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Dynamic mechanical properties of flaxseed gum based edible films

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ABSTRACT

The flaxseed gum based edible film was prepared and investigated for dynamic mechanical properties and water vapor transmission rate in this study. The glass transition temperature of the films, indicated by the peak of tan δ, was significantly decreased by the increasing of the glycerol concentration. The storage modulus and the temperature at which it reached a maximum value were decreased by the increasing of the glycerol concentration. The glass transition temperature of the flaxseed gum based edible films was also increased by the increasing of the flaxseed gum concentration. The increasing of the glycerol and flaxseed gum concentration could lead to the increase of water vapor transmission rate of the flaxseed gum films.

1. Introduction

Edible film, which is environmentally friendly and renewable, could be widely used in food packaging to reduce the usage of plastic material. It would thus help to reduce the carbon emission to the air. Edible films can be used to inhibit the migration of moisture, oxygen, carbon dioxide, aromas, and lipids in order to extent the shelf life of food (Ghanbarzadeh & Oromiehi, 2009; Mikkonen et al., 2007). Furthermore, some edible films would not become waste after usage because they could be simply eaten together with the food. Starch has been used to prepare edible films for a long time in food industry (Famá, Flores, Gerschenson, & Goyanes, 2006). Although there are low cost, renewable and biodegradability advantages of using starch-based films, they present low mechanical resistance and high moisture sensitivity when compared to traditional petroleum plastic films. Thus, polysaccharides such as chitosan, pectin, guar gum, carrageenan gum, and galactomannan have been studied as potential materials for edible or biodegradable films to achieve better mechanical properties (Chillo et al., 2008; Giancone et al., 2008; Huang, Yu, & Xiao, 2006; Mikkonen et al., 2007).

Many investigations have been carried out for the preparation and property modification of edible films. The dynamic mechanical properties and water resistance properties are among the most important properties of edible films (Chillo et al., 2008). The mechanic property of edible films is of vital importance, because it determines the usage fields of the films and how much they could replace the plastic films. Dynamic mechanical analysis has been widely used in the determination of the material properties. It could supply useful information about the mechanical nature of the material, such as storage modulus, loss modulus, and the glass transition temperature (Li, Li, Wang, Özkan, & Mao, 2010). The mechanical properties of edible films above the glass transition temperature (T_g) could experience a significant change, thus influencing the use of these materials (Zhou et al., 2009). For example, it is expected that the permeation of gas and vapor molecules through a film will be higher above T_g, where polymer chains are more mobile (Mathew & Abraham, 2008). Several studies have been done using the dynamic mechanical analysis on edible films, such as whey protein isolate films, guar gum films, and galactoglucomannan films (Huang et al., 2006; Mikkonen et al., 2008; Zinoviadou, Koutsoumanis, & Biliaderis, 2010).

Polysaccharide-based films are commonly plasticized to overcome film brittleness and to avoid the cracking of films during subsequent handling and storage (Hernandez-Izquierdo & Krochta, 2008). Glycerol is the most commonly used plasticizer for edible films because of its stability and compatibility with hydrophilic biopolymeric packaging chain (Cervera et al., 2004). The plasticizers could increase free volume and thereby allow increased backbone chain segmental mobility (Gontard & Ring, 1996).

Flaxseed gum is natural carbohydrate polysaccharide extracted from flaxseed (*Linum usitatissimum*), which makes up about 8% of the seed weight (Mazza & Biliaderis, 1989). It has been shown

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that the flaxseed gum has a good potential to be used in the food industry as thickener, emulsifier, stabilizer, etc. (Chen, Xu, & Wang, 2006a; Wang et al., 2008; Wang, Wang, Li, Xue, & Mao, 2009; Wang, Li, Wang, Li, & Adhikari, 2010). Flaxseed gum had been used for the preparation of edible films by Chen, Xu, and Wang (2006b). It has been found that the tensile strength of flaxseed gum film was increased with the increasing of flaxseed gum concentration, but it was decreased with the increasing of plasticizer concentration. Flaxseed gum has also been used to strengthen the starch films to achieve better mechanical performance (Li, Wang, & Li, 2009).

