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The Effects of Drying Temperatures and Oil Contents on Properties of Biodegradable Film from Konjac Flour

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Abstract

Biodegradable film is material made of biopolymer that can degrade naturally. Konjac flour is a soluble dietary fiber obtained from the root of the konjac plant (*Amorphophallus konjac*). The objective of this research was to study the effects of drying temperatures and olive oil concentrations on physical and mechanical properties, water vapor permeability (WVP) and solubility of biodegradable film from Konjac flour. The concentrations of olive oil were varied from 0, 1 to 3% in the experiment. The film drying was performed using a heat pump dryer at drying temperatures of 45 and 55°C. The results showed that tensile strength and solubility of film were significantly decreased ($p \leq 0.05$) when the olive oil concentration and drying temperature were increased. Percentage of elongation and thickness of the film were significantly increased ($p \leq 0.05$) when olive oil concentrations and drying temperatures were raised. In addition, water vapor permeability of the film was significantly increased ($p \leq 0.05$) when the olive oil concentrations and drying temperatures were increased.

Keywords: Biodegradable film, Drying, Konjac flour, Mechanical properties, Water vapor permeability

1 Introduction

Packaging is part of marketing tool and acting as a silent salesman for consumer information purposes. It places a physical barrier between food products and the outside environment, especially those susceptible to oxidative and microbiological deterioration. The most common materials used for packaging are paper, fiberboard, plastic, glass, steel, and aluminum. Oil-derived synthetic plastics are commonly used. It is because of their advantages over other packaging materials in terms of sturdiness and low weight (Go´mez-Guille´ et al., 2009). However, they pose a serious global environmental problem by generating large volumes of non-biodegradable waste (Kirwan and Strawbridge, 2003). In addition to safety and environmental issues, recycling of plastics is complicated for technical and economic reasons (Aguado and Serrano, 1999). Thus, new biodegradable films made from edible biopolymers from renewable sources could become an important factor in reducing the environmental impact of plastic waste (Tharanathan, 2003). Proteins, lipids and polysaccharides are the main biopolymers employed to make edible films. The type of biopolymer to use in the film determines the properties of the material as a barrier to water vapour, oxygen, carbon dioxide, and lipid transfer in food systems. Films composed primarily of proteins or polysaccharides have suitable overall mechanical and optical properties but are highly sensitive to moisture and exhibit poor water

vapour barrier properties (Guilbert et al., 1996). This may represent a drawback when they are applied to food products with high moisture contents, because the films may swell, dissolve, or disintegrate upon contact with the water. However, the concept of biopolymers only is valid if the oil utilization life-cycle is shorter for biopolymers than for synthetic polymers (Scott, 2000).

Konjac glucomannan (KGM) is the main polymer which are from agricultural product, readily available, biodegradable and highly safe. They possess special nutritional and good film-forming properties (Cheng et al., 2008). For instance, KGM is found to have functions such as has the ability to lower blood cholesterol and sugar level, help with weight loss, promote intestinal activity and immune function etc (Zhang et al., 2005)

Edible films from biodegradable material can be formed by two main processes, i.e., casting and extrusion (Hernandez and Krochta, 2008). The film formation process most often reported in the scientific literature is the casting method. Briefly, it involves dissolving the biopolymer and blending it with plasticizers and/or additives to obtain a film-forming solution, which is cast onto plates and then dried by driving off the solvent. The extrusion method relies on the thermoplastic behaviour of proteins at low moisture levels. Films can be produced by extrusion followed by heat-pressing at temperatures that are ordinarily higher than 80°C. This process may affect film properties (Kaya and Kaya, 2000).

Drying at higher temperatures will affect on quality of the biodegradable film. The physical and chemical properties of the film are changes (Ekthamasut and Akesowan, 2001). This is due to the biodegradable film is a thin sheet. Most types of edible films were prepared by hot air drying (Kaya and Kaya, 2000). Heat pump dryer is one of the fastest drying methods. Although drying at a low temperature it can still reduce moisture in samples. Heat pump dryer is very good conditions for heat sensitive materials to dry, as it enables independent control of temperature and relative humidity. This technology requires far less energy, as the system can recover the latent heat in a closed loop, and be conducted independent of ambient weather conditions (Perera and Rahman, 1997). Their advantages include higher energy efficiency due to the high coefficient of performance and better product quality (Strommen et al., 2000).

