Effects of Drying Temperatures and Glycerol Concentrations on Properties of Edible Film from Konjac Flour

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***Abstract*—The objective of this research was to study the effect of drying temperatures and glycerol concentrations on properties of edible film from konjac flour. Glycerol was used as plasticizer at the concentrations of 0 and 0.3% w/v for film forming. The drying process was done using heat pump dryer at various temperatures of 45, 50 and 55 °C. Sample was dried until the final moisture content was down to 1% wet basis. Then, film samples were taken to determine various properties including, tensile strength, elongation, water vapor permeability and solubility. The results found that konjac film without glycerol had a higher tensile strength than that konjac film with 0.3% glycerol. Also, tensile strength was significantly increased with the increasing of drying temperature ($p \leqslant 0.05$). For the elongation, water vapor permeability and solubility of film, these properties of konjac film with 0.3% glycerol were higher than that konjac film without glycerol. The elongation was significantly decreased when the drying temperature increased ($p \leqslant 0.05$). Moreover, water vapor permeability and solubility were significantly difference at various drying temperature ($p \leqslant 0.05$). The drying temperature at 50 °C provided the lowest water vapor permeability and the highest solubility**

***Index Terms*—edible film, konjac, drying, plasticizer, film properties**

I. Introduction

The wide use of petroleum-derived plastics and the negative impact of these on the environment prompted the search for biodegradable materials obtained from renewable resources. The use of agriculture derived biopolymers, such as proteins and glucomannan, appears as an interesting alternative to synthetic plastics for some applications, especially those with a short life-time, such as food packaging, and generates new uses of higher added value for agriculture products [1].

Edible films and coatings have long been used to protect food products, improving food quality. The application of edible films is a technological hurdle that can modify vegetable tissue metabolism while affecting respiration; they can be conveyors of antimicrobials, antioxidants and other preservatives; they can enrich product formulation, carrying vitamins and minerals [2]. In general, edible films and coatings provide the potential to control transport of moisture, oxygen, aroma, oil, and flavor compounds in food systems, depending on the nature of the edible film-forming materials [3]. The physical and mechanical properties of edible films are a subject of great importance due to their influence on product performance and consumer acceptance.

Konjac glucomannan (KGM) is the main renewable products which are economical, readily available, biodegradable and highly safe. They possess special nutritional and/or health-protective functions as well as good film-forming properties. 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 [4].

One important component of edible films is the plasticizer which is required to overcome film brittleness and improve its flexibility and extensibility [5]. Plasticizers are low molecular weight components added to films to reduce their brittleness, by increasing the space between polymer chains and, consequently, decreasing the intermolecular attractive forces and increasing the flexibility and extensibility of the material [6], [7]. An alternative to reduce the hygroscopicity of films is the utilization of hydrophobic plasticizers.

The effect of given drying conditions depends on various characteristics of the raw material, such as a pre-existing gel phase or the occurrence of thermal gelation during drying. Moreover, various phenomena, such as the transition from an amorphous to a vitreous phase, the appearance of phase separation (thermodynamic incompatibility) and crystallization, may occur. The interaction between the physicochemical nature of biopolymers and the drying conditions is very important [8].

Banker *et al*. [9] show that found the water vapor permeability of cellulose films increased as the film

Manuscript received August 31, 2013; revised November 2, 2013.

doi: 10.12720/jomb.3.3.171-174

thickness increased. Gontard *et al.* [10] reported that water vapor barrier properties and some mechanical properties of wheat gluten film were affected by plasticizing agents, but they did not measure O_2 and CO_2 permeability. Gennadios *et al.* [11] studied the effect of temperature on edible protein-based films (corn-zein, wheat gluten and wheat/soy films) but they concentrated only on oxygen permeability values.

The objective of this research was to study the physical and mechanical properties of konjac edible film containing glycerol as plasticizer with the influence of varied temperature drying (45, 50 and 55°C) on properties of the films drying under a heat pump dryer. The results of the study will contribute to understand the effect of film composition on food quality along shelf-life.