To the best of our knowledge, no study has been done on the dynamic mechanical properties of flaxseed gum films. Thus, the effects of plasticizer (glycerol) and the flaxseed gum concentration on the dynamic mechanical properties and water vapor transmission rate of flaxseed gum based edible films were investigated in this study. This information would help both to understand the properties of flaxseed gum films and to figure out the key factors in preparing the polysaccharide based edible films.

2. Materials and methods

2.1. Flaxseed gum extraction

Flaxseed with a moisture content of 6.50% was purchased from the Hebei province of China. Glycerol of analytical grade was used as a plasticizer.

Flaxseed (100 g) was washed in water for 1 min to remove the surface dust, and then mixed with 900 mL deionized water. The flaxseed and water mixture were then stirred for 5 h at a speed of 300 rpm in a 60 °C water bath, according to the method of Cui (2001). The extracted flaxseed gum solution was filtered through 40-mesh screen. After that the extracted flaxseed gum solution was precipitated with two volumes of 95% ethanol, collected by centrifugation at 3000 rpm for 10 min using an LG10-2.4 A machine (Beijing Medical Centrifuge Corporation, Beijing, China), according to the method of Cui, Mazza, and Biliaderis (1994) with some modifications on drying method. The precipitated flaxseed gum was then dried in a hot air oven at 80 °C for 4 h.

The protein content of flaxseed gum extracted by the above method was 14.4 ± 0.2% as determined using Kjeldahl method (FOSS Kjeltec 2300 analyzer, FOSS Co., Höganäs Sweden). The nitrogen data were converted into protein values using a conversion factor of 6.25. Analysis was performed in triplicate.

2.2. Film preparation

The flaxseed gum powder obtained via above method was firstly dissolved in deionized water (pH 6.4–7.0) using a magnetic stirrer for 30 min at 25 °C to prepare the 1–2% (dry base, w/w) flaxseed gum solutions. In order to study the effects of the flaxseed gum concentration, 30% (dry base, w/w) glycerol was added as the plasticizer to 100 mL flaxseed gum solutions with various concentrations. In order to study the effects of the glycerol concentration, different amounts of glycerol was added to 100 mL 1.5% flaxseed gum. Then the mixtures were stirred to reach uniform distribution. After that, the mixtures was spread evenly onto watch glass with 15 cm diameter, and dried at 80 °C for 4 h with air blow in the oven. Finally, the dried film was carefully removed from the watch glass and stored for the further testing.

2.3. Dynamic mechanical analysis measurements

The dynamic mechanical measurements of the flaxseed gum films were carried out with DMA Q800 (TA instrument, Crawley, USA). In all the tests, to match the size of DMA film tension clamp, the samples with approximate dimensions of 5.0 mm × 30.0 mm × 0.1 mm were cut from the flaxseed gum films. The dimensions of each rectangle sample were measured by an electronic digital caliper (PRO-MAX, Fowler, USA) and input in the DMA procedure.

For all DMA measurements, a 1 mN preload force was applied to ensure that the sample material was fully elongated (had no slack). The films were tested over temperature range from 35 °C to 200 °C with a constant heating rate of 5 °C/min. The storage modulus (G'), loss modulus (G''), and tan loss angle ($\tan\delta$) were recorded during the temperature sweep test. The temperature at which G' and loss angle reach its peak was found by the software (Universal Analysis 2000, TA instrument, Crawley, USA).

2.4. Water vapor transmission

The water vapor transmission rate (WVTR) of flaxseed gum films was gravimetrically determined at 25 °C according to the modified ASTM Standard Test Method E 96-80 (ASTM, 1989) and Li et al. (2009). Films were sealed on beakers which had an internal diameter of 24 mm and an exposed area of 4.52 cm^2. The depth of beakers was 3.5 cm. Test cups were placed in a desiccator cabinet, in which 4A molecular sieve was used. The weight of the beaker was recorded every 24 h to evaluate stationary-state water vapor transmission.

