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Rheological properties of waxy maize starch and xanthan gum mixtures in the presence of sucrose

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ABSTRACT

Rheological properties of 5% (w/w) waxy maize starch (WMS)/xanthan gum (XG) mixed pastes (mixing ratios: 10/0, 9.5/0.5, 9/1) in the presence of sucrose (0%, 15%, and 30%) were studied. The 5% WMS paste displayed strong anti-thixotropic behavior based on the heating method adopted (heating in water bath for 40 min with mixing at 200 rpm). The addition of XG could accelerate the formation of new structure of the WMS/XG mixed pastes under shear conditions, and XG could also decrease the in-shear recoveries of the mixtures. Both the addition of XG and sucrose increased the apparent viscosity (η_a) and dynamic moduli (G' and G'') of the mixed pastes, with XG contributing to the solid-like properties of the mixtures obviously, while sucrose played a contrary role. Thermostable properties of the mixed pastes could be improved distinctly with the addition of XG, whereas sucrose decreased it slightly.

1. Introduction

It is reported that waxy maize was first found in China, and China has the breed advantages of waxy maize (Liu, Huang, Rong, Tian, & Yang, 2005). Compared with other starches, waxy maize starch (WMS) has many specific attributes and it is a promising raw material that can be used in food industry. WMS contains about 99% amylopectin, so it is an essentially amylose-free starch (Achayuthakan & Suphantharika, 2008). As a result, WMS shows low pasting temperature, high peak viscosity, little setback, and low final viscosity, which means it can swell easily, give sticky texture, and hardly retrograde (Hibi, 2001). Furthermore, WMS can be digested more easily than normal maize starch. Therefore, waxy maize starch is suitable to food processing, especially in the production of puffed foods or baked foods. However, native starches have many innate weaknesses, which restricted their applications. For example, they have the tendency to retrograde and undergo syneresis. In addition, gels or pastes of starches in native form like WMS are easy to break down either from prolonged heating, high shear or acidic conditions, producing weak-bodied, cohesive, rubber pastes and undesirable gels (Whistler & BeMiller, 1997).

Traditionally, starches and non-starch hydrocolloids were often used together in food systems to modify or control rheological and textural properties and improve the stability of food products (Chaisawang & Suphantharika, 2005; Nagano, Tamaki, & Funami, 2008). The effects of various hydrocolloids addition on starch gels or pastes, such as xanthan gum (XG), guar gum, gellan, carrageenan, or flaxseed gum have been studied extensively, and these studies provided useful information (Chaisawang & Suphantharika, 2005; Nagano et al., 2008; Rodríguez-Hernández, Durand, Garnier, Tecante, & Doublier, 2006; Tischer, Noseda, Freitas, Sierakowski, & Duarte, 2006; Wang et al., 2008a). It is reported that the addition of polysaccharide gums such as XG to starch pastes can cause an increase in viscosity and elasticity, and restrict retrogradation and syneresis of the starch based systems (Chaudemanche & Budtova, 2008). As a polysaccharide, XG is most extensively used in food production next only to starch (Viturawong, Achayuthakan, & Suphantharika, 2008). XG is a heteropolysaccharide which has a 1,4-linked delta-D-glucose (like cellulose) backbone, with a trisaccharide side chains consisting of two mannose molecules and one glucuronic acid molecule (Pongsawatmanit & Srijunthongsiri, 2008). The trisaccharide side chains align closely with the main chain, making the XG molecule a rather rigid, rod-like conformation, with extraordinary stability to heat, acid, and alkali (Achayuthakan & Suphantharika, 2008). Besides, XG is reported to exhibit high viscosities and significant yield stress values even at a low concentration (Morris, 1995).

Starch-containing foods are generally composed of a complicated system, and the starch/gum system often coexists with other ingredients in food products, such as sugars. Among the taste substances, sugars are the most widely used in starch products

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Nomenclature

G'	storage modulus, Pa
G''	loss modulus, Pa
K	consistency index, Pa s^n
K'	index, Pa s^n
K''	index, Pa s^n
n	flow behavior index (dimensionless)
n'	frequency exponent (dimensionless)
n''	frequency exponent (dimensionless)
R^2	correlation coefficient (dimensionless)
$\dot{\gamma}$	shear rate, s^{-1}
η_{app}	apparent viscosity, Pa s
τ	shear stress, Pa
ω	angular frequency, rad/s

(Hirashima, Takahashi, & Nishinari, 2005). The addition of sugars could affect the gelation, retrogradation, and rheological properties of starch, depending on the types as well as concentrations of both sugar and starch (Acquarone & Rao, 2003; Ahamad & Williams, 1999; Chang, Lim, & Yoo, 2004). In general, sugars can increase the peak viscosity and pasting temperature, restrict swelling and amylose leaching of starch (Ahamad & Williams, 1999; Baek, Yoo, & Lim, 2004; Krüger, Ferrero, & Zaritzky, 2003; Richardson, Langton, Bark, & Hermansson, 2003), while conflicting information has been given about the effect of individual sugars on retrogradation of starch (Arunyanart & Charoenrein, 2008; Hirashima et al., 2005). Among sugars, sucrose is one of the major food ingredients used, in order to adjust the taste of sweetness and also to alter thermal and physical properties of starch-based foods (Pongsawatmanit, Temsiripong, & Suwonsichon, 2007), and about 100 million metric tons of sucrose are produced annually (Izydorezyk, 2005).

Up to now most research has focused on the effect of sucrose on starch, while few reports concerning the effect of sucrose on starch/hydrocolloid mixtures. Therefore, the aim of this study is to investigate the rheological properties of WMS/XG mixtures in the presence of sucrose, so as to provide useful information for the processing of starch-based products. It has been demonstrated that the leached amylose molecules and certain gums would interact with each other, making the starch/gum mixtures a rather complicated system (Shi & BeMiller, 2002). However, the WMS used in this study was essentially amylose-free, which avoided the complications normally existing when amylose is present in the continuous phase (Achayuthakan & Suphantharika, 2008; Rodríguez-Hernández et al., 2006). Also, unlike amylose which is linear or slightly branched, amylopectin is highly branched and does not form gels under normal conditions (Banks & Greenwood, 1975), so rheological properties can be detected without the interference of gelation of the pastes.

It is reported that the rheological properties of starch pastes may differ greatly depending on different pasting conditions (Tischer et al., 2006). Therefore, in the preliminary study three different pasting conditions of WMS suspensions were adopted; and based on the results, appropriate heating method was chosen in the following study.

2. Materials and methods

2.1. Materials

Commercial WMS (11.5 wt.% moisture, wet basis) was purchased from Jinan Jinwang Food Co., Ltd. (Shandong Province, China). Food grade XG was obtained from Shandong Fufeng Company (Shandong Province, China).

