

Dongryel Yoo  
Byoungseung Yoo

Department of Food Science  
and Technology,  
Dongguk University,  
Seoul, Korea

## Rheology of Rice Starch-Sucrose Composites

The effect of sucrose at different concentrations (0, 10, 20 and 30%) on rheological properties of rice starch pastes (5% w/w) was investigated in steady and dynamic shear. The steady shear properties of rice starch-sucrose composites were determined from rheological parameters for power law and Casson flow models. At 25°C all the starch-sucrose composites exhibited a shear-thinning flow behavior ( $n=0.25\text{--}0.44$ ). The presence of sucrose resulted in the decrease in consistency index ( $K$ ), apparent viscosity ( $\eta_{a,100}$ ) and yield stress ( $\sigma_{oc}$ ). Dynamic frequency sweeps at 25°C indicated that starch-sucrose composites exhibited weak gel-like behavior with storage moduli ( $G'$ ) higher than loss moduli ( $G''$ ).  $G'$  and  $G''$  values decreased with the increase in sucrose concentration. The dynamic ( $\eta^*$ ) and steady-shear ( $\eta_a$ ) viscosities at various sucrose concentrations did not follow the Cox-Merz superposition rule.  $G'$  values as a function of aging time (10 h) at 4°C showed a pseudoplateau region at long aging times. In general, the values of  $G'$  and  $G''$  in rice starch-sucrose composites were reduced in the presence of sucrose and depended on sucrose concentration.

**Keywords:** Rice starch; Rheological property; Viscosity; Storage modulus; Loss modulus; Cox-Merz rule

### 1 Introduction

Starch is unique among polysaccharides because it occurs naturally as discrete particles, called granules [1]. Heating aqueous starch granules above the gelatinization temperature causes starch to paste. A starch paste can be regarded as a composite material consisting of swollen granules dispersed in a continuous biopolymer matrix [2]. Therefore, the properties of the dispersed phase, the continuous phase, and interactions between the components are important considerations for insights into the rheological properties of starch pastes [3]. In general, the rheological properties of starch pastes depend on several factors, including the type of starch, amylose/amylopectin ratio, temperature, starch concentration, pH, and the presence and concentration of solutes, such as salts, lipids, proteins and sugars.

Sugars have been known to significantly affect the rheological properties of starch pastes. Several researchers have studied the effect of sugars on rheological properties of various native starch pastes, such as corn [4], wheat [5], sago [6] and potato starch [7], from dynamic rheological parameters which are related to the sample's molecular structure. They found that the rheological

properties of starch-sugar composites depended on the type of sugar, sugar concentration and the nature of starch.

Rice is the main staple food of Oriental nations. Their traditional foods, in general, are prepared from rice and rice-related materials [8]. Therefore, rheological approaches to rice starch and rice flour have also been studied by several researchers [8–12]. Although extensive literature is available on the rheological properties of rice starch pastes, there is little information on the effect of sucrose concentration on rheological properties of rice starch pastes under both steady and dynamic shear. In particular, no attempt has been made to study rheological properties of rice starch-sucrose composites in the aging process, especially from dynamic moduli, using small deformation dynamic oscillatory rheometry. The dynamic oscillatory rheometry is useful in monitoring structure development during aging without breaking structural elements formed in the sample upon aging. In the rice starch-sucrose composite system, understanding the component interactions is also important for improving the rheological properties and storage stability of rice starch-based products. Therefore, the main objective of this study was to determine the rheological properties of rice starch pastes with different sucrose concentrations in both steady and dynamic shear, and the effect of sucrose concentration on rheological properties of rice starch pastes.

**Correspondence:** Byoungseung Yoo, Department of Food Science and Technology, Dongguk University, 3 Pil-dong, Chung-gu, Seoul 100-715, Korea. Phone: +82-2-2260-3368, Fax: +82-2-2264-3368, e-mail: bsyoo@dongguk.edu.

## 2 Materials and Methods

### 2.1 Materials

Rice starch was purchased from Sigma Co. (St. Louis, MO, USA). Sucrose used in this study was purchased from Yakuri Chemicals Co. (Kyoto, Japan).