WVTR was calculated according to Eq. (1):

$$\mathrm{WVTR} = \frac{w \times x}{A \times t} \quad (1)$$

where WVTR is water vapor transmission rate ($mg\,mm\,day^{-1}\,cm^{-2}$), w is the weight loss of the sample (mg), t is the treatment time (day), x is the thickness of the film (mm) and A is the permeation area (4.52 cm^2). Weight loss of the beaker was plotted over time. The WVTR was calculated from the slope (k), which was the term x/t of a linear regression of weight loss versus time. After 24 h a stationary WVTR was attained and, from that moment on, the weight changes (to the nearest 0.1 mg) were recorded daily over a 6 day period. The tests were carried out in duplicate.

2.5. Statistical analysis

All the experiments were carried out in triplicate in this study. The results reported are means ± standard deviation of the triplicate data. Duncan's multiple comparison tests were carried out to determine the significance when necessary at $p < 0.05$ using the SPSS 13.0 (SPSS Inc., Chicago, USA).

3. Results and discussion

3.1. Dynamic mechanical properties affected by glycerol concentration

The effect of glycerol concentration on the temperature response of tan δ of flaxseed gum film is shown in Fig. 1. The sample with 10% glycerol concentration and sample with 1% flaxseed gum concentration were both too fragile to complete all the tests. Thus the data of those two samples were not shown in this paper. The tan δ of the flaxseed gum films first experienced a high peak between 70 °C and 85 °C, and then exhibited low peak between 100 °C and 150 °C. Some of the high peaks are hard to see in figure, so the peak temperatures were picked from raw data and marked in Fig. 1. Following the low peak, the tan δ began to increase dramatically. This is caused by both the high peak of the storage modulus and the low peak of the loss modulus (data not shown). Dynamic mechanical properties of polymers can sensitively represent their molecular motion, which has a close relationship with the con-

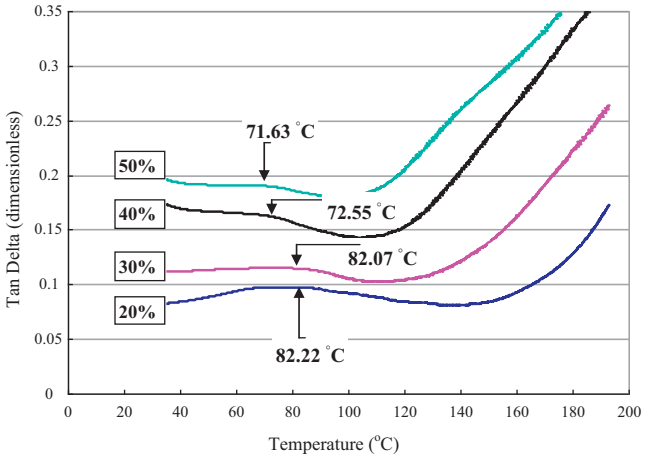

Fig. 1. Effects of glycerol concentration on the temperature response of tan δ of flaxseed gum films.

densed and chain structures of polymers. When a glassy to rubbery state transition occurred on each specific moving unit, a modulus slope or a damping peak would appear in DMA spectra (Zhou et al., 2009). The high peak of tan δ is often taken as the glass transition temperature (T_g) of macromolecule materials (Sperling, 2001). The glass transition means the temperature at which the transformation from the glass state to rubbery state takes place (Kasapis, Al-Marhoobi, & Mitchell, 2003). As shown in Fig. 1, the high peak temperatures, i.e. the T_g of the flaxseed films, are marked with arrowheads. The increase of glycerol concentration leaded to the decrease of T_g, while the tan δ values were increased by the increase of glycerol concentration. This is probably due to the plasticizing effects of glycerol on the flaxseed gum films, which could provide the films with higher thermal uniformity and molecule mobility. Thus the glass transition could be finished at lower temperature (Gontard & Ring, 1996). Similarly, the glass transition temperature of chitosan films was found to be decreased for about 20 °C while the glycerol concentration increased from 0% to 125% (Thakhiew, Devahastin, & Soponronnarit, 2010). A decrease of T_g also happened when the glycerol concentration increased from 15% to 40% in tapioca starch/decolorized hsian-tsao leaf gum films (Chen & Lai, 2008).

