97.735
2.5P_50G	2.5	50	1.25	0.025	96.225
3.5P_50G	3.5	50	1.75	0.035	94.715
2.5P_30G	2.5	30	0.75	0.025	96.725
2.5P_50G	2.5	50	1.25	0.025	96.225
2.5P_70G	2.5	70	1.75	0.025	95.725

Film thickness was measured with an electronic gauge (METRISON, Poland). Precision of the measurement was 1 µm. Prior to film thickness measurements the electronic gauge was calibrated at 74 and 139 µm using standards. Five thickness measurements were taken of each film, one in the centre and four around the perimeter, and a mean value was used in the calculations.

Colour of films on air surface was determined with a colorimeter (Minolta, Model CR-300, Japan) using the CIE colour parameters: L^* , from black (0) to white (100); a^* , from green (-) to red (+); and b^* , from blue (-) to yellow (+)

(Gennadios *et al.* 1996). Colour of the films was expressed as the total colour difference (ΔE) according to the following equation (Sobral *et al.* 2005):

$$\Delta E = \sqrt{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2} \quad (1)$$

where: ΔL^* , Δa^* and Δb^* are the differentials between a sample colour parameter and the colour parameter of a standard used as the film background ($L^* = 94.6$, $a^* = 0.80$, $b^* = -2.16$).

Tensile force and elongation at break of the films (25 mm x 100 mm), as the average of 10 determinations, were determined using a Texture Analyser TA-XT2i (Stable Microsystems, U.K.). Self-tightening roller grips were used as the probe to perform tensile assays. The initial distance of separation and velocity were adjusted to 50 mm and 1 mm s⁻¹, respectively, considering conditioned samples with the cell load of 5 kg.

Water vapour permeability of films was measured gravimetrically according to Debeaufort *et al.* (1993). Film samples were placed between two rubber-based rings on the top of a glass cell containing distilled water, which allowed fixing of the internal RH of permeation cells at 100%. Those permeation cells were introduced into a ventilated chamber maintained at 30% RH and 25°C, ensuring a relative humidity gradient between two sides of the film at 30-100%. Water vapour permeability was calculated using the following equation:

$$\text{WVP} = \frac{\Delta m \cdot e}{A \cdot \Delta t \cdot \Delta p} \quad (2)$$

where: $\Delta m/\Delta t$ – weight of moisture loss per unit of time (g s⁻¹), A – film area exposed to moisture transfer ($8.04 \cdot 10^{-4}$ m²), e – film thickness (m), Δp – water vapour pressure difference between the two sides of the film (Pa). Measurements were performed in at least three replicates.

Statistical analysis was performed with Statgraphics Plus 5.0 version program (Manugistics Corp., Rockville, MD, U.S.A.). The analysis of variance (ANOVA) and Tukey's multiple comparisons were performed to detect significant differences in film properties. The significance level was used 0.001.

RESULTS AND DISCUSSION

All pectin films were transparent, easily detachable, without pores or cracks. Films containing 30% glycerol were more brittle, and thus with 70% glycerol more flexible due to the plasticizing effect in a film network. Previous works

showed that film formed without plasticizer was very brittle and broke when peeled off. Generally, if films are formed from purely polymeric ingredients they tend to be brittle (Coupland *et al.* 2000). Banker (1966) reported that the plasticizer can retard or enhance moisture transmission depending on its concentration. Plasticizer can reduce the internal hydrogen bonding and increase the intermolecular spacing.

Edible films or coatings should be as close to colourless as possible to stimulate the appearance of common polymeric films (Rhim *et al.* 2002). The colour of films is relevant since colour has a direct impact on the appearance of the packaged product (Galus *et al.* 2012). All colour values for analysed pectin films are shown in Table 2. The lightness (L^*) decreased with higher content of pectin and increased with higher content of glycerol. All values ranged from 88.7 to 90.2. However, a^* and b^* parameters significantly increased with higher content of pectin. Films containing 70% of glycerol showed the highest values of a^* and b^* parameters. The increase in total colour difference from 2.9 to 5.9 was correlated with increasing content of pectin in analysed films. Higher content of glycerol caused a decrease in this parameter. These values are lower than values obtained for other biopolymer films based on soybean protein (Kunte *et al.* 1997) or modified starch and carboxymethyl cellulose (Ghanbarzadeh *et al.* 2010). Generally, colour experiments showed that pectin films will not affect appearance of food products when in use.

