# Sorption and Wetting Properties of Pectin Edible Films

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**Abstract**

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The water vapour sorption kinetics and isotherms of pectin films prepared by the casting method were determined. The measurement of water vapour sorption kinetics was conducted using a saturated sodium chloride solution to obtain constant relative humidity of the environment (75.3%). The measurement was carried out at the temperature of 25°C over a 24 h period. The water vapour adsorption rate was the highest in the first hours of the process. The exponential equation fitted well the experimental data of water vapour adsorption with time. Glycerol concentration in the analysed films affected the increasing water vapour adsorption. The water vapour sorption isotherms were analysed using the saturated salt solutions with water activity from 0.113 to 0.901 for 3 months at 25°C. The sorption isotherms curves had a sigmoidal shape for all films. Glycerol content affected water vapour adsorption during 3 months. Peleg's equation was appropriate for the mathematical description of the sorption isotherms. The microstructure of pectin films showed different internal arrangement as a function of the film composition.

**Keywords**: pectin films; glycerol; sorption isotherms and kinetics

With the increasing consumers' concern about the limited natural resources and the environment, the use is explored of renewable resources in view of the production of biodegradable materials that can reduce the waste disposal problems. Accordingly, a variety of renewable biopolymers have been investigated for the development of biodegradable materials to substitute or complement their non-biodegradable petrochemical-based counterparts (Alves *et al.* 2010). Active packaging systems based on the application of packaging films with incorporated antimicrobial agents provide one of the promising trends in food processing (Hanušová *et al.* 2009). The components of edible films and coatings can be divided into three categories: hydrocolloids, lipids, and composites (Kokoszka *et al.* 2010). Several studies have reported the use of polysaccharides from different sources for the preparation of films and coatings with different properties, and have indicated that these carbohydrates are promising materials (Mali *et al.* 2005).

The addition of plasticisers leads to a decrease in intermolecular forces along the polymer chains which improves the flexibility and chain mobility. They are added to enhance the film flexibility, decrease brittleness, avoid shrinking during handling and storage and, therefore, to make it easier for the film to be peeled off the support during the manufacture (Guilbert *et al.* 1996). Several studies, however, have also evidenced adverse effects of plasticisers on edible film attributes. Most of them describe an increase in gas, solute, and water vapour permeability and a decrease in cohesion affecting mainly the mechanical properties. The characteristics of films based on polyuronated matrices depend therefore on an equilibrium between

the degree of crosslinking with $Ca^{2+}$ (necessary to reduce the solubility in water but inducing brittleness) and the addition of plasticisers for a better workability. Above the critical concentration the plasticiser can exceed the compatibility limit with the biopolymer and phase separation with the plasticiser exclusion is usually observed. The commonly used plasticisers in edible carbohydrate based films are polyols, mainly glycerol and sorbitol (Yang & Paulson 2000).

Pectin is a structural heteropolysaccharide with a comples structure found in the primary cell walls of terrestrial. Pectin, when extracted from higher plants, contains smooth (linear) regions and hairy, branched regions. The linear, smooth regions are made up of $\alpha$-(1-4)-linked D-galacturonic acid residues, some of which are methylesterified. The hairy region contains a backbone of the repeating disaccharide ($\rightarrow$4)-$\alpha$-D-GalpA-($\rightarrow$2)-$\alpha$-L-Rhap-($\rightarrow$). The Rhap residues are substituted at C-4 with neutral oligosaccharide side chains composed mainly of arabinose and galactose residues. In sugarbeet pectin, these arabinose and galactose residues in the neutral sugar side chains are substituted by ferulic acid residues linked at C-2 (arabinose) or C-6 (galactose) positions (Siew & Williams 2008). The degree of esterification, describing the percentage of acid groups present in the ester form, determines to a large extent the solubility of pectin and its gelling and film forming properties, and hence its industrial applicability. The degree of methylesterification varies with the origin of the plant source and the processing conditions e.g. storage, extraction, isolation, and purification (Kirby *et al.* 2008).