2.2. Preparation of WMS pastes with different pasting conditions

WMS suspension (5.0 wt.%) was prepared by adding 2.5 g WMS into 47.5 g deionized water at room temperature (about 25 °C). Three different pasting methods were adopted to study the effect of heating condition on the rheological properties of WMS paste: (1) about 1.3 ml WMS suspension was heated in the rheometer (TA Instruments Ltd., Crawley, UK) with heating and cooling cycles in the following manner: the slurry was held at 50 °C for 1 min, then increased to 95 °C at a heating rate of 12 °C/min and held at 95 °C for 2 min. It was subsequently cooled to 50 °C at 12 °C/min, and held at 50 °C for 1 min. During the entire treatment, the sample was held under steady shear at a shear rate of 200 s^{-1} (Park, Ibáñez, Zhong, & Shoemaker, 2007). (2) Well mixed WMS suspension (50 g) in conical flask was heated in a water bath at 95 °C for 6 min with a constant mixing rate of 200 rpm, then the paste was rapidly cooled in another water bath at 40 °C for 1 h before testing. (3) Well mixed WMS (50 g) suspension was heated in a water bath at 95 °C for 40 min with a constant mixing rate of 200 r/min. Then the paste was rapidly cooled in another water bath at 40 °C for 1 h before testing. During heating and cooling in (2) and (3), the conical flask was sealed with a rubber stopper to minimize moisture lost.

2.3. Preparation of WMS pastes mixed with XG and sucrose

The 5 wt.% WMS/XG mixtures at different mixing ratios (10/0, 9.5/0.5, 9/1) containing 0%, 10% and 30 wt.% sucrose were prepared as follows. Pre-calculated amounts of XG were added to preweighed deionized water in conical flask at room temperature, with moderate magnetic stirring for at least 30 min to completely dissolve the gum. Then, WMS and sucrose were added to achieve a total weight of 50 g, with magnetic stirring for another 30 min to dissolve sucrose and hydrate WMS. After that, the well dispersed mixtures were heated in a water bath at 95 °C for 40 min, with a constant mixing rate of 200 rpm. Then the pastes were rapidly cooled in another water bath at 40 °C for 1 h before measurements.

2.4. Determination of rheological properties

Rheological properties of all samples were measured using AR2000ex rheometer (TA Instruments Ltd., Crawley, UK) with an aluminum parallel plate geometry (40 mm diameter, 1 mm gap). The sample was placed into the rheometer which was equilibrated to 25 °C. The edge of the sample was covered with a thin layer of silicon oil to prevent water evaporation during measurements. For each sample, steady flow, in-shear structural recovery, viscoelastic and thermostable properties were determined.

2.4.1. Steady flow measurements

Steady flow measurements were performed at 25 °C to obtain shear rate versus shear stress data. The shear rate was programmed to increase from 0 to 300 s^{-1} in 3 min, followed immediately by a reduction from 300 to 0 s^{-1} in the next 3 min. The power law model expressed in Eq. (1) was fitted to the experimental data both from the ascending and descending segments of the shear cycle.

$$\tau = K\dot{\gamma} \tag{1}$$

where τ is the shear stress (Pa), $\dot{\gamma}$ is the shear rate (s^{-1}), K is the consistency index (Pa s^n), and n is the flow behavior index (dimensionless).

2.4.2. In-shear structural recovery measurements

In-shear structural recovery of the samples was determined according to the procedure of Mezger (2002) with some modifications. The sample was loaded into the rheometer at 25 °C, then a three stepped shear flow test was performed as follows: (1) a constant shear rate of 1 s^{-1} was applied for 120 s (with pre-shear at 1 s^{-1} for 30 s) and subsequently (2) a constant shear rate of 300 s^{-1} was applied for 60 s and then (3) a constant shear rate of 1 s^{-1} was applied for 180 s. The in-shear recovery value was calculated as the ratio of average apparent viscosity (η_a) obtained during the first 120 s of the third step to the average η_a value determined in the first step.

2.4.3. Viscoelastic properties

Small amplitude oscillatory tests were performed at 25 °C over the frequency range of 0.1–10 Hz (0.628–62.8 rad/s). The strain amplitude for the frequency sweep measurements was selected as 1%, which was in the linear viscoelastic region for all samples. The mechanical spectra were obtained recording storage modulus (G'), loss modulus (G'') and loss tangent ($\tan\delta = G''/G'$) as a function of angular frequency.

2.4.4. Thermostable properties

Thermostable properties of the samples were determined according to the procedure of Paula (Tischer et al., 2006) with small changes. After the sample was equilibrated at 5 °C for 2 min, the temperature of the sample was increased to 85 °C and subsequently its temperature was decreased to 5 °C with a ramp rate of 5 °C/min for both heating and cooling. For the tests a constant frequency of 1 Hz and a strain amplitude of 1% were used.

2.5. Statistical analysis

All experiments described above were made in triplicate for each sample. The data presented were the means and standard deviations of each experiment. A one-way analysis of variance (ANOVA) and Tukey's test were used to establish the significance of differences among the mean values at the 0.95 level of confidence. The statistical analysis were performed using SPSS (2003) version 13.0 for Windows program (SPSS Inc., Chicago, IL, USA).

3. Results and discussion

3.1. Steady flow measurements

3.1.1. Different pasting conditions on the flow properties of WMS pastes

The flow curves of WMS pastes with different pasting conditions are shown in Fig. 1. The WMS paste heated in rheometer (A) exhibited clockwise hysteresis loop, indicating that shear stress obtained from the increasing-order of shear rate (up curve) is larger than the corresponding value of the decreasing-order of shear rate (down curve), which suggested thixotropic behavior of the paste (Acquarone & Rao, 2003). The WMS paste heated in water bath for 6 min (B) was characterized by a combined hysteresis loop, i.e., clockwise loop (thixotropic behavior) at higher shear rates (50–300 s^{-1}) and counterclockwise loop (anti-thixotropic behavior) at lower shear rates (0–50 s^{-1}), and the counterclockwise loop was relatively small compared with the clockwise loop. Similar mixed behavior (thixotropic and anti-thixotropic) for WMS pastes was reported by Achayuthakan and Suphantharika (2008). For the WMS paste heated in water bath for 40 min (C), the counterclockwise loop completely dominated the hysteresis loop area, suggesting a significant anti-thixotropic behavior of the paste, and similar flow curves were observed for pastes from cross-linked WMS (Acquarone & Rao, 2003).

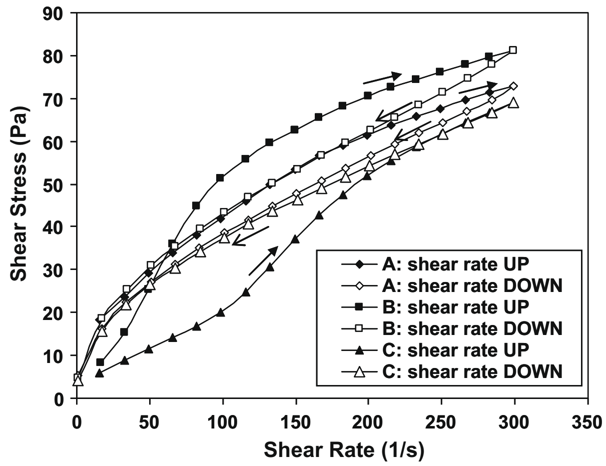

Fig. 1. Flow curves of 5% w/w WMS pastes with different pasting conditions measured at 25 °C: (A) pasting in rheometer; (B) pasting in water bath for 6 min; and (C) pasting in water bath for 40 min. The closed symbols represent the increasing of shear rate (i.e., up curve) and open symbols represent the decreasing of shear rate (i.e., down curve).