II. Materials and Methods

A. Film Preparation

Biodegradable films were prepared using a following process; this consists of blends konjac flour (1% w/v) in 100 ml distilled water with magnetic stirring at room temperature for 3 h. The KOH 0.5M (0.14%w/v) was added to the solution and stirring was continued for 15 min. The mixture was then left to stand for 1 h at room temperature. This is followed by addition of plasticizer (0.3% w/v glycerol) with constant stirring for 30 min.

B. Film Drying

After the solution was prepared, the mixture was then cooled to room temperature. The solution mixture (33.75 ml) was casted onto stainless plates (13 x 7 cm^2) and dried in a heat pump dryer at 45, 50 and 55°C until dried films could be released intact from plates. The dry films obtained were peeled off and stored in a desiccators containing saturated sodium chloride (NaCl) solution with 75% (RH) at room temperature until for further analysis. Control films were prepared in the same way without the addition of glycerol. Each film formulation was prepared in triplicates.

C. Tensile Strength and Elongation Tests

The measurement of the mechanical properties of konjac films was carried out using Texture Analyzer (model TA.XT.Plus). Films were cut into strips with a test dimension of 30 mm wide and 100 mm long strip. Initial grip separation and cross-head speed were set at 60 mm and 20 mm/min, respectively. Tensile strength was calculated by dividing the maximum load for breaking the film by its initial cross-sectional area. Percent elongation was determined by dividing the film elongation at rupture by the initial grip separation.

D. Water Vapor Ttransmission Rate

Water vapor transmission rate (WVTR) of the films was determined according to the modification method of Ekthamasut and Akesowan [12]. An aluminum cup with 5.2 cm diameter and 4.2 cm height was contained 100 g silica gel (0% RH) that was dried in oven at 120 ℃ for 24 hr. The headspace of the aluminum cup was 1 cm after silica gel adding. 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 desiccators containing Magnesium Nitrate (Mg $(NO_3)_2$ (50% RH). The aluminum cups were weighed at 24 h intervals for 8 days storage. Weight loss graphs were plotted against with the storage time. The slope of each line was calculated by linear regression ($r^2 \geqslant 0.99$). The measured WVTR of the films was determined as follows:

$$WVTR = (slope / A) \tag{1}$$

where WVTR is the water vapor transmission rate (g m-2 h-1) through a film, was calculated by linear regression from the points of weight gain and time, during constant rate period divided by the exposed film area (m2), WVTR measurements were replicated three times for each batch of films.

E. Solubility in Water

Solubility is defined as the percentage of film dry matter solubilized which was immersion in water. It was determined by the application method of Oh *et al*. [13] The dry matter of film (W) was determined by drying the 2 cm diameter disks in hot air oven at 105 ℃ for 3 h. Disks were cut, weighed and immersed in 100 ml of distilled water with stirring for 1 h at room temperature. The weight of filter paper was recorded (a1). After film solubility for 1 h, the remained films were filtration pass through the filter paper No.4 and dried by hot air oven at 105 ℃ for 25 min to determine the final weight of dry matter of film and filter paper (a2). The percentage of solubility in water of the films was determined as follows:

$$\%\ solubility\ in\ water = \frac{[W - (a_2 - a_2)]}{W} \tag{2}$$

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

III. Result and Discussion

The effects of drying temperature and plasticizer contents (0% and 0. 3% w/v) on physical properties, mechanical properties, water vapor permeability and solubility of edible konjac films was investigated. The experimental results are as following.

A. Tensile Strength and Elongation Properties

Tensile strength and elongation properties of the films tested with a difference of drying temperature and plasticizer concentration. The results are shown in Table I and Table II.

It was found that the glycerol concentration significantly affected the tensile strength and percent elongation of the films. In all cases, the films without glycerol had the highest tensile strength and lowest percent elongation. On the other hand, when glycerol was added, the films were more flexible.

In relation to the effect of the drying conditions on the mechanical properties, it was found that the increase of temperature drying caused increase of the tensile strength and decrease of elongation.

TABLE I. TENSILE STRENGTH TESTS

Glycerol (%)	Drying Temperature (℃) 45	50	55
0	23.08^{Ab}±3.56	23.58^{Ab}±3.77	25.64^{Aa}±4.6
0.3	13.15^{Ba}±2.94	13.90^{Ba}±1.91	11.61^{Bb}±1.12

Unit of tensile strength in Mpa
A, B. Means in same column with different letters are significantly different ($p \leqslant 0.05$).
a, b. Means in same row with different letters are significantly different ($p \leqslant 0.05$).