Water activity differences among the food components, the food domains, and the environment outside the package introduce the driving forces for the water transport. The water transport ceases when there is no difference in water activity, i.e., when the water activities converge to a common equilibrium value. For the systems in which this equilibrium water activity is undesirable for one or more of the components, the product shelf-life is determined by the dynamics of the water transport process. In addition, the understanding of the relative importance of the different mechanisms controlling the moisture transfer through hygroscopic films is also important for designing new films with improved and selective barrier properties. Consequently, both the equilibrium properties and the kinetics of water transport through the packaging material are of great importance

(Muller *et al.* 2009). The sorption isotherms characterise the relationship between the water content and its activity at constant temperature and have been widely conduced on edible films (Shih *et al.* 2011). The results obtained by experimental determination of equilibrium water vapour sorption isotherms are treated by different models. In more recent years, the Guggenheim, Anderson, and de Boer (GAB) equation has been used by many authors for the description of sorption isotherms over a wider range of relative humidity than Brunauer, Emmett, and Teller (BET) model could cover (Volková *et al.* 2012). The selection of an appropriate mathematical model of the sorption behaviour is difficult because of the complex chemical composition and structure of food (Raharitsifa & Ratti 2010). The Peleg's model has been used to describe the sorption processes in various foods, this paper presents its application to pectin edible films.

Since moisture is adsorbed at different rates by different materials or by the same material under different humidity conditions, it appeared interesting to characterise the whole sorption process and not only the equilibrium conditions. Appropriate selection of the plasticiser type and concentration will be helpful in controlling the moisture content and moisture adsorption rate of a film, thereby improving the film stability under varying relative humidity conditions (Mali *et al.* 2005).

The information on the effects of pectin and plasticiser on pectin films and their sorption characteristics is not available at present. In this context, the objectives of this study were to determine the effects of pectin and glycerol concentrations on the water vapour sorption kinetics and isotherms of pectin films. The microstructure of the cross section of pectin films was also presented.

## MATERIAL AND METHODS

Apple amidated pectin type NEJ-A2 standardised by adding of sucrose, containing 10% of neutral sugars and not less than 65% of galacturonic acid with the degree of metoxylation less than 50% of galacturonic acid residues was obtained from PEKTOWIN (Jaslo, Poland). Anhydrous glycerol and calcium chloride were purchased from POCH S.A. (Gliwice, Poland). The salts for the preparation of saturated solutions for the determination of water vapour sorption isotherms, including

lithium chloride, potassium acetate, magnesium chloride, potassium carbonate, magnesium nitrate, sodium nitrate, sodium chloride, ammonium sulphate, and barium chloride were supplied by POCH S.A. (Gliwice, Poland). Saturated salt solution of sodium chloride (POCH, Gliwice, Poland) was used to control relative humidity of 75% at 25°C for water vapour sorption kinetics.

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 the 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 (RH) in a ventilated climate chamber. After this time the dried films 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. The compositions and symbols of the films are given in Table 1.

Water content was determined through the weight loss revealed by the film after 4 h drying at 105°C. Three repetitions were made.

The measurement of water vapour sorption kinetics was conducted in two repetitions for each type of film using a stand which ensured continuous measurement of the mass increase in conditions of constant temperature and relative humidity. Saturated sodium chloride solution was used to obtain constant relative humidity of the environment (75.3%). The measurement was carried out at 25 ± 1°C over the 24-h period. The investigated samples consisted of 0.25 g of films, and their mass increase was registered by means of the "measurement for DOS" computer software (IBM Corporation, Armonk, USA). An exponential equation (Kowalska *et al.* 2006) was used for the mathematical interpretation of the obtained results:

$$u = a + b(1 - \exp^{(-c\tau)}) \qquad (1)$$

where:
$u$     – water content (g water/g d.m.)
$a, b, c$ – constant parameters of equation
$\tau$     – time (h)

Water vapour sorption isotherms determinations were conducted based on the static gravimetric method (Spiess & Wolf 1987) by equilibrating film samples (0.25 g in triplicate) at 25°C in desiccators with saturated salt solutions at water activity from 0.113 to 0.901. Water content of the samples was determined as the weight gain at equilibrium. Water activity of the samples after storage was evaluated by using Aqualab Series 3 Quick Start (Decagon Devices, Inc., Pullman, USA).

