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Effect of sucrose on dynamic mechanical characteristics of maize and potato starch films

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ABSTRACT

The dynamic mechanical properties of prepared maize and potato starch films were evaluated for mixtures containing 0%, 10% and 15% (w/w) of sucrose at temperatures ranging from 40.0 to 140.0 °C. The spectra of storage modulus (G'), loss modulus (G''), and loss factor ($\tan\delta$) of starch films were acquired. Remarkable reduction in the glass transition temperature of maize and potato starch films was observed with the increasing sucrose content. The spectra of storage modulus (G'), loss modulus (G''), and loss factor ($\tan\delta$) were measured for the second and third time after two and seven days, respectively. The peaks of loss factor ($\tan\delta$) appeared at 59.81 ± 1.86 °C and 95.96 ± 1.67 °C after two-day-storage, but only one peak appeared at 85.46 ± 5.50 °C after seven days. A shifting trend from higher to lower temperature for loss factor was observed after seven days.

1. Introduction

Edible films have wide application in industry for their specified thermal and mechanical properties. Starch is an ideal source for producing both edible and biodegradable films. Starch is a major food component and a complex biodegradable carbohydrate made up of thousands of glucose units. Potato cultivars and maize have high starch content (Hermansson & Svegmark, 1996). Gelatinization and retrogradation are two main procedures of starch film formation. The performance of film formation is highly related to its granular structure and the proportion of amylose and amylopectin contents in starch. These properties have significant influence on starch gelatinization as well as starch film formation. Recent studies indicate the significance of temperature on starch formation and on mechanical properties. The thermal properties of starch changed as gelatinization and retrogradation happened under certain temperature regions (Wang et al., 2008). While the mechanical properties, especially the dynamic viscoelasticity, are a good implication of internal structural change at a macro scale, it is necessary to measure the properties of starch film through dynamic mechanical testing under continuous temperature change. Fredriksson, Silverio, Andersson, Eliasson, and Aman (1998) reported that starch from potato cultivars exhibited the highest retrogradation enthalpies and the cereal starches exhibited the lowest; the DP_w (the weight-average degree of polymerization) had a positive correlation to the gelatinization and retrogradation enthalpies.

Polymers are a series of viscoelastic materials. The dynamic mechanical method is a good example of introducing the measurement of the physical properties of industrial polymers into the research field of food polymers. Dynamic mechanical analysis (DMA) is a modern technique that can trace the macro-behaviors of materials caused by changes or transformations inside the microstructure of materials. It measures the changes in displacement or the material response (e.g. strain) under certain applied force (e.g. stress) as a function of time, temperature and frequency. It provides important information like hysteresis loss or damping which can describe mechanical behaviors of polymers in dynamic viscoelasticity (Shoemaker, 1992; Wendlandt, 1986). Many investigations (Chen et al., 2007; Mano, Vaz, Mendes, Reis, & Cunha, 1999; Thomazine, Carvalho, & Sobral, 2005; Laaksonen & Roos, 2000) reported that DMA had a great functionality in measuring the viscoelastic properties of biopolymers as well as starch-based films. It was suggested (Jiang, Qiao, & Sun, 2006) that a good miscibility in thermoplastic acetylated starch and poly(ethylene-*co*-vinyl alcohol) blends had been detected via DMA. The moduli represent the mechanical characteristics of starch viscoelastic system during gelatinization and retrogradation. The important aspect of retro-

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Nomenclature

G'	storage modulus
G''	loss modulus
$\tan\delta$	loss factor
T	temperature, °C
T_1	temperature at a certain point, °C
T_2	temperature at a certain point, °C
T_p	maximum peak temperature, °C
T_g	glass transition temperature, °C

gradation is the increase of storage modulus (G'), which is treated as the measurement of retrogradation.

Recent studies indicated that the existence of a plasticizer was able to affect the temperature of transitions like T_g. It was suggested (Da Róz, Carvalho, Gandini, & Curvelo, 2006) that shorter glycols and sorbitol were effective while monohydroxyl alcohols and high molecular weight glycols failed in starch plasticization. It was reported (Cheer & Lelievre, 1983) that less than 20% sucrose concentration in a wheat starch–water–sucrose system caused increase in apparent viscosity, yield stress, dynamic viscosity and dynamic rigidity. The plasticizing effect of water showed that a decreasing trend might occur in the glass transition temperature (T_g) of high molecular weight glutenin subunits with the increase of water content (Castelli, Gilbert, Caruso, Maccarrone, & Fisichella, 2000).