### 2.2 Preparation of starch pastes

Starch dispersions (5%, w/w) were prepared by mixing starch with distilled water and sucrose to obtain 10, 20 and 30% (weight basis) sugar levels. A dispersion with no added sugar (control) was also prepared. The starch-sucrose dispersion was moderately stirred for 1 h at room temperature, and then was heated at 95°C in a water bath for 30 min with mild agitation provided by a magnetic stirrer, as described by Chang et al. [4]. At the end of the heating period, the hot starch paste was immediately transferred to the rheometer plate for the measurement of rheological properties.

### 2.3 Rheological measurements

Steady shear properties were obtained at 25°C with a rheometer (AR 1000, TA Instruments, New Castle, DE, USA) using a parallel plate system (4 cm dia.) at a gap of 500 µm. For steady-shear measurements, the sample was loaded onto the platen of the rheometer and the exposed sample edge was covered with a thin layer of light paraffin oil to prevent evaporation or absorption of atmospheric moisture during measurements. The sample was sheared continuously from 0.4 to 500 s<sup>-1</sup>. To analyze time-dependent flow behavior, the starch pastes were sheared first in ascending order (0.4–500 s<sup>-1</sup>) and then in descending order.

In order to describe the steady-shear rheological properties of samples, the data were fitted to the well-known power law (Eq. 1) and Casson (Eq. 2) models:

$$\sigma = K\dot{\gamma}^n \quad (1)$$

$$\sigma^{0.5} = K_{oc} + K_c\dot{\gamma}^{0.5} \quad (2)$$

where  $\sigma$  is the shear stress (Pa),  $\dot{\gamma}$  is the shear rate (s<sup>-1</sup>),  $K$  is the consistency index (Pa·s<sup>*n*</sup>),  $n$  is the flow-behavior index (dimensionless), and  $(K_c)^2$  is the Casson plastic viscosity ( $\eta_c$ ). Casson yield stress ( $\sigma_{oc}$ ) according to the Casson model (Eq. 2) was determined as the square of the intercept ( $K_{oc}$ ), that was obtained from linear regression of the square roots of shear rate-shear stress data. Using magnitudes of  $K$  and  $n$ , the apparent viscosity ( $\eta_{a,100}$ ) at 100 s<sup>-1</sup> was calculated.

Dynamic-shear properties were obtained from frequency sweeps over the range of 0.63–63 rad/s at 3% strain. The 3% strain was in the linear viscoelastic region. Frequency sweep tests were also performed at 25°C. TA rheometer Data Analysis software (version VI. 1.76) was used to obtain the experimental data and to calculate storage modulus ( $G'$ ), loss modulus ( $G''$ ) and complex viscosity ( $\eta^*$ ).

In order to investigate the changes in  $G'$  during aging, the sample was kept at 4°C for 10 h. In order to relax the samples before the measurement in the aging processes, all samples were allowed to rest at the initial temperatures (4°C) for 5 min. Storage ( $G'$ ) and loss moduli ( $G''$ ) were monitored during aging at 1 Hz and 3% strain, and were also measured at the end of the aging period from frequency sweeps over the range of 0.1–10 Hz at 3% strain. The 3% strain was in the linear viscoelastic region. Rheological measurements in steady and dynamic shear were performed in duplicate. Results reported were an average of the two measurements.