The differences in flow behaviors of the WMS pastes should be ascribed to different heating conditions. Generally speaking, when WMS suspensions pasted more completely with more heating time and agitation, the anti-thixotropic property became more significant. In the paste of native WMS, the continuous phase is essentially amylopectin dissolved during swelling of the granules. Therefore, all three pastes here could be regarded as biphasic system, i.e., suspensions of degraded starch granules embedded in a continuous matrix of amylopectin (Rodríguez-Hernández et al., 2006). And thus, both the corrupted granules and the continuous phase of amylopectin together determined the rheological properties of the WMS pastes. For the paste heated in rheometer, the clockwise hysteresis loop observed can be explained as structural breakdown by shear field, then a new structure of the paste formed, which maintained a shear-thinning characteristic on following shear sweeps (Achayuthakan & Suphantharika, 2008). Of course, the new developed texture here was less resistant to shear field, so the apparent viscosity decreased in the down curve. For the paste pasting in water bath for 40 min (Fig. 1C), a lot of amylopectin molecules dissolved in the continuous phase after sufficient heating and agitation. It is reported that amylopectin should be responsible for shear-thickening properties and also shear-induced structure formation and alteration, i.e., anti-thixotropic behavior of the paste (Dintzis, Berhow, Bagley, Wu, & Felker, 1996). Besides, previous investigation attributed the anti-thixotropic behavior of retorted cross-linked WMS dispersion to granule cluster formation: as the dispersions were sheared, starch granules rearranged to form clusters which in turn resulted in an increase in apparent viscosity (Chamberlain, Rao, & Cohen, 1999). However, in this study it appears that when the WMS dispersions gelatinized more thoroughly with more granules dissolved in the continuous phase (micrographs not shown), the anti-thixotropic property became more significant. Similar phenomenon was reported by Al-Malah, Azzam, and Abu-Jdayil (2000). Therefore, the anti-thixotropic behavior of the WMS pastes may be attributed to amylopectin dissolved in the continuous phase. For the paste pasting for 40 min, when subjected to shear conditions, maybe the well dispersed amylopectin matrix formed a more stable structure quickly, and thus the flow curve displayed anti-thixotropic behavior. For the WMS paste pasting for 6 min (Fig. 1B), both the amount and dispersion degree of amylopectin in the continuous phase was smaller, thus the new structure which was more shear-resistant formed later, compared with the WMS paste pasting for 40 min. Therefore, its flow curve displayed thixotropic behavior at first, and then anti-thixotropic behavior after the new structure formed. Certainly, more investigation about the anti-thixotropic behavior of the WMS paste should be done in the future.

Besides, Korus, Juszczak, Witczak, and Achremowicz (2004) suggested the counterclockwise loop cannot be classified as typical anti-thixotropy, and one definition of the anti-thixotropy is an increase in apparent viscosity with time under constant shear rate (Ferguson & Kembowski, 1995). However, for the samples displayed counterclockwise loop in this study, increase in apparent viscosity with time under constant shear rate was also observed. Fig. 2D shows the shear stress against time curve for the WMS-0.5% XG-30% sucrose sample subjected to a constant shear rate of 300 s^{-1}. As can be seen from Fig. 2D, the sample shows anti-thixotropic behavior since the shear stress increases with increasing of time. Therefore, the counterclockwise loop of the curves could be regarded as anti-thixotropic behavior, as many reports did (Achayuthakan & Suphantharika, 2008; Acquarone & Rao, 2003; Dintzis et al., 1996; Tattiyakul & Rao, 2000). As the anti-thixotropic behavior of the native WMS paste was rarely reported and worthy of investigation, the heating method of water bath at 95 °C for 40 min was adopted in the following studies.

3.1.2. Steady flow measurements of the mixed pastes

The flow curves of the samples are given in Fig. 2. As shown in Fig. 2, it could be observed that all the samples displayed a counterclockwise loop area, which indicated the anti-thixotropic behavior. It is reported that for the WMS/XG mixed paste with 1.0% XG and 5.0% WMS, the majority of the loop was found to be slightly anti-thixotropic (Achayuthakan & Suphantharika, 2008). However, in our study all the samples displayed distinctly anti-thixotropic property, suggesting such a behavior could be ascribed to WMS based on the heating method adopted (heating in water bath for 40 min). From Fig. 2, it could also be observed that the flow

