



Empirical modeling of moisture sorption characteristics and mechanical and barrier properties of cassava flour film and their relation to plasticizing–antiplasticizing effects

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ABSTRACT

Moisture sorption kinetics of cassava flour film was investigated at various relative humidities (RH). Their curves were fitted to a Peleg model. This film was also studied for its sorption isotherm. Empirical models were tested to fit the experimental data. Moisture sorption was more rapid in the initial stages; less moisture was adsorbed as adsorption time increased. GAB and Lewicki models were found to be better-fitted models. Effects of sorbitol concentration and RH on mechanical properties of films were examined. The tensile strength (TS) of films vastly decreased with increasing sorbitol concentration. The percentage of elongation increased significantly with increasing sorbitol content; however, it declined when sorbitol content was above 40 g/100 g. Modulus of elasticity (*E*) of films displayed a similar trend to TS. The *E* of this film was inversely linear to RH, whereas its elongation was exponentially proportional to RH. Effects of sorbitol concentration on barrier properties of films were also examined. The water vapor transmission rate of film was gradually reduced with increasing sorbitol content; however, it dropped dramatically with sorbitol content above 40 g/100 g. The oxygen transmission rate of film significantly decreased with rising sorbitol content. This is helpful in understanding the performance of edible films under varying RH conditions and plasticizer contents.

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1. Introduction

Over the last four decades, extensive research has been undertaken on the use of edible films and coatings. They have been used as moisture, gas or solute barriers in food packaging to prolong shelf-life and improve overall food quality. Potential sources and applications of edible films and coatings have been reviewed by Debeaufort, Quezada-Gallo, and Voilley (1998) and Tharanathan (2003). Owing to the hydrophilic properties of starch and flour, they provide a minimal barrier to moisture (Kester & Fennema, 1986). Nevertheless, these films possess good barrier properties to oxygen, carbon dioxide and lipids, and protect against lipid oxidation (Banker, 1966).

Cassava (*Manihot esculenta* Crantz) or tapioca is one of the most economically important crops in Thailand, Brazil and other tropical countries. It is an abundant and cheap agricultural source of starch and flour production. In Thailand, cassava flour consists mostly of starch (94–97.5 g/100 g dry basis), with a small amount of protein

(0.3 g/100 g); moisture content typically ranges 13–14 g/100 g. An amylose content of approximately 17 g/100 g is responsible for the strong film-formation characteristics of cassava flour film (Bangyekan, Aht-Ong, & Srikulkit, 2006).

Sorption characteristics of cassava flour film are crucial for the design, modeling and optimization of its drying, storage and transport. Sorption isotherms provide information on the moisture-binding capacity of products at a determined relative humidity, and are a useful means of analyzing the moisture plasticizing effect and the effect on mechanical properties (Al-Muhtaseb, McMinn, & Magee, 2002; Bell & Labuza, 2000). Chirife & Iglesias (1978) reviewed 23 isotherm models and their use for fitting sorption isotherms of foods and food products.

The phenomena of plasticization and antiplasticization have begun to receive increasing attention in the last decade from food scientists and technologists (Chang, Karim, & Seow, 2006). Low-molecular-mass compounds or diluents, acting as external plasticizers, are an integral part of polymeric systems in which they serve to enhance the flexibility and workability of the otherwise rigid neat polymers. Nevertheless, they may serve as mechanical

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antiplasticizers when present at low concentrations, leading instead to the polymer-diluent blends becoming stiffer than the neat polymer (Sears & Darby, 1982).

Plasticizers are normally added to a polymeric matrix in order to overcome the film brittleness (Lieberman & Gilbert, 1973). The properties of cassava films have been extensively studied (Alves, Mali, Beléia, & Grossmann, 2007; Chang et al., 2006; Famá, Goyanes, & Gerschenson, 2007; Mali, Sakanaka, Yamashita, & Grossmann, 2005; Müller, Yamashita, & Laurindo, 2008; Paes, Yakimets, & Mitchell, 2008; Parra, Tadini, Ponce, & Lugão, 2004; Souza et al., 2012; The, Debeaufort, Voilley, & Luu, 2009). However, there is still a lack of comprehensive analysis of sorption kinetics and information on the plasticizing–antiplasticizing effects on the properties of cassava films. As exposed to the environmental atmosphere, the moisture content of the film will not equilibrate, resulting in a ceaseless alteration of tensile properties of the films via moisture sorption (Cho & Rhee, 2002). In addition, plasticizer concentrations significantly affect both water transmission and oxygen transmission rates of edible films. Consequently, the changes in mechanical and barrier properties of a film are affected by the rate of moisture adsorption of films and the concentration of plasticizers, respectively.

The objective of this study is to determine the moisture sorption characteristics of cassava flour film and the plasticizing–antiplasticizing effects on mechanical and barrier properties of cassava flour films.