The objective of this paper was to investigate the effects of drying temperatures and olive oil concentrations on properties of biodegradable film. Konjac flour was used as biodegradable base for film forming. Heat pump dryer was used for drying process. The drying temperature was set at 45 and 55°C. The olive oil were varied from 1 and 3% w/w. Properties of film that were evaluated including tensile strength, elongation, thickness, solubility and water vapor permeability.

2 Materials and Methods

2.1 Materials

Konjac glucomannan (food grade) is obtained from the Yunnan Genyun Konjac Resource Corp., Kunming (Biojade, Yunnan, PR China). Potassium hydroxide and Glycerin were obtained from Thai Food and Chemical Co., Ltd. Olive oil was purchased from a retail store.

2.2 Film Preparation

A film-forming solution was prepared by weighed of konjac flour (1% w/v) of 100 ml distilled water and constant stirring via at room temperature (25°C) for 3 h. The 0.14% w/v potassium hydroxide (KOH) was added before blending for 15 min. The mixture was then left to stand for 1 h at room temperature. The glycerol was added at 30% of the weight of Konjac flour and blending for 20 min. Olive oil was added in the amount of 1 and 3% w/v konjac and homogenized at 12,500 rpm for 5 min.

2.3 Film Drying

Each conditions of film were prepared by the solution 33.75 g portion was poured and spread onto a level, rectangular Perspex plate fitted with rims around the edge to give a 7.5x3.5 cm^2 film forming area. The solution were allowed to dry at the temperatures of 45 and 55°C using a heat pump dryer for the obtained dried film. The obtained dry film was peeled off and stored in a desiccator containing saturated sodium chloride (NaCl) solution with 75% RH at room temperature until analysis.

2.4 Film Properties Determination

1) Film thickness determination. The film thickness was measured using a micrometer (Mitutoyo, Tokyo, Japan) with an accuracy of 0.001 mm. Each film sample was measured at six random positions along the strip; an average value was reported. The mechanical properties and Water vapor permeability (WVP) were calculated using the average thickness of each film sample.

2) Tensile strength and elongation tests. The measurement of the mechanical properties of biodegradable film was carried out using a texture analyzer (Texture Analyzer model TA.XT. Plus, Stable Micro Systems LTD, UK). After conditioning a biodegradable film, sample was cut into a 25.4 mm wide and 100 mm long strip. Initial grip separation 60 mm and speed were set at 60 mm min^{-1}, respectively. Tensile strength was calculated by dividing the maximum load for breaking the film by its cross-sectional area. Percent elongation was determined by dividing the film elongation at rupture by the initial grip separation.

3) Water vapor permeability of the films was determined using an aluminum cup with a diameter and height of 5.2 cm and 4.2 cm, respectively. Aluminum cup contained 100 g silica gel (0% RH) that was dried in oven (BINDER model ED 53 E2, Tuttlingen, Germany) at 120°C for 24 h. The headspace for the aluminum cup was 1 cm from the opening of the aluminum cup. Films were cut circularly with a diameter slightly larger than the diameter of the aluminum cup. They were covered and sealed using melted paraffin. These aluminum cups were then placed in a desiccator containing distilled water (100% RH). The aluminum cups were weighed at 1 h intervals over 5 h periods. Weight gain graphs were plotted with respect to time. The slope of each line was calculated by linear regression ($R^2 \geq 0.99$). The measured WVP of the films was determined as follows (Sobral et al., 2001).

$$WVP = (WVTR \times t)\, DP^{-1} \quad (1)$$

Where *WVTR* is the water vapor transmission rate (g m^{-2} h^{-1}) through a film, calculated from the slope of the straight line divided by the exposed film area (m^2) and *t* is the mean film thickness (mm), and *DP* is the partial water vapor pressure difference (Pa) across the two sides of the film. For each type of film, *WVP* measurements were replicated three times for each batch of films.