The Peleg equation (Peleg 1993) was used to fit the experimental data of water vapour sorption isotherms of pectin films:

$$u = A \times a_{\text{w}}^{B} + C \times a_{\text{w}}^{D} \qquad (2)$$

where:
$A, B, C, D$ – constants of equation
$a_{\text{w}}$     – water activity
$u$     – water content (g water/g d.m.)

The film microstructure was observed by scanning electron microscopy (Quanta 200; FEI, Brno, Czech Republic). A 5 mm × 5 mm film was fixed on the support using silver paste, with an angle of 90° to the surface, which allowed the observation of the film cross section. No particular film preparation was necessary. The films were observed at a magnification of 5000× and an intensity of 20 kV.

The adequacy of the fitted exponential equation and Peleg's model was evaluated by the determination coefficient ($R^2$) and mean relative error (*MRE*) (Jamali *et al.* 2006).

Table 1. Composition of pectin film-forming solutions

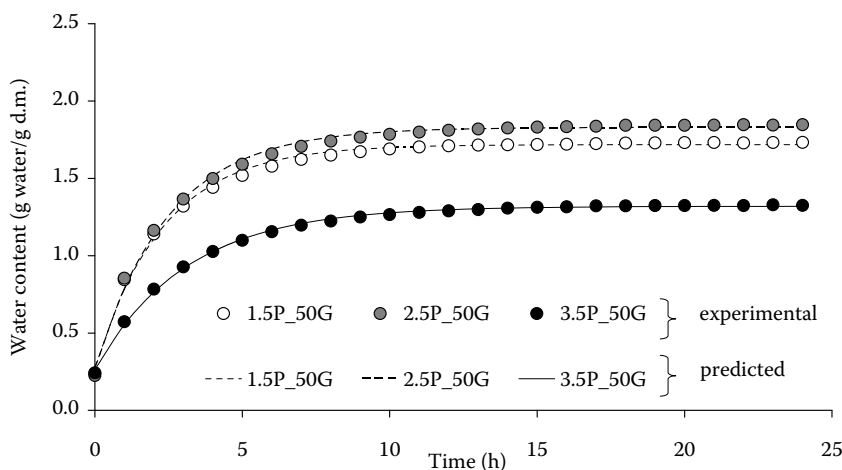| Film | Pectin (g) | Glycerol (g) | CaCl$_2$ (g) | Water (g) |
|------|-----------|--------------|-------------|-----------|
| 1.5P_50G | 1.5 | 0.75 | 0.015 | 97.735 |
| 2.5P_50G | 2.5 | 1.25 | 0.025 | 96.225 |
| 3.5P_50G | 3.5 | 1.75 | 0.035 | 94.715 |
| 2.5P_30G | 2.5 | 0.75 | 0.025 | 96.725 |
| 2.5P_50G | 2.5 | 1.25 | 0.025 | 96.225 |
| 2.5P_70G | 2.5 | 1.75 | 0.025 | 95.725 |

Figure 1. Water content as a function of adsorption time for films with different content of pectin

## RESULTS AND DISCUSSION

All pectin films were easily detachable, transparent, without any cracks and pores. The films with 30% of glycerol were more brittle, and those with 70% of glycerol more flexible due to the plasticising effect in the film network. Preliminary work showed that the film formed without the plasticiser was very brittle and broke when being peeled off. BANKER (1966) reported that the plasticiser can retard or enhance the moisture transmission depending on its concentration. The plasticiser can reduce the internal hydrogen bonding and increase the intermolecular spacing.

### Water vapour sorption kinetics

*Effect of pectin content.* The effect of the pectin content on the water vapour sorption kinetics of the analysed films at relative humidity of 75.3% is presented in Figure 1. A significantly lower water vapour sorption was observed with the film with the highest (3.5%) content of pectin. This could

be the result of the structure formation. Higher values of the water content during 24 h of water vapour sorption were obtained for the films with 1.5 and 2.5% of pectin which can be explained by the difference in the thickness and film microstructure (Figure 5). The films with higher contents of pectin had a higher film thickness. The films with a high thickness and more discontinuities in the structure had a lower ability to absorb water vapour during a short time (24 h). None of the analysed films reached the equilibrium within the 24 h timeframe set for the tests. The water vapour sorption kinetics curves of the analysed films had a similar course. The films differed in the initial water content, which means that the driving forces of the sorption process also differed. Depending on the water activity of the environment, the sorption proceeded with different intensity. Regardless of that, in each case the largest changes in the water content were observed during the initial 5 h of the process. BUONOCORE *et al.* (2003) also observed the most intensive adsorption process in the first hours. For the mathematical description