The goal of the present investigation was to characterize the temperature dependence of the dynamic storage modulus and the loss factor of the maize and potato starch film samples at various sucrose contents.

2. Materials and methods

2.1. Materials

Commercial grade maize starch and potato starch were purchased from Xinghua Starch Plant (Chaoyang District, Beijing, China). Sucrose was obtained from Beijing Chemical Plant. Bone dry maize starch and potato starch were obtained using a convective oven (101-3, Shanghai Luda Instrument Co., Ltd., China) at 103 ± 2 °C for 6 h under constant air pressure and stored at ambient temperature (20 °C) in a sealed desiccator for further use. Deionized (DI) water was used, when required.

2.2. Film preparation

The flow chart for preparing maize and potato starch film samples is illustrated in Fig. 1. The main steps and parameters of sample preparation were as follows. Varying amounts of dried starch and sucrose were dissolved in 90 ml of DI water so that the total volume was 100 ml. The proportions of sucrose, starch and DI water are listed in Table 1. All the solid components (sucrose, maize starch and potato starch) were weighed using an electronic balance (AB135-S, Mettler Toledo, Switzerland). The beaker was placed in a water-bath. The mixture suspension was heated up to 75.0 °C at the rate of 0.67 °C/min under common air pressure and maintained at 75.0 °C for more than 20 min with continuous stirring at a speed of 200 rpm. After the completion of gelatinization, the obtained mixture was spread evenly on the flat surface of glass board to form the starch film via drying. The thickness of the starch paste was less than 0.5 mm. When the starch paste dried completely, sample of the starch films with different sucrose content were obtained. In order to match to the size of DMA film tension clamp, samples with dimension 5.0 × 30.0 × 0.1 mm were cut from the starch films mentioned above by a razor blade and the edges of each film were carefully smoothed with fine sand paper. The dimensions of each square were measured by an electronic digital caliper (PRO-MAX, Fowler, USA). All samples were reserved at ambient temperature (23 °C) for dynamic mechanical measurements.

2.3. Dynamic mechanical analysis (DMA)

The dynamic mechanical measurements were conducted using a Q800 Dynamic Mechanical Analyzer (TA Instruments, New Castle, USA) with film tensile clamp at single-frequency scanning mode of 0.35 Hz (angular frequency $\omega = 2.2$) and a heating rate of 3.0 °C/min over a temperature range from 40.0 °C to 140.0 °C, except P0 (potato starch film with 0% sucrose), which ran to 135.0 °C. A dynamic strain of 5 μm and amplitude of 1 μm were applied in all the DMA experiments. Before and the measurements, the temperature of samples and the instrument was well equilibrated at 40.0 °C. To prevent water loss during the experiment, the exposed surface of each sample was partially wrapped with aluminum foil, leaving the bottom and top in direct contact with the clamp holders (Pereira & Oliveira, 2000). All necessary thermal and mechanical calibrations of the instrument were performed before the experiments according to its operation manual (TA Instruments, 2004). The *Poisson* ratio was 0.38. All measurements were conducted at least in duplicate.

2.4. DMA results analysis

The glass transition and the onset temperature values were analyzed and calculated using TA Instruments Universal Analysis 2000

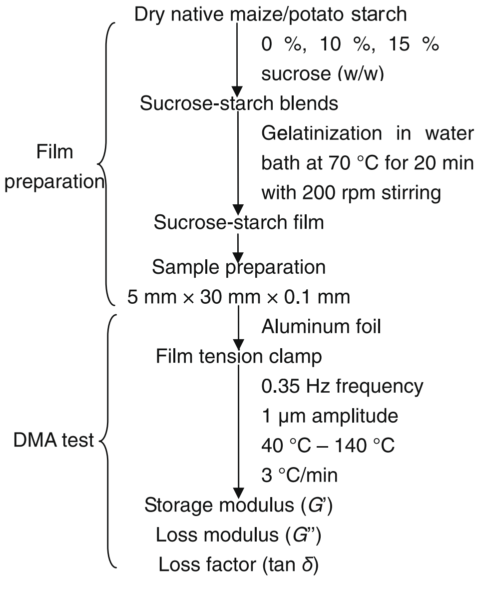

Fig. 1. Flow chart of starch film preparation and DMA test.