4) Solubility in Water is defined as the percentage of film dry matter solubilized after 3 h of immersion in distillation water. The initial percentage of dry matter was determined by drying 2 cm diameter disks in a hot air oven at 105°C for 3 h to determine the weight of film dry matter (*W*). Disks were cut, weighed and immersed in 100 ml of distilled water, with periodic stirring for 1 h at room temperature. The remained films were taken out and filter used filter paper No.4 to determine the weight of filter paper (a_1). It was dried by hot air oven at 105°C for 25 min to determine the final weight of dry matter (a_2). Solubility is reported as the difference between initial and final dry matter with respect to initial dry matter. The measured solubility in water of the films was determined as follows (Rhim et al., 1999).

$$\% \text{ solubility in water} = [W - (a_2 - a_1)]\, W^{-1} \quad (2)$$

where W is weight of film dry matter, a_1 is the weight of filter paper and a_2 the final weight of filter paper dry matter.

3 Results and Discussion

The effects of drying temperature (45 and 55ºC) and olive oil concentrations (0, 1 and 3% w/w) on physical properties, mechanical properties, water vapor permeability and solubility of biodegradable films was investigated. The experimental results are as following.

3.1 Tensile Strength

The tensile strength of biodegradable films containing various olive oil concentrations decreased as oil concentrations was increased. As it can be observed in Table 1, the plasticizer additives glycerol and olive oil have an indicated that all additives significantly influenced tensile strength of the films. The films mixed with more than 1% of olive oil concentrations presented lower values of tensile strength. Therefore, glycerol and olive oil additive acts as a plasticizer concentration agent, changing film mechanical behavior. But there was no significant difference observed in the tensile strength among films at various olive oil concentrations at 55ºC. The films drying at low temperature resulted in slow moisture removal of film solution. This effect caused the better intermolecular interactions and increase mobility of the macromolecules on film structures. Therefore, the structure of dried film was stronger when drying at low temperature. This was similar behavior with the report of Menegalli et al. (1999) that the effect of drying conditions on the mechanical properties was verified with the increase of drying temperature, independent of the relative humidity, caused decrease of the tensile strength and increase of elongation.

Table 1 Results of tensile strength (N mm^{-2}) of film obtained from different drying temperatures and olive oil concentrations.

Olive oil (%w/w)	Temperature (ºC)	
	45	55
0	$12.68^{Aa} \pm 0.46$	$12.02^{Ba} \pm 0.46$
1	$4.56^{Ab} \pm 0.86$	$3.01^{Bb} \pm 0.14$
3	$3.06^{Ac} \pm 0.20$	$2.51^{Bb} \pm 0.03$

^{A,B}Same row with different letters are significantly different ($p \leq 0.05$).

^{a,b}Same column with different letters are significantly different ($p \leq 0.05$).

3.2 Elongation

The elongation at break is a measure of the film's stretch before breaking (Krochta and Mulder-Johnston, 1997). It was shown that trend for elongation value of these films were opposite to tensile strength. The addition of olive oils resulted in more elastic films. Generally, as the film structure softened, tensile strength decreased and elongations were increased in all temperatures and olive oil concentrations. The results are shown in Table 2.

Table 2 Results of elongation (%) of film obtained from different drying temperatures and olive oil concentrations.

Olive oil (%w/w)	Temperature (ºC)	
	45	55
0	$6.88^{Bc} \pm 3.60$	$7.92^{Ac} \pm 1.40$
1	$13.14^{Bb} \pm 1.57$	$24.47^{Ab} \pm 0.73$
3	$27.15^{Ba} \pm 3.03$	$55.12^{Aa} \pm 2.35$

^{A,B}Same row with different letters are significantly different ($p \leq 0.05$).

^{a,b}Same column with different letters are significantly different ($p \leq 0.05$).

This finding was agreed with the previous report that the glycerol concentration significantly affected the tensile strength and percent elongation of the

films (Oses et al., 2009). The films without olive oil had the highest tensile strength and lowest percent elongation.

3.3 Thickness

Biodegradable films produced without olive oils were smooth and transparent having mean thickness about 0.04 mm. When olive oils were added, films became thicker (Table 3). As the concentration of the olive oils increased, the thickness of the films increased. This was due to the fact that olive oil acted as plasticizing agents, to reduce the intermolecular interactions and decrease mobility of the macromolecules. This was similar behavior with the report of Pereda et al. (2012) that a strong interaction between the polymer and the olive oil produced a crosslinking effect, which decreases the free volume and the molecular mobility of the polymer; in this sense, this effect can also been considered as an important increase in the local viscosity of the oil in the vicinity of chitosan chains. For drying temperature, the thickness of films increased with the increase of drying temperature. Since the low drying temperature caused the slow moisture lost of the film solution. Hence, these films shriveled and led to a decrease in the film thickness.