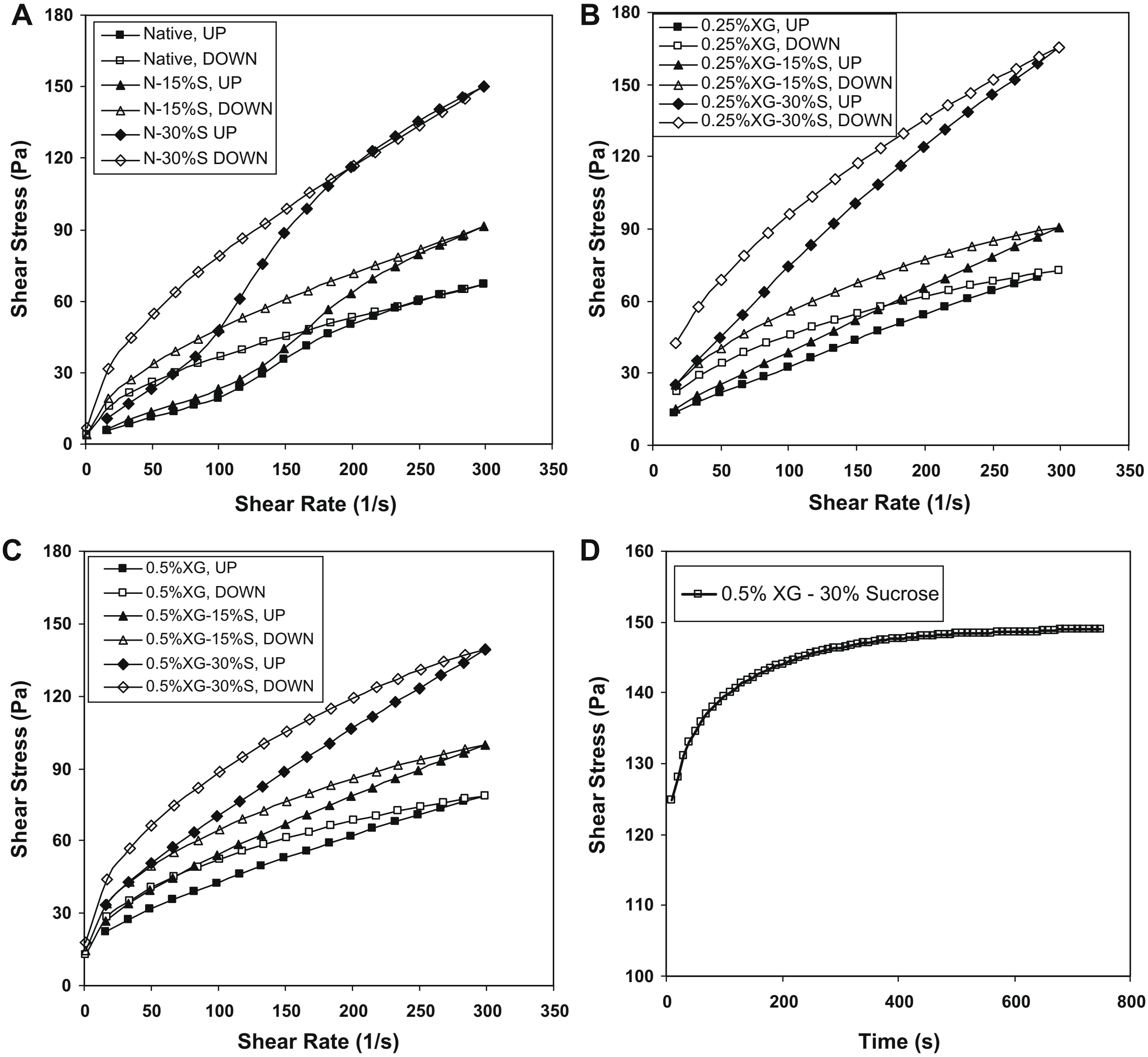

Fig. 2. Flow curves of 5% w/w WMS/XG mixed pastes at mixing ratios of 10/0 (A), 9.5/0.5 (B), 9/1 (C) containing 0%, 15%, and 30% w/w sucrose, and the shear stress against time for the 5% w/w WMS/XG mixed paste at a mixing ratio of 9.5/0.5 containing 30% w/w sucrose at a shear rate of 300 s^{-1} (D). Measurements were made at 25 °C.

curve of native WMS paste was characterized by a counterclockwise loop existing in the majority of the shear rate range, and such situation did not change after the addition of sucrose. However, after XG was added, the counterclockwise loops completely covered all the shear range, indicating XG played a role of accelerating the formation of the new structure under shear conditions. As shown in Fig. 2, the maximal value of shear stress was less than 180 Pa, which was in agreement with Tattiyakul and Rao (2000) that the anti-thixotropic behavior occurred when the shear stress values were less than 200 Pa. The consistency index (*K*), flow behavior index (*n*), and apparent viscosity at shear rate of 100 s^{-1} ($\eta_{a,100}$) as a function of both XG and sucrose concentration were obtained from the power law model, as shown in Table 1. From Table 1, a much bigger value of *K* can be observed in the down flow curve than in the up flow curve, indicating a more significant thickening effect of XG in the down phase of shear, in agreement with the anti-thixotropic behavior of the samples. Besides, the *n* value of the down curve is smaller than the corresponding value of the up curve, indicating an increase in pseudoplasticity of the new-formed structures.

To both up and down curves, the *K* value has a weak trend to increase with sucrose at a given XG concentration. However, XG played a determinant role on the increase of *K* due to its remarkable thickening effect. XG could also influence the flow behavior index (*n*) evidently. In general, *n* decreased with the addition of XG, indicating XG could increase the pseudoplasticity of the mixtures. However, when at a given XG concentration, increase in sucrose caused a small increase of *n* for both up and down curves, indicating the pseudoplastic behavior of the mixtures was weakened by sucrose. Similar results were reported for cross-linked WMS/sucrose mixed pastes (Acquarone & Rao, 2003). Such results could be attributed to the stabilizing effect of the water structure caused by sucrose (Aee, Hie, & Nishinari, 1998; Tait, Suggett, Franks, Ablett, & Quickenden, 1972). It is reported that the pseudoplastic behavior of the macromolecule solution could be ascribed to the disentanglement of long chain molecules, which reduced the intermolecular resistance to flow under shear conditions (Nurul, Azemi, & Manan, 1999). However, when the water was immobilized by sucrose, the thermal motion of the stabilized water in the mixtures decreased (Chang et al., 2004), therefore the disentanglement of long chain molecules was suppressed under shear conditions, and thus the pseudoplasticity of the mixtures decreased.

Both the addition of sucrose and XG could increase $\eta_{a,100}$ of the up and down flow curves. To sucrose, it is reported a low content of sucrose (<20%) could enhance the swelling of starch granules, leading to the increase in viscosity of the pastes (Hirashima et al., 2005). In addition, it is suggested sucrose perhaps changed the structure of water as a solvent, and immobilized water molecules (Aee et al., 1998). The degree of modification of water structure induced by different polyols was determined by their steric compatibility with the pattern of hydrogen bonding between water molecules. For sucrose, the spacing of equatorial hydroxyl groups on sugar rings matches the “lattice” structure of liquid water, so the equatorial hydroxyl groups in a sugar molecule could effectively substitute for a water molecule, and hence the hydration of sugar hydroxyl groups has a stereospecific nature, which produces a stabilizing effect of the water structure (Aee et al., 1998; Tait et al., 1972). So when the water was immobilized by sucrose, the thermal motion of the stabilized water in the mixtures decreased, and thus the motion of the polysaccharide chains was suppressed under shear conditions, causing the increase of the apparent viscosity (Chang et al., 2004). Krüger et al. (2003) also pointed out that solutes which have compatibility with water structure, just like sucrose, may have a greater access to the hydration layer of the starch chains, and this situation creates a localized environment of increased viscosity. It could also be observed in Table 1 that the increment of apparent viscosity is not proportional with the increment of sucrose concentration, i.e., 30% sucrose could more effectively increase the apparent viscosity of the pastes, and this trend didn’t change with the presence of XG.

The addition of XG could also increase $\eta_{a,100}$ of the mixtures, as can be seen in Table 1. The increase in viscosity of the starch–hydrocolloid blends was usually attributed to the synergistic effect of the XG, which could be interpreted as a result of phase separation of different polysaccharides induced by the thermodynamic incompatibility between them (Annable, Fitton, Harris, Phillips, & Williams, 1994). Because of the mutual exclusion of starch and hydrocolloid, the microstructures of the starch/hydrocolloid gels formed into a starch-rich disperse phase and a hydrocolloid-rich continuous phase, and the increased concentration in their respective micro-domains increased the overall viscosity of the mixtures (Annable et al., 1994).