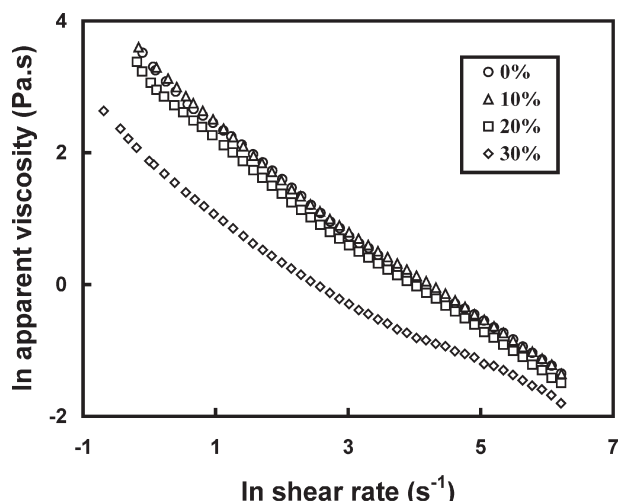
### 2.4 Swelling power

Starch dispersions (0.5%, w/w) were prepared by mixing starch with distilled water and sucrose to obtain 0, 10, 20 and 30% (weight basis) sugar levels. The starch-sucrose dispersion was moderately stirred for 1 h at room temperature, and then was heated at 95°C in a water bath for 30 min, as described previously. The hot starch paste was cooled to room temperature in an iced water bath and centrifuged at 8000 × g for 20 min. The supernatant was decanted and the swelling power was determined as the ratio of weight of sediment to weight of dry starch [13]. No correction for solubles was made due to the high sugar concentrations used. Results reported were an average of three measurements.

## 3 Results and Discussion

### 3.1 Steady-shear properties

The viscosity flow curves of rice starch-sucrose composites for the relationship between shear rate and apparent viscosity at different sucrose concentrations (0, 10, 20 and 30%) are shown in Fig. 1. The apparent viscosity values decreased with the increase in shear rate, indicating that rice starch pastes are non-Newtonian fluids. Therefore, the steady-shear properties of rice starch-sucrose composites were determined from rheological parameters for the power law and Casson flow models used in the present study. The steady-shear rheological data (Tab. 1) show the magnitudes of all parameters ( $\eta_{a,100}$ ,  $K$ ,  $n$  and  $\sigma_{oc}$ ) obtained from flow models. All rice



**Fig. 1.** Viscosity flow curves at 25°C of rice starch pastes (5%, w/w) with different sucrose concentrations.

**Tab. 1.** Effect of sucrose concentration on steady-shear rheological properties of rice starch-sucrose composites at 25°C.

Sucrose concentration [%]	Apparent viscosity, $\eta_{a,100}$ [Pa·s]	Consistency index, $K$ [Pa·s <sup><i>n</i></sup> ]	Flow behavior index, $n$ [–]	Yield stress $\sigma_{oc}$ [Pa]
0 (Control)	0.74	23.3	0.25	27.8
10	0.74	22.5	0.26	27.3
20	0.68	18.9	0.28	22.8
30	0.32	4.11	0.44	7.94

starch-sucrose composites had shear-thinning behavior with values of flow behavior index ( $n = 0.25$ – $0.44$ ). The  $n$  values increased with the increase in sucrose concentration. This shear-thinning behavior can be explained by breaking of an entangled polysaccharide molecule network during shearing, as described by *Bhandari et al.* [14] and *Morris* [15]. In an entangled network system, the rate of disruption of existing intermolecular entanglements becomes greater than the rate of reformation of intermolecular entanglement with increasing shear rate, resulting in less resistance to flow and a lower apparent viscosity. All rice starch pastes showed low magnitudes of  $\sigma_{oc}$  in the range of 7.94–27.8 Pa. The magnitudes of apparent viscosity ( $\eta_{a,100}$ ), consistency index ( $K$ ) and Casson yield stress ( $\sigma_{oc}$ ) obtained from flow models decreased with the increase in sucrose concentration. There also was little change in the magnitudes of rheological parameters ( $\eta_{a,100}$ ,  $K$ ,  $n$  and  $\sigma_{oc}$ ) between control (0%) and 10% sucrose concentrations, and an essential change in those in the concentration range of 10–30%. In particular, the rice starch-sucrose composite at 30%

concentration showed the greatest reduction in values of  $\eta_{a,100}$ ,  $K$  and  $\sigma_{oc}$ . This tendency is in reasonably good agreement with that found in previous studies of corn starch-sucrose composites [16]. From these results, it was concluded that the rice starch-sucrose composites had a shear-thinning behavior with small magnitudes of yield stresses, and that their steady-shear properties were apparently influenced by sucrose concentration.