Table 3 Results of thickness (mm) of film obtained from different drying temperatures and olive oil concentrations.

Olive oil	Temperature (°C)	
(%w/w)	45	55
0	$0.040^{c} \pm 0.000$	$0.040^{c} \pm 0.000$
1	$0.138^{Bb} \pm 0.000$	$0.170^{Ab} \pm 0.001$
3	$0.269^{Ba} \pm 0.004$	$0.390^{Aa} \pm 0.002$

^{A,B}Same row with different letters are significantly different ($p \leq 0.05$).

^{a,b}Same column with different letters are significantly ($p \leq 0.05$).

3.4 Water vapor permeability

WVP of these films was increased by adding olive oils concentrations (Table 4).

Table 4 Results of WVP (g mm (kPa h m²)$^{-1}$) of film obtained from different drying temperatures and olive oil concentrations.

Olive oil	Temperature (°C)	
(%w/w)	45	55
0	$0.476^{Bb} \pm 0.030$	$1.276^{Ac} \pm 0.030$
1	$0.560^{Bb} \pm 0.045$	$1.533^{Ab} \pm 0.051$
3	$0.666^{Ba} \pm 0.051$	$2.226^{Aa} \pm 0.100$

^{A,B}Same row with different letters are significantly different ($p \leq 0.05$).

^{a,b}Same column with different letters are significantly different ($p \leq 0.05$).

The result indicated that water vapor transfer generally occurs through the hydrophilic portion of the film and depends on the hydrophilic-hydrophobic ratio of the film components. Table 4 shows the WVP of the films. The film drying at temperature of 45°C has lower than the films drying at 55°C. A similar effect was observed by Kaya and Kaya (2000) reported that increasing WVP with the increasing temperature were observed in whey protein isolate edible films were dried using microwave drying or at room conditions, increase in WVP with increase temperature was observed for methyl cellulose and hydroxypropyl methylcellulose films (Park and Chinnan, 1995). The presence resulted of olive oil concentrations are increased caused an increase in WVP. The effect found in this research may be related to the hydrophobic property and the thickness of the films. Since the WVP were calculated using the average thickness of each film sample (Eq. 1). The thicknesses of edible films are increased with the drying temperature and olive oil concentration leading an increase in WVP.

3.5 Solubility

The water solubility of these films was decreased by adding olive oils concentrations (Table 5). This could be explained by the fact that vegetable oils, with the help of hydrophobic substances that dispersed in the films, changed the polarity of the components (Ekthamasut and Akesowan, 2001). In addition, the increase of drying temperature caused the decrease of the solubility in films. This was similar with the observed by Tápia-Blácido et al. (2005) the films based with amaranth flour found that the solubility in water was affected by temperature variation and by drying humidity.

Table 5 Results of solubility (%) of film obtained from different drying temperatures and olive oil concentrations.

Olive oil	Temperature (°C)	
(%w/w)	45	55
0	$78.16^{Aa} \pm 0.68$	$70.11^{Ba} \pm 2.39$
1	$56.02^{Ab} \pm 0.19$	$38.51^{Bb} \pm 0.07$
3	$24.45^{Bc} \pm 0.64$	$26.95^{Ac} \pm 0.37$

^{A,B}Same row with different letters are significantly different ($p < 0.05$).

^{a,b}Same column with different letters are significantly different ($p < 0.05$).

4 Conclusions

The effects of olive oil concentrations and drying temperatures on physical properties, mechanical properties, water vapor permeability and solubility of biodegradable film were studied. From the experimental results of properties appeared that the elongation, thickness and water vapor permeability were significantly increased with increase in the olive oil concentrations. The tensile strength and percent solubility of the films decreased with an increase in the concentrations of olive oil. The olive oil concentrations had statistically significant on all condition of edible films prepared by different temperatures drying. The tensile strength and percent solubility of the films decreased with an increase in the drying temperatures but the elongation, thickness and water vapor permeability were significantly increased with the drying temperatures increase.

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