As indicated by Table 1, when the concentration of sucrose was 15%, $\eta_{a,100}$ increased with the increase in XG concentration. However, when sucrose content was 30%, $\eta_{a,100}$ decreased a little as XG increased from 0.25% to 5% (from 0.75 ± 0.02 to 0.71 ± 0.02 Pa s of the up curves, 0.95 ± 0.01 to 0.88 ± 0.02 Pa s of the down curves). Such results indicated sucrose at high concentration (30%) may influence the synergistic effect in viscosity of XG. It is well known that water is a particularly effective solvent for biopolymers, because of its ability to form hydrogen bonds with hydroxyl groups and other polar substituents of the polymer chains (Evageliou, Richardson, & Morris, 2000). However, as indicated above, sucrose could exhibit high compatibility with liquid water, and the equatorial hydroxyl groups of sucrose molecule could effectively hydrate with water molecules (Aee et al., 1998). Therefore, the conformational ordering and intermolecular association of

Table 1
The Power law parameters and apparent viscosity ($\eta_{a,100}$) for 5% w/w WMS/XG mixed pastes at mixing ratios of 10/0, 9.5/0.5, 9/1 containing 0%, 15%, and 30% w/w sucrose.[A]

XG (%)	Sucrose (%)	Up curve				Down curve			
		$\eta_{a,100}$ (Pa s)	K (Pa s^n)	n	R^2	$\eta_{a,100}$ (Pa s)	K (Pa s^n)	n	R^2
0	0	0.20 ± 0.00^{a}	0.31 ± 0.02^{a}	0.94 ± 0.01^{a}	0.974	0.37 ± 0.01^{a}	4.33 ± 0.21^{a}	0.47 ± 0.01^{a}	0.997
0	15	0.23 ± 0.00^{b}	0.29 ± 0.00^{a}	1.00 ± 0.00^{b}	0.968	0.48 ± 0.01^{b}	4.78 ± 0.26^{a}	0.50 ± 0.01^{b}	0.998
0	30	0.48 ± 0.02^{c}	0.54 ± 0.03^{b}	1.00 ± 0.01^{b}	0.983	0.79 ± 0.01^{c}	8.19 ± 0.32^{b}	0.50 ± 0.01^{b}	0.997
0.25	0	0.33 ± 0.01^{a}	2.07 ± 0.04^{a}	0.61 ± 0.01^{a}	0.986	0.45 ± 0.01^{a}	8.53 ± 0.26^{a}	0.37 ± 0.00^{a}	0.994
0.25	15	0.39 ± 0.00^{b}	2.27 ± 0.07^{a}	0.63 ± 0.01^{b}	0.985	0.55 ± 0.01^{b}	8.67 ± 0.20^{a}	0.41 ± 0.00^{b}	0.996
0.25	30	0.75 ± 0.02^{c}	3.41 ± 0.12^{b}	0.68 ± 0.00^{c}	0.995	0.95 ± 0.01^{c}	13.80 ± 0.74^{b}	0.43 ± 0.01^{c}	0.994
0.5	0	0.43 ± 0.00^{a}	5.78 ± 0.24^{a}	0.45 ± 0.01^{a}	0.984	0.52 ± 0.00^{a}	12.23 ± 0.30^{a}	0.32 ± 0.01^{a}	0.992
0.5	15	0.54 ± 0.01^{b}	6.54 ± 0.14^{b}	0.47 ± 0.00^{b}	0.987	0.64 ± 0.01^{b}	13.81 ± 0.16^{b}	0.34 ± 0.00^{b}	0.992
0.5	30	0.71 ± 0.02^{c}	7.08 ± 0.14^{c}	0.51 ± 0.00^{c}	0.986	0.88 ± 0.02^{c}	16.86 ± 0.36^{c}	0.37 ± 0.00^{c}	0.992

Mean ± standard deviation values in the same column for each solution followed by different superscripts are significantly different ($p \leqslant 0.05$).

[A] Assays were performed in triplicate at 25 °C.

biopolymers can often be promoted by introduction of sucrose as cosolute (Evageliou et al., 2000). So when the concentrations of XG and sucrose were both relatively high (0.5% and 30%), the intermolecular association of soluble starch phase and XG perhaps could be promoted, thus the phase separation originated from incompatibility between unlike polysaccharides weakened, and consequently the apparent viscosity decreased.

3.2. In-shear structural recovery measurements

3.2.1. Different pasting conditions on the in-shear structural recovery of WMS pastes

The in-shear structural recovery test was carried out in order to investigate the capability of the pastes to recover their original structure under low shear conditions after decomposition under high-shear conditions (Mezger, 2002). The viscosity profiles as a function of time for the WMS pastes with different pasting conditions are present in Fig. 3. For the paste heated in rheometer (A), the apparent viscosity of the first step was much higher than the other two samples. It is reported by Steeneken (1989) that in dilute paste of starch (8.9%), viscosity is governed by the volume fraction of starch granules. Therefore, to the latter two samples in which most granules dissolved during heating, the apparent viscosity of the first step was much lower.

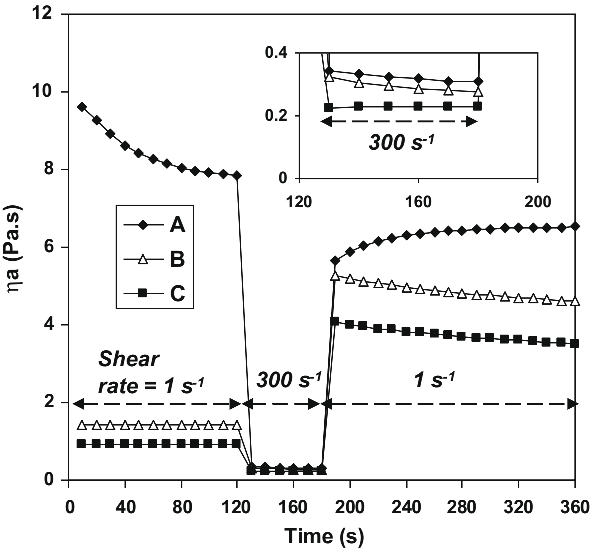

Fig. 3. In-shear structural recovery of the WMS pastes with different pasting conditions measured at 25 °C: (A) pasting in rheometer; (B) pasting in water bath for 6 min; and (C) pasting in water bath for 40 min. The inset shows the detailed section of the high shear stage (300 s^{-1}).

The recovery of the WMS paste heated in rheometer was 0.78 ± 0.05, indicating the initial structure of the paste was damaged during the high-shear condition at the shear rate of 300 s^{-1}, and the new structure formed was less resistant to shear field. Similar recovery value (about 0.5) was reported for the WMS paste heated in the RVA with similar heating program as here in a rheometer (Achayuthakan & Suphantharika, 2008). The recoveries of the WMS pastes heated in water bath for 6 min and 40 min were 3.52 ± 0.04 and 4.08 ± 0.09, respectively, suggesting that the new structures formed after deformation under high-shear conditions were more stable and resistant to shear. The viscosity values of step 3 after high-rate shear increased to 3–4 times compared with step 1, which was in agreement with the anti-thixotropic behavior of the pastes as described before. The recovery value of the WMS paste pasting for 40 min (C) was a litter higher than the one pasting for 6 min (B), which could be explained as follows. As discussed before, the anti-thixotropic behavior could be ascribed to amylopectin in the continuous phase. For the pastes pasting for 40 min, more amylopectin molecules dissolved in water due to longer heating and mixing time, which maybe leading to a more obvious anti-thixotropic behavior, so a higher recovery value can be observed.