Table 1
The proportion of sucrose, starch and DI water[a].

Samples		Sucrose (g)	Starch (g)	DI water (ml)
Maize starch films	M0	0	10.0	90
	M10	1.0	9.0	90
	M15	1.5	8.5	90
Potato starch films	P0	0	10.0	90
	P10	1.0	9.0	90
	P15	1.5	8.5	90

[a] M refers to maize starch film; P refers to potato starch film; 0, 10 and 15 refer to sucrose content (%); DI water refers to de-ionized water.

Version 4.3A Software (TA Instruments-Waters LLC, USA). Values are expressed in means ± standard deviations ($n = 3$).

3. Results and discussion

Water acts as one of the most excellent plasticizers for biopolymers (Sothornvit & Krochta, 2005). The increased water content will significantly decrease the T_g of starch. Synthetic effects were observed when both water and sucrose were added into starch. The dynamic mechanical properties of the starch film depended on those three components (starch, water and sucrose), in which two of them (water and sucrose) were plasticizers. Moisture loss is a major problem for the DMA measurement of food materials, thus DMA experiments require strict sample preparation procedures especially for food materials. Some procedures like wrapping and coating the sample with aluminum foil and silicone oil should be taken to prevent heating caused water loss during measurement.

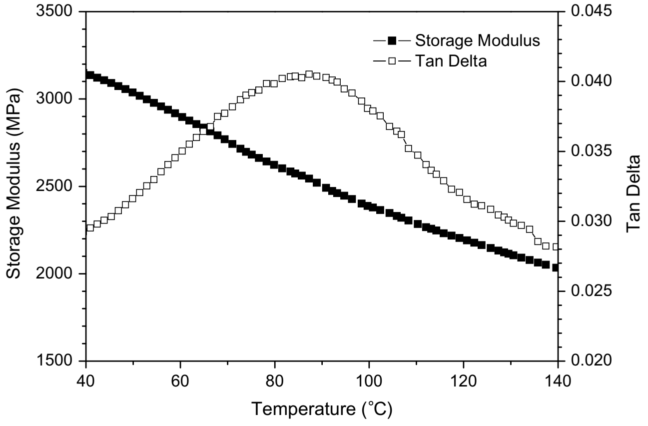

Fig. 2. DMA spectra as a function of temperature: maize starch film with 0% sucrose.

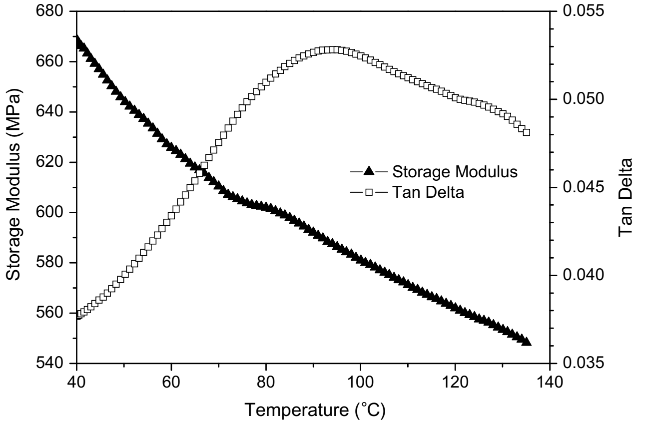

Fig. 3. DMA spectra as a function of temperature (from 40.0 to 135.0 °C): potato starch film with 0% sucrose.