Table 2. Parameters of exponential equation fitted water vapour sorption kinetics for pectin films

| Films | Coefficients of equation | | | $R^2$ | MRE (%) | Water content (g water/g d.m.) | | | |
|---|---|---|---|---|---|---|---|---|---|
| | | | | | | experimental | | predicted | |
| | $a$ | $b$ | $c$ | | | $u_0$ | $u_{24}$ | $u_0$ | $u_{24}$ |
| 1.5P_50G | 1.72 | 0.84 | 0.43 | 0.996 | 1.74 | 0.24 | 1.73 | 0.28 | 1.72 |
| 2.5P_50G | 1.83 | 0.85 | 0.40 | 0.996 | 2.01 | 0.23 | 1.85 | 0.27 | 1.84 |
| 3.5P_50G | 1.32 | 0.80 | 0.32 | 0.998 | 1.11 | 0.24 | 1.33 | 0.26 | 1.32 |
| 2.5P_30G | 1.58 | 0.86 | 0.43 | 0.998 | 1.44 | 0.19 | 1.59 | 0.22 | 1.58 |
| 2.5P_50G | 1.83 | 0.85 | 0.40 | 0.996 | 2.01 | 0.23 | 1.85 | 0.27 | 1.84 |
| 2.5P_70G | 2.15 | 0.89 | 0.34 | 0.993 | 3.01 | 0.23 | 2.18 | 0.31 | 2.15 |

$R^2$ – correlation coefficient, MRE – mean relative error, $u_0$ – initial water content, $u_{24}$ – final water content

of the relationship between the water content in the analysed films and the sorption time range an exponential equation was chosen (Figure 1 and Table 2). The following factors had a substantial influence on the choice of Eq. (2) for modelling the water vapour sorption curves over the investigated time range (Table 2). A high correlation coefficient ($R^2$) and a comparatively low mean relative error (*MRE*) for most of the relation for the experimental and predicted data concerning water content at the beginning and after 24 h of water vapour sorption. The correlation coefficient ($R^2$) of the chosen exponential equation for the examined films with different contents of pectin was high (Table 2). The values of *MRE* were low. The considerable *MRE* value increase resulted from the discrepancies between the experimental and predicted initial water contents in the films. All samples were marked by similar experimental and calculated water contents after 24 h of the sorption process; therefore, it was decided to apply an exponential equation for the description of sorption kinetics. The equation chosen was used also to describe the shape of water vapour sorption kinetics in food products, i.e. freeze-dried strawberries (Ciuszyńska & Lenart 2010).

The experimental initial water content in the films with different contents of pectin was about 0.22 g water/g d.m. per day and more higher after 24 h of the adsorption process (Table 2). The predicted values of the water content in the pectin films resembled the experimental ones. The absorbed water vapour in pectin films made a plasticisation effect in the material thus facilitating the adsorption. The highest sorption rate (*c* parameter of Eq. 1) was obtained for the film with the lowest pectin content.