The storage modulus of flaxseed gum films with various glycerol concentrations during heating process is shown in Fig. 2. The storage modulus of all the films experienced a significant transition between 100 °C and 135 °C, after that the storage modulus began to decrease with the increasing temperature. Storage modulus is the symbol of mechanical strength of material, while the peak storage modulus value means the films reach the maximum mechanical properties (Huang et al., 2006). The storage modulus values of the films also decreased with the increasing glycerol concentration. This is because the glycerol as a plasticizer could soften the texture of the flaxseed gum films, which thus leads to the lower storage modulus. The temperatures at which storage modulus reached its peak were marked using arrow head in Fig. 2. These peak temperatures of the storage modulus also decreased with the increasing of glycerol concentration. Higher glycerol concentration in flaxseed gum films and mix polysaccharide films (made from guar gum and locust bean gum) has already been found to increase the elasticity by increasing the elongation at break, but decrease the tensile strength at the same time (Chen et al., 2006b; Mikkonen et al., 2007). The tensile strength of gelatin films and tapioca starch/decolorized hsian-tsao leaf gum films were also found to be decreased by the increasing plasticizer concentration (Chen & Lai, 2008; Thomazine, Carvalho, & Sobral, 2005).

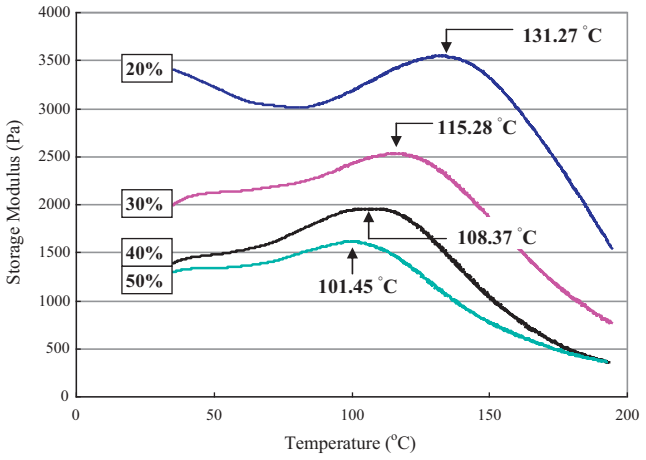

Fig. 2. Effects of glycerol concentration on the temperature response of storage modulus of flaxseed gum films.

The detailed dynamic mechanical properties of flaxseed gum films at the point when storage modulus reached its peak are illustrated in Fig. 3. It could be found that the storage modulus significantly decreased with the increase of glycerol concentration. Mechanical strength of edible chitosan films was also significantly decreased indicated by the decreasing tensile strength along with the increase of glycerol concentration (25–125%) (Thakhiew et al., 2010). This also happened in pullulan films with water as plasticizer (Lazaridou, Biliaderis, & Kontogiorgos, 2003). The loss modulus of the flaxseed gum films were not significantly affected by the glycerol concentration. A possible reason is that the glycerol is a Newtonian fluid with relatively low viscosity, which then did not change the viscosity of the mixture too much. The tan δ value was increased by the increasing glycerol concentration, which was mainly caused by the decrease of storage modulus.

3.2. Dynamic mechanical properties affected by gum concentration

The tan δ of flaxseed gum films during heating process as affected by the flaxseed gum concentration is shown in Fig. 4. The tan δ of flaxseed gum films first experienced a peak around 70–90 °C, which was recognized as the glass transition in this study, then decreased to the lowest value. After that the tan δ increased with the increasing of temperature. In general, the T_g of flaxseed gum films increased along with the increase of flaxseed gum concentration, although the difference between 1.5% and 1.75% sample was not significant. Higher T_g might be the evidence of high level cross-linked structure, since the corn starch T_g has been found to be increased by higher cross-link levels (Chung, Woo, & Lim, 2004). The film with higher flaxseed gum concentration was thicker than the low concentration ones since the liquid volume was constant when preparing the films. Although the thickness has been measured and input into DMA to eliminate the effects of sample shape on the mechanical properties, the high concentration film may require more time to totally equilibrate during the glass transition than the low concentration one. And this delay of equilibration might lead to the increase of T_g. The increase of gum concentration was also found to slightly raise the glass transition temperature of tapioca starch/decolorized hsian-tsao leaf gum films at 15% or 25% glycerol concentration (Chen & Lai, 2008).