The correlation curves for storage modulus and loss factor as a function of temperature are illustrated in Fig. 2 (maize starch film with 0% sucrose) and Fig. 3 (potato starch film with 0% sucrose). The two figures show extremely similar trends of the storage modulus and loss factor of the sample M0 (maize starch film with 0% sucrose) and sample P0 (potato starch film with 0% sucrose). The storage modulus of the two samples decreased continuously with the increasing temperature although they had different values. The loss factor of the sample M0 and sample P0 increased first then decreased as the temperature continued to increase. This agrees with the proposal of Lee, Kim, and Nishinari (1998) that the network structure of starch gel with sucrose content was weakened by heating. The loss modulus (not illustrated) increased to the peak value of 105.0 ± 0.9 MPa at 80.22 ± 5.30 °C (M0) and 31.43 ± 0.03 MPa at 88.75 ± 2.21 °C (P0) and then decreased gently till the end of the experiment. It was found that the terminal values of loss modulus (G'') were lower than the points under their own initial temperatures (not illustrated). Loss factor increased with the increase of temperature. Peak values of loss factor reached 0.04046 ± 0.00007 at 86.48 ± 1.87 °C (M0) and 0.05290 ± 0.00001 at 95.13 ± 0.87 °C (P0), respectively. The loss factor then decreased abruptly and reached a stable point at the end. While the frequency of polymer chain motion was equal to the frequency provided by the instrument, the friction force caused by chain motion between molecules would consume mechanical energy quickly as $\tan\delta$ reached the maximum value. The $\tan\delta$ peak value appeared later than the peak value of loss modulus G''. Within the temperature range from 120.0 °C to the end, there was an unexpected inflection point. A possible reason could be the evaporation of the residual moisture in the film even though it was covered with aluminum foil. The curves of loss modulus and loss factor showed the same trends. The only difference was that the damping peak on loss modulus G'' occurred earlier than that of $\tan\delta$. It can be explained with the following derivation (Akay, 1993; Li, Lee-Sullivan, & Thring, 2000). Suppose the inflection point of storage modulus G' and the peak value of loss modulus (G'') occurred at the moment $T = T_1$, such that the relation between $\tan\delta$ and temperature can be described as follows:

$$\frac{d(\tan\delta)}{dT} = \frac{d}{dT}\left(\frac{G''}{G'}\right) = \frac{\left(\frac{dG''}{dT}\right)G' - G''\left(\frac{dG'}{dT}\right)}{G'^2}$$

For

$$\frac{dG''}{dT}|_{T=T_1} = 0$$

therefore,

$$\frac{d(\tan\delta)}{dT} = -\frac{G''\left(\frac{dG'}{dT}\right)}{G'^2}$$

While

$$\frac{dG'}{dT}|_{T=T_1} < 0$$

so,

$$\frac{d(\tan\delta)}{dT}|_{T=T_1} > 0$$

This indicates that $\tan\delta$ is still going to increase at the moment $T = T_1$. Equally, it means that $\tan\delta$ will still increase after the peak value appeared in the storage modulus curve and it will reach another peak at temperature T_2 ($T_2 > T_1$).

Starch films demonstrated a relaxation process under a relatively lower frequency (0.35 Hz) and within the temperature range from 40.0 to 140.0 °C, except P0, which ran to 135.0 °C. This process was associated with a certain molecular motion, which directly led to the viscoelasticity changes along with the increased temperature. When a small oscillating sine stress is applied to the sample at a certain temperature range, its strain will appear later than stress due to the viscoelasticity of sample. Such relaxation phenomena were more significant in the loss modulus spectra and the loss factor spectra than in the storage modulus spectra. This probably directly resulted from the complicated structure of starch and its derivatives. Dynamic mechanical properties of polymers can sensitively represent their molecular motion, which has a close relationship with the condensed and chain structures of polymers. When a crystalline to amorphous state transition happened on each specific moving unit, a modulus slope or a damping peak would appear in DMA spectra. Storage modulus changed slightly along with the temperature when the relaxation time was long. The peak of loss modulus or tan δ was also flat and wide at that moment. The tan δ peak was getting narrower and higher along with the increasing sucrose content. Changes in the shape of peaks indicated that the transition rate was becoming higher.