***Effect of glycerol content.*** Water content as a function of the adsorption time for the pectin films with different contents of glycerol is presented in Figure 2. Water vapour sorption kinetics curves of the films analysed had similar courses. The films with 70% of glycerol showed the highest values of the water content compared to those in the films containing 30 and 50% of glycerol. None of the films reached the equilibrium within the 24 h timeframe set for the tests. An increase in water content in edible films with increased glycerol concentration has been previously observed. The increasing effect of glycerol concentration on the water content was demonstrated for whey (Oses *et al.* 2009), soy (Cho & Rhee 2002), starch (Talja *et al.* 2008), and carrageen (Karbowiak *et al.* 2006) films as well for composite (alginate and pectin) films (da Silva *et al.* 2009). Mali *et al.* (2005) analysing the water vapour adsorption showed increasing water content values due to glycerol or sorbitol concentration. A higher plasticiser content unfastens the polymeric structure allowing more charged sites to become accessible to water molecules. More important, glycerol itself is very hygroscopic and, therefore, probably responsible for most of the water uptake (da Silva *et al.* 2009). For the mathematical description of the relationship between the water content in the analysed films and sorption time range an exponential equation was chosen (Figure 2 and Table 2). The correlation coefficient of the chosen exponential equation for the examined films with different contents of pectin was high (Table 2). The values of the mean relative error were low. The predicted values of the initial and final (after 24 h of process) water contents in the pectin films resembled to the experimental ones. The highest
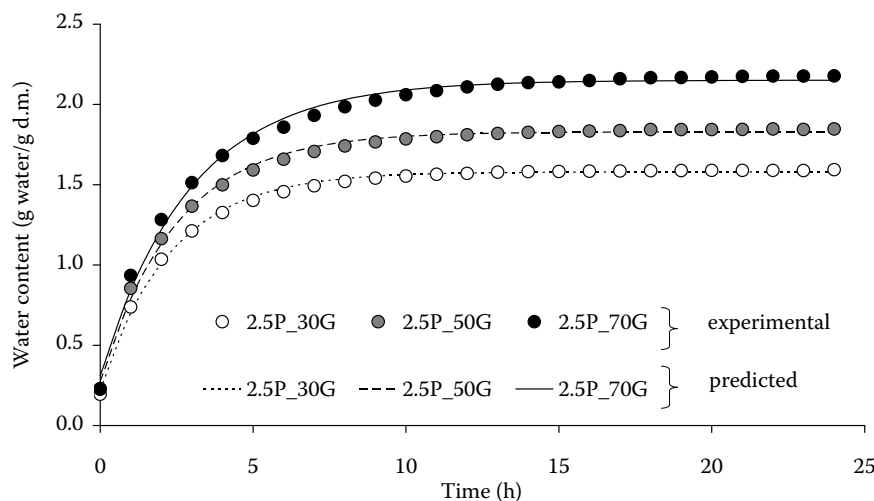


Figure 2. Water content as a function of adsorption time for pectin films with different content of glycerol
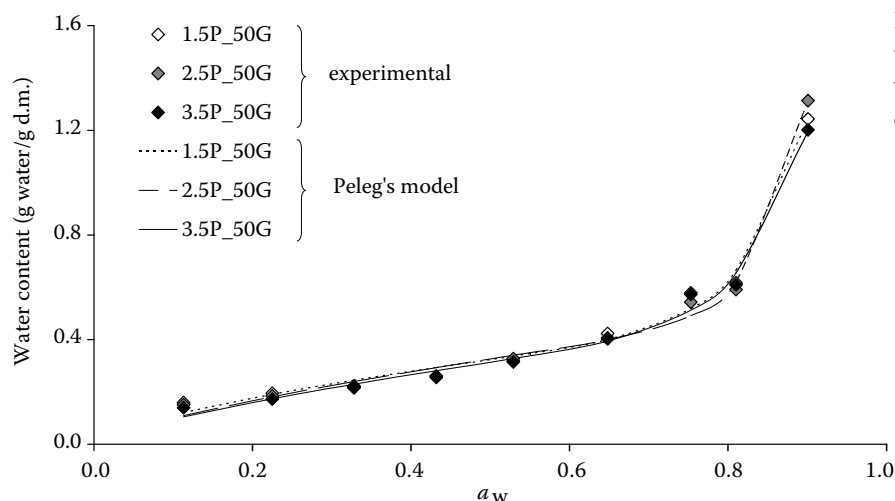
Figure 3. Experimental data and isotherms curves by Peleg's equation for water vapour sorption of films with different content of pectin

sorption rate (*c* parameter of Eq. 1) was obtained for the film with the lowest glycerol content.

## Water vapour sorption isotherms

***Effect of pectin content***. Figure 3 shows the sample graphic points obtained from Peleg's model adjusted to experimental water vapour sorption isotherm data for pectin films with different contents of pectin in dry matter. The high coefficient of determination (> 0.989) and the low value of the mean relative error confirmed good fitting of Peleg's model to the experimental data (Table 3). The water vapour sorption isotherms for all pectin films showed a similar course which was not affected by the pectin content.