TABLE II. ELONGATION TESTS

Glycerol (%)	Drying Temperature (℃) 45	50	55
0	3.57^{Ba}±1.30	2.63^{Bb}±0.64	2.69^{Bb}±0.56
0.3	20.61^{Aa}±3.75	13.13^{Aa}±3.98	7.05^{Ab}±0.49

Unit of elongation in percentage
A, B. Means in same column with different letters are significantly different ($p \leqslant 0.05$).
a, b. Means in same row with different letters are significantly different ($p \leqslant 0.05$).

B. Water Vapor Transmission Rate

The WVP of Konjac flour films prepared without plasticizer is lower than that of glycerol containing films (Tables III). The better water vapor barrier properties of edible films containing 0.3% glycerol plasticizer compared with those of the films without glycerol might be due to the fact that glycerol is hygroscopic [14].

TABLE III. WATER VAPOR TRANSMISSION RATE

Glycerol (%)	Drying Temperature (℃) 45	50	55
0	2.75^{Ba}±0.62	1.38^{Bb}±0.15	2.55^{Ba}±0.50
0.3	6.26^{Aa}±0.46	4.34^{Ab}±0.87	5.36^{Aa}±0.04

Unit of water vapor transmission rate in (g/m^2h)
A, B. Means in same column with different letters are significantly different ($p \leqslant 0.05$).
a, b. Means in same row with different letters are significantly different ($p \leqslant 0.05$).

C. Water Solubility

For the film solubility, it was observed that (Equation 2), The films kept their physical integrity, In general, it was observed (Table IV) that the variation on the drying conditions caused modifications in the films solubility, Which is an indicative of low solubility at without plasticizer which varied between 15.77 and 23.01%, independently of the drying conditions and high solubility at 0.3 glycerol which varied between 51.01 and 65.26%. However, it was not possible to establish a correlation between drying temperature and glycerol concentrations. Pérez-Mateos *et al*. [15] determined 88% of solubility of films based on fish-skin gelatin and sunflower oil plasticized with sorbitol and glycerol. Tápia-Blácido *et al*. [8] working with films based in amaranth flour, verified that the solubility in water was affected by temperature variation and by drying humidity. Gómez *et al*. [16] working with films based on bovine- and fish skin gelatin plasticized with blends of glycerol and sorbitol, determined solubility equal to 34.3 and 39.9%, respectively.

TABLE IV. WATER SOLUBILITY

Glycerol (%)	Drying Temperature (℃) 45	50	55
0	17.36^{Bb}±2.66	23.01^{Bc}±2.20	15.77^{Ba}±1.29
0.3	62.81^{Aa}±1.39	65.26^{Aa}±1.90	51.01^{Ab}±6.37

Unit of water solubility in percentage
A, B. Means in same column with different letters are significantly different ($p \leqslant 0.05$).
a, b. Means in same row with different letters are significantly different ($p \leqslant 0.05$).

IV. CONCLUSIONS

The effect of drying temperatures at 45, 50 and 55 ℃ and glycerol concentrations at 0 and 0.3% w/v on properties of edible film from konjac flour was studied. The experimental results can be drawn as the following:

1) The drying curves of the film were found that the time needed to equilibrium moisture content increased with the glycerol concentration.
2) The tensile strength of the films decreased with an increase with the glycerol concentration and while the drying temperature at 45 and 50 ℃ at same plasticizer were not statistically significant *($p \leqslant 0.05$).*
3) The elongation, percent solubility and water vapor permeability were significantly increased with the glycerol concentration *($p \leqslant 0.05$).*
4) The percent solubility at 45 and 50 ℃ at same plasticizer were not statistically significant *($p \leqslant 0.05$).*

ACKNOWLEDGMENT

The authors would like to express their sincere thanks to Faculty of Engineering, Mahasarakham University and the National Research Council of Thailand (NRCT) for financial support. Also, we wish to thank Mahasarakham University Development fund for conference participation

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