DMA is also a helpful technique to estimate the stiffness increase caused by the sucrose addition. Fig. 4 shows the influence of sucrose content on the loss factor (tan δ) of potato starch film. At the beginning of heating, the tan δ values increased slowly until reaching a peak and then decreased. The tan δ peak values of 0.05290 ± 0.00001 (P0), 0.07930 ± 0.00001 (P10) and 0.09427 ± 0.00008 (P15)were reached at 95.13 ± 0.87 °C (P0), 85.52 ± 1.98 °C (P10), and 63.08 ± 0.74 °C (P15), respectively. In correspondence, the loss modulus peaks (illustrated in Fig. 5) showed similar profiles to the loss factor. Pure potato starch film had an extremely lower loss modulus value and a lower peak height. The temperature of the peak point decreased from 88.75 ± 2.21 °C (P0) to 74.94 ± 3.44 °C (P10) and finally to 57.36 ± 2.29 °C (P15) with increasing sucrose contents. The corresponding peak loss modulus values were 31.43 ± 0.03 MPa (P0), 126.0 ± 0.69 MPa (P10) and 209.90 ± 0.30 MPa (P15) for 0%, 10%, and 15% of sucrose, respectively. It can be seen that the absolute values of loss modulus increases with the sucrose content. The higher the sucrose concentration, the earlier the tan δ peak occurred and the higher the peak value. The delay between loss modulus peak and loss factor peak provides additional evidence indicating that the tan δ peak appeared at a higher temperature than that of the loss modulus peak. The decrease of glass transition temperature (temperature at peak point) is probably due to the existence of sucrose. All maize starch films containing sucrose presented a similar parabolic trend in loss factor. Similar results was also reported by Famá, Flores, Gerschenson, and Goyanes (2006) that an increased sorbate content in cassava starch biofilm displaced the glass transition temperature of a glycerol-rich phase toward lower temperatures. It was found out that the tan δ peaks moved from higher temperature to a lower value with the increase of sucrose content. DMA provides three curves representing the changes during heating. Generally, glass transition can be defined by the change in storage modulus, the peak in loss modulus or the peak in loss factor (Rahman, Al-Marhubi, & Al-Mahrouqi, 2007). Most commonly, people use loss factor peak as the symbol of glass transition. This point reveals that it is the most sensitive motion detected by the instrument.

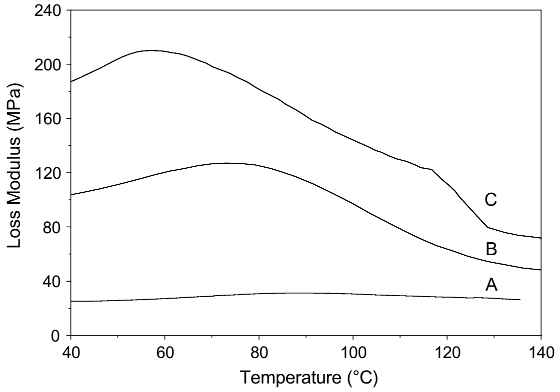

Fig. 5. Effect of sucrose content on the loss modulus of potato starch film (A, 0% sucrose, from 40.0 to 135.0 °C; B, 10% sucrose; C, 15% sucrose).

As shown in Fig. 6, two tan δ peaks of 0.06148 ± 0.00018 and 0.06356 ± 0.00001 were obtained at 59.81 ± 1.86 °C and 95.96 ± 1.67 °C, respectively, for maize starch after two days retrogradation. But after seven days retrogradation, only one peak remained. It is proposed that sucrose and maize starch merged into one stable system, which is appeared as a symbol of starch retrogradation under the existence of sucrose content. The peak of tan δ decreased to 0.04049 ± 0.00020 and the corresponding temperature increased to 85.46 ± 5.50 °C. Similar changes happened in the potato starch films. The loss factor (tan δ) decreased and storage modulus (G') increased sharply because of retrogradation. For samples with added sucrose, the loss factor of retrograded starch films still remained higher than that of the samples without sucrose. Chung and Lim (2000) reported very similar results, which were also influenced by physical aging. The retrogradation rate became slower with the sucrose content increased from 0% to 10% and 15%.