***Effect of glycerol content***. Water vapour sorption isotherms of pectin films with different contents of glycerol are displayed in Figure 4, and the Peleg's model parameters are shown in Table 3. The high coefficient of determination (> 0.992) and the low value of the mean relative error confirmed good fitting of Peleg's model to the experimental data. The

addition of plasticiser provides more active sites by exposing its hydrophilic hydroxyl groups in which the water molecules could be adsorbed (Mali *et al.* 2005). The water sorption isotherms for the films showed a sigmoidal shape and were influenced by the plasticiser concentration (Figure 4). Higher levels of the plasticiser increased the films water affinity and this result could be attributed to the hydrophilicity of the plasticiser, whose hydroxyl groups are capable to interact with water by means of hydrogen bonds. The films with a higher content of glycerol showed a higher capacity to adsorb water in all concentrations and at all relative humidity conditions. Glycerol molecules are small and possess a high capacity to interact with starch chains, enhancing the molecular mobility and increasing the free volume in the film matrix. Besides, glycerol has a more hydrophilic character than sorbitol and these combine effects contributed to the higher water affinity of glycerol films (Sothornvit & Krochta 2001).

The initial water content was similar in all pectin films. The equilibration time of 3 months for the sorption isotherms was selected, which was higher than those used by other researchers in the litera-

Table 3. Sorption isotherm constants and coefficient of determination ($R^2$) for Peleg's equation for pectin films

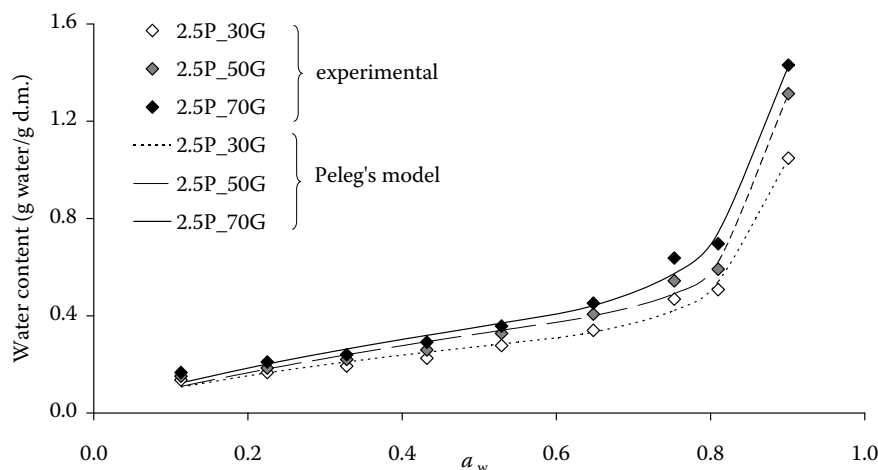| Film | Coefficients of equation | | | | $R^2$ | *MRE* (%) |
|------|------|------|------|------|-------|-----------|
|      | *A* | *B* | *C* | *D* | | |
| 1.5P_50G | 2.55 | 11.6 | 0.51 | 0.66 | 0.989 | 8.73 |
| 2.5P_50G | 4.06 | 15.5 | 0.54 | 0.74 | 0.993 | 7.93 |
| 3.5P_50G | 2.38 | 11.6 | 0.52 | 0.73 | 0.991 | 7.36 |
| 2.5P_30G | 2.41 | 12.6 | 0.42 | 0.63 | 0.992 | 6.95 |
| 2.5P_50G | 4.06 | 15.5 | 0.54 | 0.74 | 0.993 | 7.93 |
| 2.5P_70G | 3.14 | 12.1 | 0.58 | 0.71 | 0.992 | 7.52 |

Figure 4. Experimental data and isotherms curves by Peleg's equation for water vapour sorption of pectin films with different content of glycerol

ture (Jangchud & Chinnan 1999). The change in the water content with $a_w$ was small in all films for RH of 0 to 0.75 (Figures 3 and 4), which was also observed by other researchers (Jangchud & Chinnan 1999; Shih *et al.* 2011). In the range of water activity between 0.75 and 0.90 the water content increases sharply. The water vapour sorption isotherms of pectin followed typical type III behaviour according to Brunauer *et al.* (1940). The shape of all sorption isotherms is common for high sugar food, which absorbs a relatively small amount of water at low water activities and large amounts at a high water activity. Similar observations were presented by Ozilgen (2011) for dried food products.