3.2.2. In-shear structural recovery measurements of the mixed pastes

The data of the in-shear recovery experiments are given in Table 2. As can be seen from Table 2, the addition of XG could obviously affect the in-shear structural recoveries at a given concentration of sucrose, while sucrose seems had no obvious effect on the recoveries with the concentrations used. To the pastes without sucrose, the recovery values decreased distinctly with the increase of XG content, and such trend still existed with the presence of sucrose. For example, when no sucrose was present, the addition of 0%, 0.25% and 0.5% XG could decrease the recovery values of the pastes from 4.08 ± 0.09 to 1.61 ± 0.00 and 1.14 ± 0.00; when under the sucrose content of 15% and 30%, the recoveries decreased from 4.68 ± 0.07 to 1.59 ± 0.01 and 1.16 ± 0.00, and from 4.44 ± 0.03 to 1.71 ± 0.00 and 1.21 ± 0.00, respectively.

Such results were different from Achayuthakan and Suphantharika (2008) that in-shear structural recovery of the WMS/XG mixed pastes increased with increasing XG concentration. The differences should originate from different heating conditions. In the report of Achayuthakan and Suphantharika (2008), the samples were heated in RVA with similar heating program as here used in a rheometer. Thus, the recovery of the native WMS paste in their report was low (about 0.5) and comparable with the WMS paste heating in the rheometer in this study (about 0.8). Even after the addition of XG, the recoveries of the mixtures in their report were less than

Table 2
In-shear recovery properties of the 5% w/w WMS/XG mixed pastes at mixing ratios of 10/0, 9.5/0.5, 9/1 containing 0%, 15%, and 30% w/w sucrose.[A,B]

XG (%)	Sucrose (%)	Step 1 (Pa s)	Step 2 (Pa s)	Step 3 (Pa s)	Recovery (phase3/phase1)
0	0	0.94 ± 0.03^{a}	0.23 ± 0.00^{a}	3.85 ± 0.03^{a}	4.08 ± 0.09^{a}
0	15	0.82 ± 0.00^{b}	0.29 ± 0.01^{b}	4.03 ± 0.04^{a}	4.68 ± 0.07^{b}
0	30	1.48 ± 0.04^{c}	0.49 ± 0.01^{c}	6.56 ± 0.18^{b}	4.44 ± 0.03^{c}
0.25	0	5.09 ± 0.00^{a}	0.22 ± 0.01^{a}	8.20 ± 0.03^{a}	1.61 ± 0.00^{a}
0.25	15	5.35 ± 0.08^{a}	0.28 ± 0.01^{b}	8.50 ± 0.11^{a}	1.59 ± 0.01^{a}
0.25	30	7.90 ± 0.18^{b}	0.51 ± 0.01^{c}	13.48 ± 0.31^{b}	1.71 ± 0.00^{b}
0.5	0	11.61 ± 0.19^{a}	0.25 ± 0.00^{a}	13.29 ± 0.22^{a}	1.14 ± 0.00^{a}
0.5	15	13.01 ± 0.05^{b}	0.32 ± 0.00^{b}	15.06 ± 0.11^{b}	1.16 ± 0.00^{b}
0.5	30	15.05 ± 0.12^{c}	0.45 ± 0.00^{c}	18.14 ± 0.10^{c}	1.21 ± 0.00^{c}

Mean ± standard deviation values in the same column for each solution followed by different superscripts are significantly different ($p \leqslant 0.05$).

[A] Assays were performed in triplicate at 25 °C.

[B] The program in this test was: step 1, shear rate at 1 s^{-1} for 120 s; step 2, shear rate at 300 s^{-1} for 60 s; step 3, shear rate at 1 s^{-1} for 180 s.

1. Therefore, it seemed the addition of XG played different roles to the in-shear structural properties of the WMS paste when the pasting conditions were different. The reason may be that in the report of Achayuthakan and Suphantharika (2008), the pasting of WMS by RVA was not very sufficient, and still many granule ghosts and fragments remained which determined the in-shear structural recovery properties of the pastes, just like the one heated in the rheometer in this study. When suffered from high-rate shear conditions, the swollen gelatinized granules which were very weak in structure might be sheared into smaller fragments (Wang et al., 2008b), thus the resistance to shear decreased after the high-shear step. However, as an efficient thickener, the addition of XG could reduce the breakage of starch ghosts under high-shear conditions; besides, the rigid XG molecule could promote association of the gelatinized WMS granules efficiently (Abdulmola, Hember, Richardson, & Morris, 1996). So under such conditions, XG stabilized the structures of the mixed pastes, and increased the in-shear structural recoveries.

In this study, since most granules of the pastes dissolved into the continuous phase or broke into smaller fragments based on the heating method (40 min heating in water bath), it is supposed that the continuous phase determined the rheological properties of the WMS paste. Therefore, the reduction of the in-shear structural properties with the addition of XG could be explained as follows. As described before, the anti-thixotropic properties of the pastes should be ascribed to amylopectin, therefore the substitution of XG for WMS decreased the anti-thixotropic behavior of the mixed pastes. However, more important reason should be ascribed to increase in viscosity caused by XG. As can be observed in Table 2, the apparent viscosity of step 1 increased 5–10 times after XG (0.25% and 0.5%) was added, while the viscosity increase caused by anti-thixotropic behavior from step 1 to step 3 changed little. As a result, the in-shear structural recovery of the samples was obviously weakened by XG. Since increase in apparent viscosity of the samples caused by 0.5% XG was bigger than 0.25% XG did, the recovery values of the mixed pastes containing 0.5% XG were smaller than the ones with 0.25% XG.

It could also be observed in Table 2 that the apparent viscosity values at a shear rate of 300 s^{-1} (step 2) of the mixed pastes changed very little with the increase in XG when at a given sucrose concentration. Such results were in agreement with Speers and Tung (1986), who reported the addition of XG could increase low shear-rate viscosity of the fluid foods while having little effect on the viscosity of the food at high shear rate, because of its unique rigid, rod-like conformation. However, when XG content was constant, the increase in sucrose concentration of the samples could increase the apparent viscosity both at high and low shear rate (200 and 1 s^{-1}), as can be seen in Table 2.