2. Materials and methods

2.1. Materials

Cassava flour was purchased from E.T.C. International Trading Co., Ltd. (Bangkok, Thailand). Glycerol, sorbitol and polyethylene glycol with $M_w = 400$ (PEG-400) were used as plasticizers, and were obtained from Sigma Chemical Co. (St. Louis, MO). Lithium chloride (LiCl), magnesium chloride ($MgCl_2$), magnesium nitrate ($Mg(NO_3)_2$), potassium iodide (KI), sodium chloride (NaCl), potassium chloride (KCl) and potassium nitrate (KNO_3) were also purchased from Sigma Chemical Co.

2.2. Preparation of cassava flour film

Cassava flour film solution was prepared by dissolving 5.0 g of cassava flour in 100 g of aqueous solution, and then heating at its gelatinization temperature of 70 °C while stirring until a viscous and transparent solution was observed. Weights equivalent to 20, 30, 40 and 50 g/100 g glycerol, sorbitol or PEG-400 were then added as film plasticizers, to prevent brittleness of the film. The resulting solutions were degassed in an ultrasonic water bath (Model 275D; Crest Ultrasonics Corp., Trenton, NJ, USA) for 10 min. Cassava flour film was cast by pouring film-forming solution (50 mL) onto a flat 21 × 29 cm glass plate, which previously wrapped with a linear low-density polyethylene film. The plates were dried at 60 °C in a ventilated oven for 3 h.

2.3. Moisture sorption kinetics and isotherm

A standard gravimetric methodology (weighing samples equilibrated in thermally stabilized desiccators) was used for determination of the adsorption kinetics. Film specimens (25 × 25 mm) were completely dried in a vacuum oven at 70 °C and 76 mmHg for 48 h, and then in a desiccator over P_2O_5 for 2 weeks. The dried samples (in triplicate) were placed into desiccators with saturated salt solutions at 30 °C. The salt solutions included LiCl, $MgCl_2$, $Mg(NO_3)_2$, NaCl and KNO_3 of known RH: 11.3, 32.4, 51.4, 75.7 and 92.5%, respectively (Greenspan, 1977). At the conditions of relative

humidity higher than 60%, a trace of ethanol was used as a fungistatic agent. Weights of samples as a function of time were measured; moisture content was then measured by drying in an oven at 105 °C for 3 h (Lane, 1998). A set of experiments was performed in two replications. The results were expressed on a dry-weight basis as g/100 g dry sample. Water activity (a_w) was determined using a Testo 650 water activity instrument (Testo, Lenzkirch, Germany). Moisture adsorption curves of the samples were fitted to a mathematical model suggested by Peleg (1988):

$$M_t = M_0 + [t/(k_1 + k_2t)] \quad (1)$$

where M_t is the moisture after time t ; M_0 is the initial moisture; and k_1 and k_2 are constants. If M_t and M_0 are given in g/100 g on a dry basis, and the time units are in hours (h), then the units of k_1 and k_2 will be h per g/100 g (dry basis), and the reciprocal of g/100 g (dry basis), respectively. According to this model, the equilibrium moisture (M_E) is given by:

$$M_E = M_0 + 1/k_2 \quad (2)$$

Similarly, the momentary sorption rate dM_t/dt is given by:

$$dM_t/dt = k_1/(k_1 + k_2t)^2 \quad (3)$$

and the initial rate is given by $1/k_1$. One of the characteristics of Eq. (1) is that it can be transformed into a linear relationship in the form:

$$t/[M_t - M_0] = k_1 + k_2t \quad (4)$$

The latter offers a simple way to test the applicability of the model to sorption data, and to calculate its parameters by linear regression.

With respect to the test duration, this can be formulated as follows. Let R be the ratio:

$$R = [M_t - M_0]/[M_E - M_0] \quad (5)$$

It follows from the mathematical structure of Eq. (1) that if, for example, $t_{1/2}$ is the time needed to reach $[M_E - M_0]/2$, i.e. half the added moisture to reach equilibrium, then the time to reach any given level of R , namely t_R , is given by:

$$t_R/t_{1/2} = R/(1 - R) \quad (6)$$

A standard gravimetric methodology was used for determination of the adsorption isotherms. The film specimens were prepared and conditioned as described in Section 2.3. The dried samples in triplicate were equilibrated over saturated salt solutions inside desiccators at 30 °C for 4 weeks. The salt solutions included LiCl, $MgCl_2$, $Mg(NO_3)_2$, KI, NaCl, KCl and KNO_3 of known RH: 11.3, 32.4, 51.4, 67.9, 75.7, 83.6 and 92.5%, respectively (Greenspan, 1977). Moisture content was then measured after drying in an oven at 105 °C for 3 h (Lane, 1998). A set of experiments was performed in four replications. The results were expressed on a dry-weight basis as g/100 g dry sample. Water activity was determined using a water activity instrument.

2.4. Moisture sorption isotherm modeling

Isotherm models from the literature (Berg & Bruin, 1981; Toledo, 1991) were selected for modeling the experimental data of adsorption isotherms of cassava-flour-based baked samples. Those models are expressed and rearranged as given below.

GAB (Guggenheim–Anderson–de Boer) model (Berg & Bruin, 1981):

$$m_e = m_o C k a_w / (1 - a_w) [1 + (C - 1) k a_w] \quad (7)$$

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