Table 2. L^* , a^* , b^* colour parameters and total colour difference (ΔE) of pectin films. Values followed by the same letter are not significantly different at the $p < 0.001$

Film	L^*	a^*	b^*	ΔE
1.5P_50G	90.2 ± 0.1^c	0.68 ± 0.02^a	4.1 ± 0.4^a	2.9 ± 0.3^a
2.5P_50G	89.1 ± 0.2^{ab}	0.69 ± 0.04^a	5.8 ± 0.2^b	4.9 ± 0.2^b
3.5P_50G	88.9 ± 0.4^{ab}	0.74 ± 0.02^{ab}	6.7 ± 0.6^{bc}	5.9 ± 0.6^{bc}
2.5P_30G	88.7 ± 0.4^a	0.79 ± 0.02^b	7.2 ± 0.3^c	6.4 ± 0.4^c
2.5P_50G	89.1 ± 0.2^{ab}	0.69 ± 0.04^a	5.8 ± 0.2^b	4.9 ± 0.2^b
2.5P_70G	89.5 ± 0.6^b	0.79 ± 0.07^b	6.0 ± 0.9^b	5.0 ± 0.9^b

Mechanical properties of edible films are a very important parameters in their use and are often the crucial factor for food applications. Table 3 shows tensile force and elongation at break of pectin films. Values of tensile force ranged significantly ($p < 0.001$) from 8.9 to 21.6 N when content of pectin increased from 1.5 to 3.5%. It can be explained by the more compact structure due to the higher content of film-forming component. Addition of 50 or 70% glycerol caused a decrease in

tensile force from 24.3 to 14.8-15.1 N. The values of elongation at break were similar for films with different content of pectin (5.9-6.2%) and an increase in these values was observed when glycerol content increased. These results are similar to those presented in the literature for polymeric films. Altenhofen da Silva *et al.* (2009) reported a decrease in tensile strength and increase in elongation at break of pectin films crosslinked by Ca^{2+} with increasing glycerol content from 5 to 10%. Zsivanovits *et al.* (2004) showed the mechanical properties as a function of counterion (K^+ , Ca^{2+} , Mg^{2+}), concentration and extent of hydration. Guerrero *et al.* (2010) analysed soy films with various concentrations of glycerol in the range of 30-50% and found a significant increase in elongation at break. The effect of glycerol on the elongation analysis has been widely described in the literature. Some good examples are the work on peanut proteins (Jangchud and Chinnan 1999), pea proteins (Zhang and Han 2008) and oxidised potato starch (Hu *et al.* 2009).

Table 3. Tensile force and elongation at break of pectin films. Values followed by the same letter are not significantly different at $p < 0.001$

Film	Tensile force (N)	Elongation at break (%)
1.5P_50G	8.9 ± 1.3^a	6.2 ± 0.9^a
2.5P_50G	15.1 ± 1.6^b	5.9 ± 1.2^a
3.5P_50G	21.6 ± 2.9^c	6.0 ± 1.6^a
2.5P_30G	24.3 ± 3.2^c	4.9 ± 1.2^a
2.5P_50G	15.1 ± 1.6^b	5.9 ± 1.2^a
2.5P_70G	14.8 ± 1.9^b	12.4 ± 2.0^b

The films with higher content of plasticizers are more flexible and the force needed for them to break decreases. The decreases in film strength and increases in ability to elongate with increasing glycerol content, indicated by decreasing tensile strength and increasing elongation at break values, may be attributed to the reduction in the number of intermolecular cross-links between pectin molecules within films (Gontard *et al.* 1993, Shaw *et al.* 2002b). Doolittle (1965) suggested that plasticizer molecules bind with water molecules and shield active centres along polymer chains, thereby decreasing intermolecular interactions and increasing intermolecular distance.