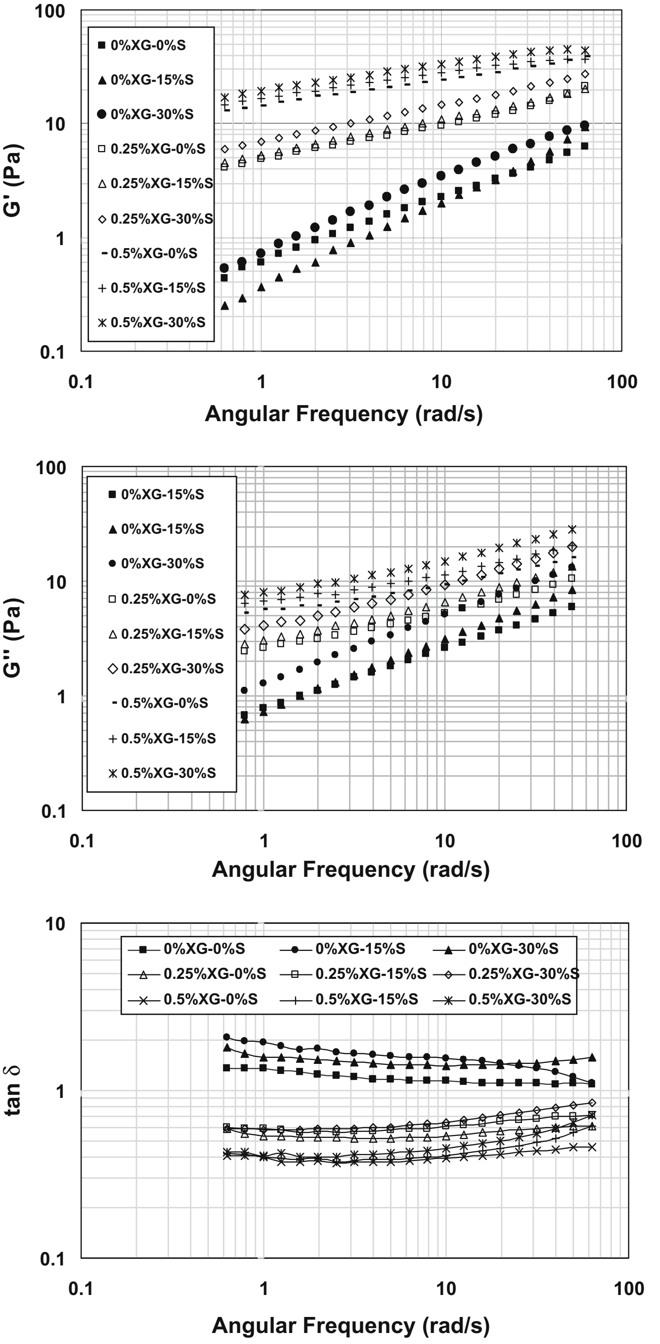

Fig. 4. Variation of G', G'' and tanδ with angular frequency for 5% w/w WMS/XG mixed pastes at mixing ratios of 10/0, 9.5/0.5, and 9/1 containing 0%, 15%, and 30% w/w sucrose. Measurements were made at 25 °C and 1% strain.

3.3. Viscoelastic properties of the mixed pastes

Fig. 4 shows the variation of G', G'', and tanδ as a function of angular frequency for selected WMS/XG/sucrose mixed pastes at different mixing ratios at 25 °C and a strain amplitude of 1.0%. In general, both the addition of XG and sucrose increased G' and G''. As can be seen from Fig. 4A, the G' of WMS samples was increased nearly three orders of magnitude at low XG concentrations, suggesting XG could dominate the elastic properties of the mixtures. Increase in sucrose could also increase the G' of the pastes at a given XG content, but the enhancement in the values of G' caused by the addition of sucrose is very limited compared to the addition of XG. For the WMS/sucrose mixed pastes containing 15% sucrose, the value of G' decreased at low frequencies (0–15 rad/s) and then increased at high frequencies (15–62.8 rad/s) compared with the value of G' for the WMS paste, while the addition of 30% sucrose could increase the value of G' over the whole frequency range. When 0.25% or 0.5% XG was present in the mixtures, the values of both G' and G'' increased with the increasing of sucrose.

The tan δ values of the samples without the addition of XG were all larger than unity in the frequency range used, indicating a liquid-like behavior of the mixtures (Fig. 4C). However, the addition of XG could significantly decrease the tan δ values of the mixtures, whether sucrose was present or not. After the addition of XG (0.25% and 0.5%), all the mixtures displayed a solid-like property with the value of tan δ less than unity, and such property became more significant with the increasing of the XG concentration from 0.25% to 0.5%. It could also be observed that the addition of sucrose increased the value of tan δ at a given XG concentration, suggesting that the addition of sucrose decreased the solid-like properties of the mixed pastes. Such results were different from Pongsawatmanit et al. (2007), who reported the addition of sucrose could decrease the tan δ value of 5% tapioca starch/xyloglucan mixed pastes at mixing ratio of 10/0 and 8/2. Perhaps the reason could be ascribed to different varieties of starches used. The WMS used in this study was amylose-free, so the paste could not form the gel structure as described before (Banks & Greenwood, 1975). The solid-like properties might be strengthened by the addition of sucrose through the association between sucrose and amylose gel-network in the study of Pongsawatmanit et al. (2007).

The frequency dependence of both G' and G'' can be described by the following power law relationship (Rao, 1999; Özkan, Xin, & Chen, 2002):

$$G' = K'\omega^{n'} \tag{2}$$

$$G'' = K''\omega^{n''} \tag{3}$$

where K', and K'' are constants and n', and n'' may be referred to as the frequency exponents, and ω is the angular frequency. The values of K', K'', n', and n'' for the pastes are given in Table 3. As can be seen from Table 3, the addition of XG obviously decreased the

Table 3
Influences of XG and sucrose on n', n'', K', and K'' at 25 °C for 5% w/w WMS/XG mixed pastes at mixing ratios of 10/0, 9.5/0.5, 9/1 containing 0%, 15%, and 30% w/w sucrose.[A]

XG (%)	Sucrose (%)	G'			G''		
		K'	n'	R^2	K''	n''	R^2
0	0	0.60 ± 0.03^a	0.57 ± 0.02^a	0.998	0.78 ± 0.00^a	0.52 ± 0.01^a	1.000
0	15	0.35 ± 0.01^b	0.76 ± 0.02^b	0.997	0.72 ± 0.02^b	0.63 ± 0.01^b	0.999
0	30	0.80 ± 0.04^c	0.62 ± 0.01^a	0.995	1.31 ± 0.01^c	0.59 ± 0.00^c	1.000
0.25	0	4.81 ± 0.09^a	0.32 ± 0.01^a	0.993	2.55 ± 0.04^a	0.34 ± 0.01^a	0.988
0.25	15	5.08 ± 0.17^a	0.33 ± 0.01^a	0.998	2.86 ± 0.05^b	0.38 ± 0.00^b	0.991
0.25	30	7.05 ± 0.05^b	0.33 ± 0.01^a	0.998	3.97 ± 0.04^c	0.40 ± 0.00^c	0.992
0.5	0	14.09 ± 0.34^a	0.24 ± 0.01^a	0.997	5.25 ± 0.07^a	0.27 ± 0.01^a	0.981
0.5	15	17.27 ± 0.32^b	0.20 ± 0.01^b	0.987	6.43 ± 0.08^b	0.28 ± 0.00^b	0.980
0.5	30	19.37 ± 0.47^c	0.23 ± 0.02^{ab}	0.994	7.56 ± 0.07^c	0.32 ± 0.00^c	0.983

Mean ± standard deviation values in the same column for each solution followed by different superscripts are significantly different ($p \leqslant 0.05$).

[A] Assays were performed in triplicate at 25 °C and 1% strain.