A large number of equatiosn have been suggested to model the general sorption function, $M = f(a_w)$. To be successful, any modelling equation should give as good a fit as possible to the experimental data over a range of $a_w$ using a minimum of adjustable parameters (Coupland *et al.* 2000). Peleg (1993) constructed Eq. (2) for generating the sigmoidal form frequently observed. This model has four adjustable parameters (*A*, *B*, *C*, *D*), so it is expected to give generally better fits than the others. Peleg's equation has been widely used to describe sorption isotherms for many products. Lewicki (1998) applied this model to describe the sorption isotherms for 27 products. He demonstrated that the highest probability of fitting the experimental data with the minimum mean relative error is guaranteed by Peleg's model. In a similar vein, Palou *et al.* (1997) surmised that Peleg's model was best suited for the description of isotherms for cookies and chips, because the relative standard deviation obtained was lower than 7%. Ciurzynska and Lenart (2010) se-

lected Peleg's model as the most appropriate for the mathematical descriptions of the sorption isotherms for osmotically dehydrated freeze-dried strawberries. Kowalska *et al.* (2005) affirmed that the characteristic sigmoidal shape of the observed isotherms is related to the occurrence of a monomolecular sorption range in the milieu of water activity $a_w < 0.3$ a multi-layer sorption for $0.3 < a_w < 0.65$, and capillary condensation at $a_w > 0.65$. Peleg's model was also used to describe the sorption isotherms for edible films, i.e. whey films (Coupland *et al.* 2000).

## Film microstructure

***Effect of pectin content***. Scanning electron microscopy observations were carried out to contribute to a better insight in the homogeneity and the microscopic structure of films. Figure 5 shows the scanning electron micrographs cross section for films with different contents of pectin (Table 1). Based on various film-forming polysaccharides, single polymeric films may have different structures (Phan *et al.* 2009). The appearances of both sides of the film were different. The film side facing the casting plates was shiny while the other one was dull. The films obtained from 1.5% and 3.5% of pectin showed more structural discontinuities. It can be results of drying condition or chemical composition (amount of pectin). Film containing 3.5% of pectin showed uneven air side surface in contrary to other films which were smooth. A different internal arrangement was observed as a function of the film composition. Pectin network is less organised at higher concentration in the films. It could be also observed, that the
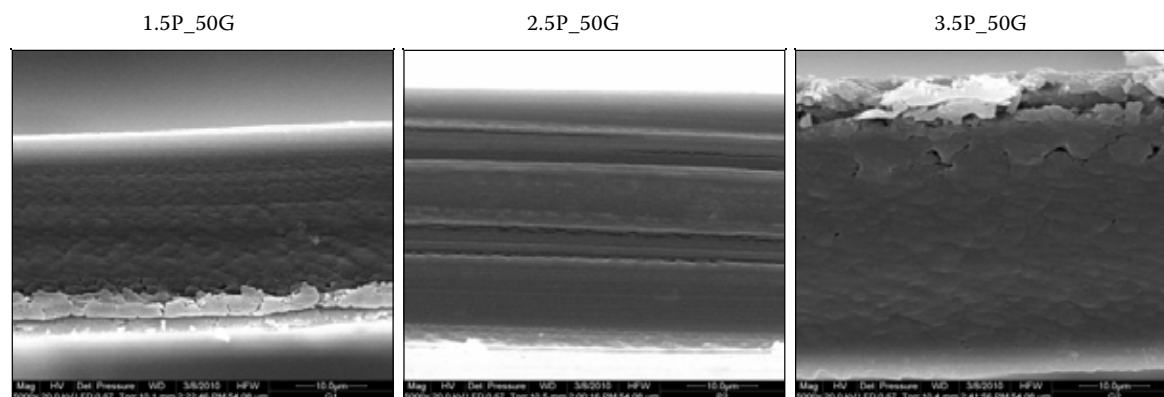
1.5P_50G                          2.5P_50G                          3.5P_50G