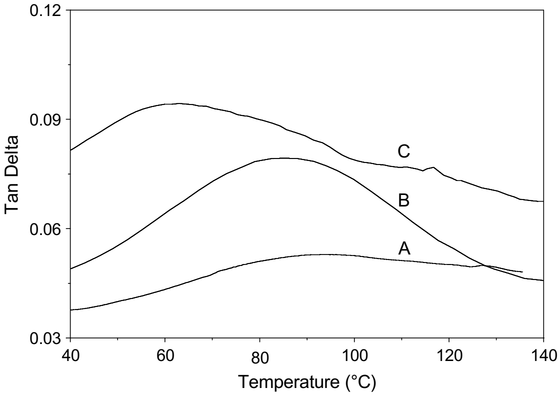

Fig. 4. Effect of sucrose content on the loss factor (tan δ) of potato starch film (A, 0% sucrose, from 40.0 to 135.0 °C; B, 10% sucrose; C, 15% sucrose).

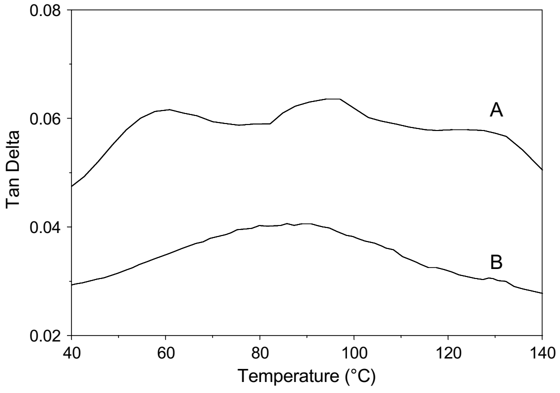

Fig. 6. Effect of aging time on the loss factor (tan δ) of pure maize starch film (A, aged for two days; B, aged for seven days) vs. temperature.

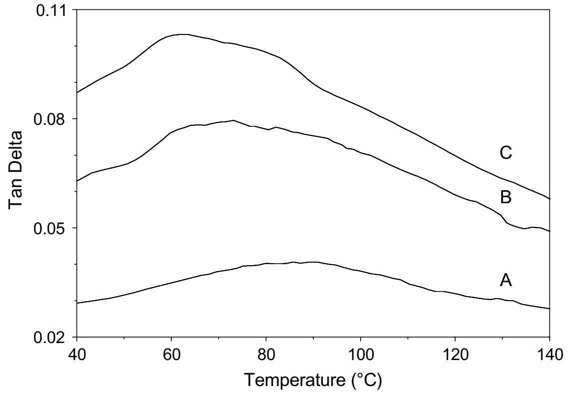

Fig. 7. Effect of sucrose content on the loss factor (tan δ) of seven-day-aged maize starch film (A, 0% sucrose; B, 10% sucrose; C, 15% sucrose) vs. temperature.

Fig. 7 shows maize starch film with different sucrose contents aged for seven days. The tan δ peak increased from 0.04049 ± 0.00020 (M0) to 0.07884 ± 0.00083 (M10), and finally to 0.1029 ± 0.0004 (M15) and the corresponding temperature decreased sharply from 85.46 ± 5.50 °C (M0) to 73.22 ± 3.32 °C (M10) and finally to 63.35 ± 2.84 °C (M15) when the sucrose content increased from 0% to 10%, and finally to 15%, respectively. The peak of tan δ appeared at higher temperature with the increase of sucrose content in the maize starch film. In other words, the aging rate of maize starch film was restricted by sucrose to some extent. It is possible that the glass transition region of the starch film can be moved to adapt the practical demands through adding certain amount of sucrose. This might control the mechanical damping of starch films during the preservation of heating process.

4. Conclusions

While applied with tensile force, the storage modulus of maize and potato starch films decreased as the temperature increased; and a peak of loss factor indicating glass transition region was obtained. There was a delay between the modulus peak and transition. The delay between the storage/loss modulus peak and loss factor (tan δ) peak could be considered as the range of glass transition. The glass transition temperature (tangent peak point corresponding temperature) decreased with the addition of sucrose in maize and potato starches. The loss factor of maize starch film was also affected by the retrogradation. Different sucrose content added to the maize starch films showed changes in tan δ peak at different temperature after seven days. It was proposed that if an optimal ratio of sucrose and starch content could be found, an equivalent point of the mechanical damping and the life span of maize starch film would reached.

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