According to the results displayed in Table 4, there is a significant increase of water permeability values with the higher content of pectin. The results show how varying the concentration of pectin could influence the water barrier property of the films as studied for whey protein films by McHugh *et al.* (1994). It was ob-

served that with an increase of glycerol content there was an increase in water vapour permeability values of pectin films, but the differences were not significant and ranged from 1.50 to 1.68 g m⁻¹ Pa⁻¹ s⁻¹. An increase of plasticizer concentration normally causes an increase in the permeability of hygroscopic or hydrophilic films due to a reorganisation of the film network and a consequent increase of the free volume (Cuq *et al.* 1997). The hydrophilic nature of plasticizer (nature and amount) significantly affects the moisture barrier ability of biopolymer films. Similar results were noted by Shaw *et al.* (2002a); when the concentration of glycerol was 50, 60 and 70% (w/w) in protein films, water vapour permeability increased slightly when the glycerol amount increased in the film-forming solution. These results correspond with those of McHugh and Krochta (1994) and Bodnar *et al.* (2007) who reported that water vapour permeability values increased with increasing glycerol concentration. Both glycerol and water act as a plasticizer with a synergistic effect dominated by the plasticizing power of water.

Table 4. Water vapour permeability of pectin films. Values followed by the same letter are not significantly different at $p < 0.001$

Film	Water vapour permeability (g m ⁻¹ Pa ⁻¹ s ⁻¹)
1.5P_50G	1.21 ± 0.23 ^a
2.5P_50G	1.63 ± 0.18 ^a
3.5P_50G	2.25 ± 0.23 ^b
2.5P_30G	1.50 ± 0.13 ^a
2.5P_50G	1.63 ± 0.18 ^a
2.5P_70G	1.68 ± 0.02 ^a

CONCLUSIONS

1. Lightness of films decreases with the higher content of pectin at the concentration range of 1.5-3.5%, but colour parameters a^* and b^* increase. Finally, the increase in total colour difference is correlated with increasing pectin concentration. Films containing the largest content of glycerol have the highest value of total colour differences.

2. Mechanical properties of pectin films are significantly influenced by pectin and glycerol concentration. The highest value was obtained at the highest pectin concentration and the lowest glycerol content. Elongation at break of the films does not depend on the pectin concentration, but the highest value was observed for the highest glycerol concentration.

3. Water vapour permeability of films depends on pectin concentration but not significantly on glycerol content. The lowest value of permeability was obtained for the lowest pectin concentration.

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BARWA, WŁAŚCIWOŚCI MECHANICZNE I PRZEPUSZCZALNOŚĆ PARY WODNEJ FILMÓW PEKTYNOWYCH

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Streszczenie. Celem pracy było zbadanie wpływu ilości pektyny i glicerolu na barwę, właściwości mechaniczne i przepuszczalność pary wodnej filmów pektynowych. Filmy pektynowe otrzymano z wodnych roztworów powłokotwórczych pektyny jabłkowej o stężeniu 1,5; 2,5 i 3,5%. Glicerol został dodany jako plastyfikator w ilości 30, 50 i 70%. Barwa została zmierzona w systemie $L^*a^*b^*$, obliczono bezwzględną różnicę barwy. Właściwości mechaniczne wyrażono jako siłę zerwania oraz wydłużenie. Przepuszczalność pary wodnej zmierzono w temperaturze 25°C przy różnicy w wilgotnościach względnych środowiska 30-100%. Na podstawie otrzymanych wyników zaobserwowano wzrost bezwzględnej różnicy barwy, przepuszczalności pary wodnej i siły zerwania filmów zawierających wyższe ilości pektyny. Glicerol wpłynął na zwiększenie wydłużenia i przepuszczalności pary wodnej analizowanych filmów.

Słowa kluczowe: filmy jadalne, pektyna, barwa, właściwości mechaniczne, przepuszczalność pary wodnej