The storage modulus of flaxseed gum films with various flaxseed gum concentrations as a function of temperature is shown in Fig. 5. The storage modulus of all the samples shows similar trend in the manner that the storage modulus was first increased then decreased with the increasing of the temperature. The peak value

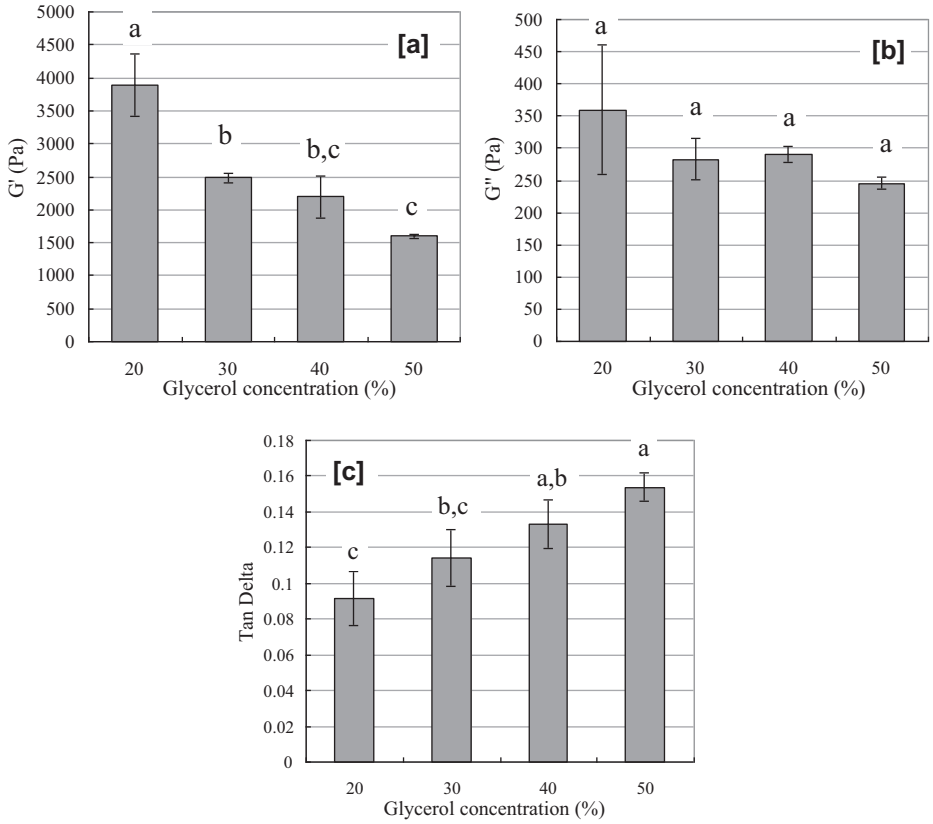

Fig. 3. Effects of glycerol concentration on the storage modulus, loss modulus, and loss angle of flaxseed gum films at the temperature of peak storage modulus ([a] for storage modulus; [b] for loss modulus; and [c] for tan loss angle) (values of each bar with different labels were significantly different ($p < 0.05$)).

of storage modulus was observed between 110 °C and 130 °C. Only the 2% flaxseed gum film shows significant higher value in storage modulus, while no significant difference was found among other samples ($p > 0.05$). After reaching a peak value, the storage modulus values were decreased with the increasing of temperature, which also contributed to the increase of tan δ as shown in Fig. 4.

The temperature at which storage modulus reached its peak was gathered and shown in Fig. 6. The storage modulus and loss modulus shows similar trend. It could be found that both the storage modulus and the loss modulus firstly decreased with the increasing of gum concentration. But the effect of gum concentration became insignificant when the concentration was higher than 1.5%. Only with the 1.25% gum sample shows significant higher modulus values than other samples ($p < 0.05$). The tan δ shows no significant difference among all the samples.