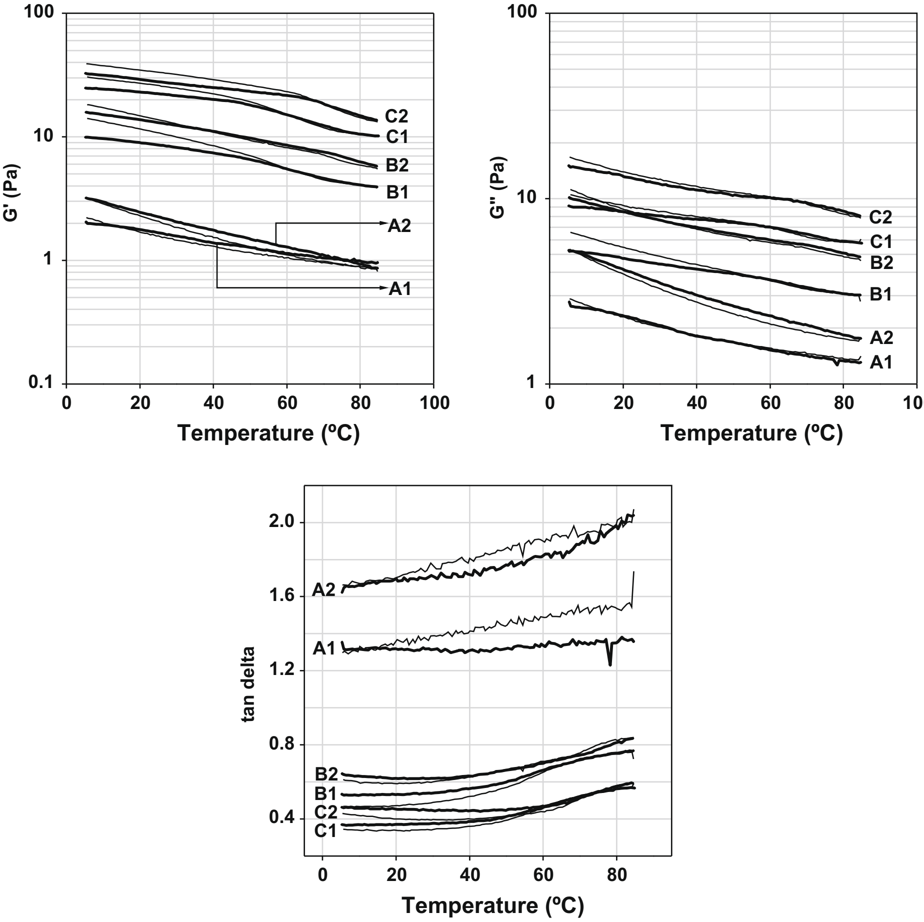

Fig. 5. Temperature dependence of G', G'' and tan δ for 5% w/w WMS/XG mixed pastes at mixing ratios of 10/0, 9.5/0.5, 9/1 containing 0%, 15%, and 30% w/w sucrose. Measurements were made at 1 Hz and 1% strain. A1: 0.0% XG–0.0% Sucrose; A2: 0.0% XG–30.0% Sucrose; B1: 0.25% XG–0.0% Sucrose; B2: 0.25% XG–30.0% Sucrose; C1: 0.50% XG–0.0% Sucrose; C2: 0.50% XG–30.0% Sucrose. Thick solid lines represent heating part and thin solid lines represent cooling part.

n' and n'' values of the samples whether sucrose was present or not, indicating that the values of G' and G'' increased more steadily with frequency with the addition of XG. Obviously, the values of K' and K'' also increased with the addition of XG, which could be ascribed to an increase in the stiffness of the continuous phase of the composite systems due to the thickening effect of XG (Alloncle & Doublier, 1991). Increase in the concentration of sucrose increased the values of n'', but had little effect on the values of n' at a given XG content, suggesting that the addition of sucrose decreased the solid-like properties and stabilities of the mixed pastes.

3.4. Thermostable properties of the mixed pastes

The temperature dependences of G', G'', and $\tan\delta$ of the paste mixtures are presented in Fig. 5 (the rheograms of the paste mixtures containing 15% sucrose were not given for clear illustration). The WMS paste without the addition of XG and sucrose (A1) and the WMS paste with 30% sucrose (A2) showed a liquid-like behavior since the G'' was greater than G' for these pastes (i.e., the values of $\tan\delta$ were greater than unity for these pastes (A1 and A2)). Both the G' and G'' decreased with the increasing of temperature for the A1 and A2. For the (A1), no hysteresis was observed for G'' in the consequent cooling procedure, while G' decreased only slightly.

Compared with the samples containing no XG (A1 and A2), the values of G' and G'' increased and the value of $\tan\delta$ decreased significantly at the same temperature after the addition of XG (i.e., for the samples B1 (0.25% XG-0.0% Sucrose), B2 (0.25% XG-30.0% Sucrose), C1 (0.5% XG-0.0% Sucrose), and C2 (0.5% XG-30.0% Sucrose)). These observations suggest that these mixtures became stiffer and more solid like material with the addition of XG. During the cooling procedure of the samples B1, B2, C1, and C2, both the G' and G'' became larger and $\tan\delta$ became smaller, which may indicate a strengthened thickening effect of XG during cooling. It is reported phase separation and exclusion effects between dislike polysaccharides could contribute to enhancements in the viscoelastic properties of the starch/hydrocolloid blends (Mandala, Michon, & Launay, 2004), while promotion of intergranular association by hydrocolloid has also been proposed (Abdulmola et al., 1996). Since most WMS granules dissolved during pasting based on the heating method adopted, it is possible that phase separation and exclusion effects of the WMS/XG mixtures became more prominent during cooling procedure, thus enhancement in viscoelastic property of the mixtures occurred. After the addition of sucrose to the WMS/XG mixtures, both the G' and G'' of the paste samples (B2 and C2) were increased but $\tan\delta$ was decreased, suggesting that even though the overall stiffness of the paste samples was increased with the addition of sucrose, the paste samples B2 and C2 became more liquid-like material compared to B1 and C1, respectively. During the cooling phase, the increment observed in the G' and G'' of the samples B2 and C2 (i.e., the samples with 30% sucrose addition) were slightly less compared to the samples without sucrose addition (B1 and C1). These observations suggest that the addition of XG could improve and dominate the thermostable properties of the paste mixtures, while the decrease in thermal stability caused by sucrose addition was comparatively small.

4. Conclusions

Sufficient pasting of the WMS suspensions could contribute to the anti-thixotropic properties of the WMS paste during steady flow and in-shear recovery experiments, which could be attributed to the formation of new structure of the paste during shear conditions. The addition of XG could help to the formation of new structure of the mixed pastes, while it also decreased the anti-thixotropic properties of the mixtures during the in-shear recovery tests. Both the addition of XG and sucrose increased the apparent viscosity and moduli (G', G'') of the mixtures. However, the addition of sucrose decreased the solid-like properties of the mixtures, while XG played an opposite role. XG could improve and dominate the thermostable properties of the mixtures, while sucrose decreased thermal stability of the mixtures slightly.

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