Figure 5. Scanning electron micrographs of cross section of films with different content of pectin (magnification 5000×). The evaporation surface of the film is on the top

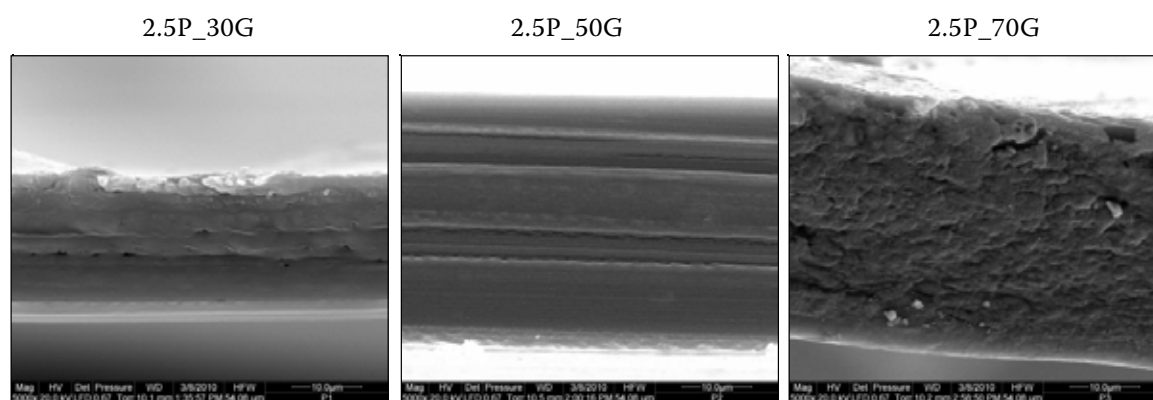2.5P_30G                          2.5P_50G                          2.5P_70G



Figure 6. Scanning electron micrographs of cross section of pectin films with different content of glycerol (magnification 5000×). The evaporation surface of the film is on the top

film thickness increased when the pectin content increased in the film forming solutions. Similar results were presented by Kokoszka *et al.* (2010) for whey films.

*Effect of glycerol content*. Pectin films with different contents of glycerol (Table 1) exhibited homogenous surface on the air and support sides (Figure 6). This could be due to the lack of miscibility of the components. The film with the highest content of glycerol (70%) showed a porous and grainy structure. The pores probably constitute plasticisation zones distributed within the film matrix. Similar observations were made with amaranth flour films by Tapia-Blacido *et al.* (2011). Pectin films with 30 and 50% of glycerol showed a multi-layer structure in contrary to the film with the highest content of glycerol, 70%, where the structure was porous. Wang *et al.* (2010) noticed a similar dependence for composite films based on whey protein isolate, gelatin, and sodium alginate.

## CONCLUSIONS

Water vapour sorption kinetics curves of the investigated pectin films have a similar course and none of them reached the equilibrium within 24 hours. Water vapour adsorption is more rapid in the initial stages of storage and lower amounts of water were adsorbed as the time advanced. Hydrophilicity of the plasticiser and its concentration are more important factors than the pectin content in determining the moisture affinity of pectin films. The exponential equation selected fits well the experimental data of water vapour sorption kinetics.

The sorption isotherm curves for the investigated pectin films have a sigmoidal shape, characteristic to most food products. On this basis, sorption isotherms can be classified as type III behaviour according to Brunauer's classification. Peleg's equation is useful to fit water vapour sorption isotherm data. The sorption isotherms of the pectin films

show that pectin is very hydrophilic, affecting the moisture change with relative humidity. The water content increases slowly for films from 0 to 0.75 water activities, but increases very sharply from 0.75 to 0.90.

A different internal arrangement is observed as a function of the film composition. Low and high contents of pectin tend to disturb the internal structure in films. The cross sections of the investigated films show a compact microstructure, which is likely caused by strong cohesion forces appearing during the slow-drying of aqueous- materials.

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