3.3. Water transmission rate of flaxseed gum films

The effects of glycerol concentration and flaxseed gum concentration on WVTR of flaxseed gum films are shown in Fig. 7. Both glycerol concentration and flaxseed gum concentration increase could significantly increase the WVTR of the flaxseed gum films. The reason that glycerol could increase WVTR is because it could make the film easier to absorb water vapor because of the plas-

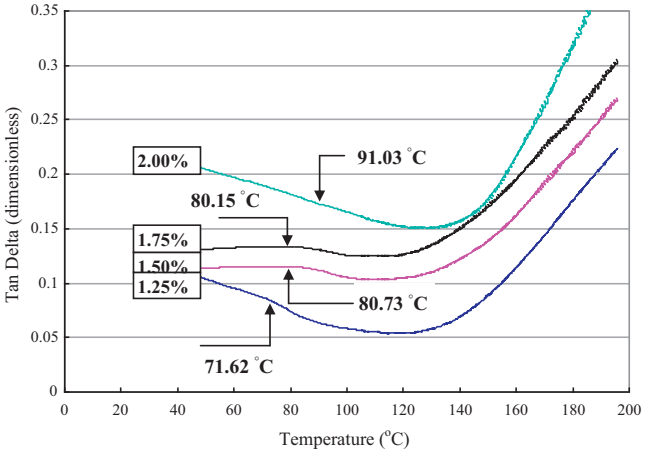

Fig. 4. Effects of flaxseed gum concentration on the temperature response of tan δ of the flaxseed gum films.

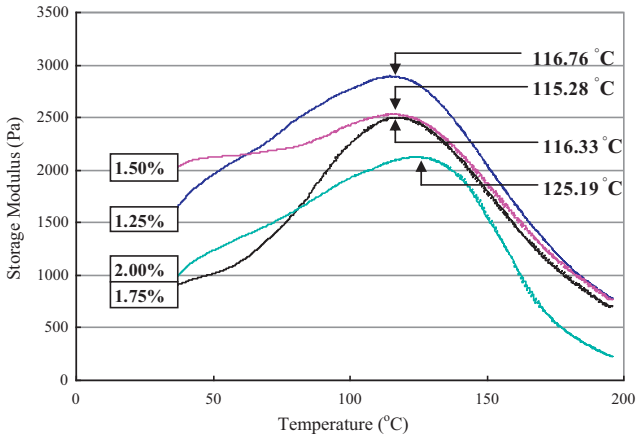

Fig. 5. Effects of flaxseed gum concentration on the temperature response of storage modulus of the flaxseed gum films.

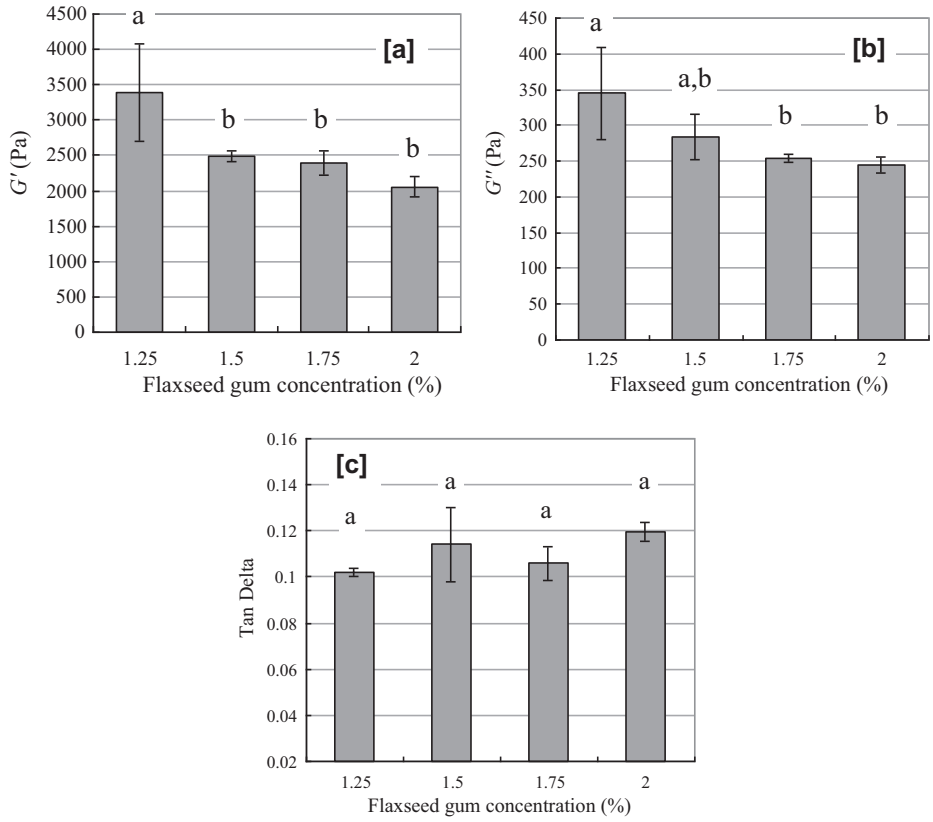

Fig. 6. Effects of flaxseed gum concentration on the storage modulus, loss modulus, and loss angle of flaxseed gum films at the transition temperature ([a] for storage modulus; [b] for loss modulus; and [c] for tan loss angle) (values of each bar with different labels were significantly different ($p < 0.05$)).

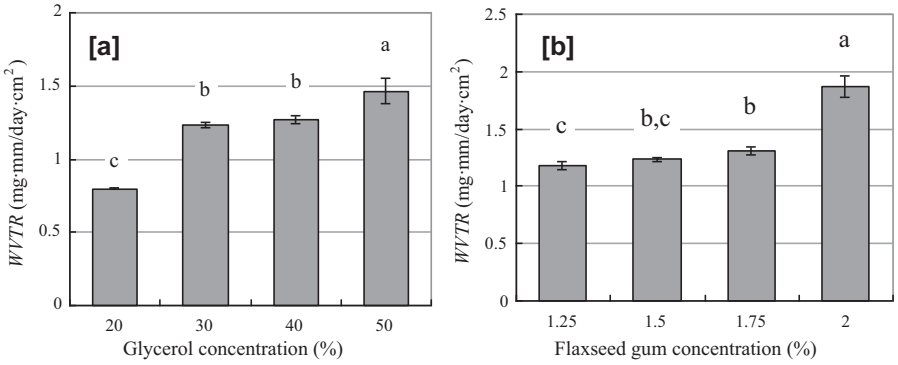

Fig. 7. Effects of composition on the water transmission properties of flaxseed gum films ([a] for effect of glycerol concentration and [b] for effect of gum concentration) (values of each bar with different labels were significantly different ($p < 0.05$)).

ticizing effect of glycerol. It has been reported that increase of the glycerol concentration in gelatin film could also lead to higher water vapor permeability due to the plasticizing effect of glycerol (Thomazine et al., 2005). The increase of flaxseed gum concentration could increase the WVTR is probably because it increased the thickness of the films but did not affect the vapor transmission. Thus the WVTR increased based on the calculation of definition. This result was consistent with that of chitosan films, in which the water transmission was also not affected much but the thickness increased when the chitosan concentration increased from 4 g/m^2 to 24 g/m^2 (Yoshida, Oliveira, & Franco, 2009). Besides, the viscoelastic properties of the films were decreased by the increase of flaxseed gum concentration (see Fig. 6). The lower values of modulus of the film could also contribute to the increase of the WVTR.

4. Conclusion

The dynamic mechanical properties of flaxseed gum films were investigated together with the water transmission rate in this study. The glass transition temperature of flaxseed gum was decreased by the increasing of glycerol concentration, due to the plasticizing effects of glycerol. Both the value of storage modulus and the temperature at which it reached its peak were decreased by the increasing of glycerol concentration, indicating that the glycerol had significant softening effects on the flaxseed gum films. The glass transition temperature of flaxseed gum was also increased by the increasing of flaxseed gum concentration, while only the 2% flaxseed gum concentration sample showed higher peak storage modulus temperature than the others. But the storage modulus value was decreased by the increasing of flaxseed gum concen-

tration. The water vapor transmission rate of flaxseed gums was significantly increased by both the glycerol and the flaxseed gum concentrations.

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