### 3.2 Time-dependent flow behavior

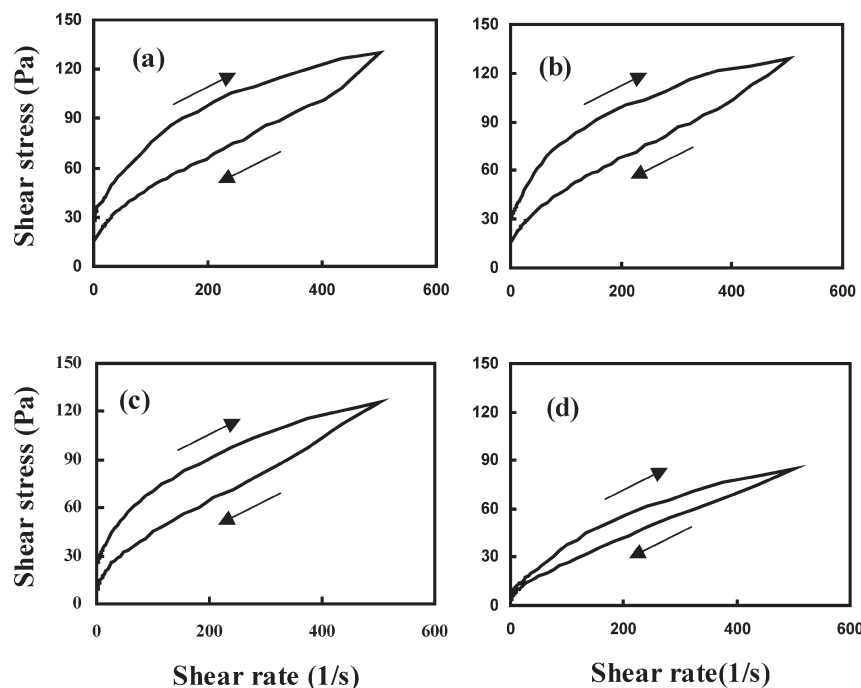
As shown in Fig. 2, a thixotropic behavior was observed in all shear stress-shear rate curves obtained by first increasing and then decreasing shear rates in the range of 0.4–500 s<sup>–1</sup>. If the sample is shear sensitive, the two flow curves do not coincide, thus causing a hysteresis loop. Classical approach to characterizing structural breakdown is the measurement of the hysteresis loop, as noted by *Weltman* [17]. The area enclosed by the hysteresis loop is an indication of the degree of structural breakdown due to shearing. There were not much differences between the loop areas in the concentration range of 0–20%, and an essential decrease in the loop area at 30% concentration. The loop area at sucrose concentrations of 30% was also smaller than those in the concentration range of 0–20%. Such small loop area means that there is a low degree of structural breakdown during shearing. Similar trends were also observed for corn starch-sucrose composites [16]. Therefore, it was concluded that the structure loss of rice starch-sucrose composites could be reduced in the presence of high sucrose concentrations (>20%).

### 3.3 Dynamic shear properties

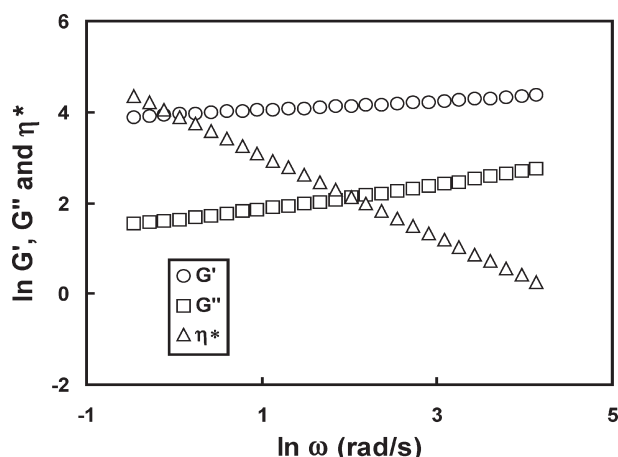
Fig. 3 shows changes in storage modulus ( $G'$ ), loss modulus ( $G''$ ) and complex viscosity ( $\eta^*$ ) as a function of the frequency ( $\omega$ ) for a typical starch dispersion (i.e. 10% sucrose) at 25°C. The magnitudes of  $G'$  and  $G''$  increased with increase in  $\omega$ .  $\ln \eta^*$  versus  $\ln \omega$  plots also showed shear-thinning power law characteristics. This tendency is in good agreement with those found for other macromolecule-sucrose composites, such as corn starch-sucrose [16], pectin-sucrose [18] and gelatin-sucrose composites [19]. In the rice starch-sucrose composite systems, the dynamic rheological data of  $\ln (G', G'')$  versus  $\ln \omega$  were also subjected to linear regression and Tab. 2 contains the magnitudes of slopes ( $n'$  and  $n''$ ), intercepts ( $K'$  and  $K''$ ) and  $R^2$  in the following equations:

$$G' = K' (\omega)^{n'} \quad (3)$$

$$G'' = K'' (\omega)^{n''} \quad (4)$$



**Fig. 2.** Thixotropic flow curves for rice starch pastes (5%, w/w) with different sucrose concentrations at 25°C: (a) 0%, (b) 10%, (c) 20%, (d) 30%.



**Fig. 3.**  $\ln (G', G'', \eta^*)$  versus  $\ln \omega$  of a rice starch paste (5%, w/w) at 10% sucrose concentration, 25°C.

**Tab. 2.** Slopes ( $n'$ ,  $n''$ ) and intercepts ( $K'$ ,  $K''$ ) of  $\ln (G', G'')$  versus  $\ln \omega$  (frequency, rad/s) data of rice starch-sucrose composites with different sucrose concentration [%] at 25°C.

Sucrose conc. [%]	$G'$			$G''$		
	$n'$	$K'$	$R^2$	$n''$	$K''$	$R^2$
0 (Control)	0.10	52.2	0.99	0.28	5.08	0.99
10	0.11	51.2	0.98	0.28	5.01	0.99
20	0.12	39.1	0.99	0.31	4.73	0.99
30	0.16	16.4	0.97	0.52	2.12	0.99

The magnitudes of  $K'$  and  $K''$  decreased with the increase in sucrose concentration, and the values of  $G'$  were about an order of magnitudes higher than those of  $G''$ . Such dependency on sucrose concentration can be explained by recognizing that the presence of sucrose reduces the amount of amylose, that is leached out of the granules during gelatinization, as suggested by *Ahmad and Williams* [20]. *Prokopowich and Biliaderis* [21] also found that such reduction of dynamic moduli values in the presence of sucrose was due to the inhibition of chain reorganization in starches. Because of the strong dependence of  $G'$  and  $G''$  on  $\omega$  with the positive slopes ( $n'=0.10$ – $0.16$ ;  $n''=0.28$ – $0.52$ ) and the magnitudes of  $K'$  (16.4–52.2) much higher than those of  $K''$  (2.12–5.08) (Tab. 2), all rice starch-sucrose composite samples exhibited behavior common to weak gels. While there was a substantial difference between the intercepts ( $K'$  and  $K''$ ) of 10 and 20% sucrose concentrations, the difference was much less between starch pastes of 0% and 10% sucrose concentrations (Tab. 2). The  $K'$  value of low sucrose concentrations (0–20%) was much greater than  $K''$  as compared to a starch paste at 30% sucrose concentration. These results show that the elastic properties of rice starch pastes can be decreased at higher sucrose concentrations due to the decrease in the rate of chain rearrangement, that tends to form a more ordered structure or crystalline structure, as indicated by *Ahmad and Williams* [20] and *Lee et al.* [22]. A similar trend was reported with corn starch-sucrose composite pastes [4, 16]. Such greater decrease in elastic properties at higher sucrose concentrations (20 and 30%)

can also be attributed to the decrease of swelling of starch granules, as shown in Tab. 3. The swelling power values of rice starch-sucrose composites with different sucrose concentrations were reduced at higher sucrose concentrations ( $\geq 20\%$ ). *Li* and *Yeh* [23] showed that low swelling starch yielded a lower peak storage modulus ( $G'_{\max}$ ) in dynamic mechanical analysis. *Savage* and *Osman* [24] and *Hoover* and *Senanayake* [25] also found that the effect on swelling power of starch depended on sugar concentration and a decrease of swelling power was observed at higher concentration ( $>20\%$ ). From these results, dynamic rheological properties in the rice starch-sucrose composite systems appear to depend on the concentration of sucrose, and to be affected by the rate of chain rearrangement and the swelling power of starch granule.

**Tab. 3.** Swelling power at 95°C as a function of sucrose concentration for 0.5% rice starch.

Sucrose concentration [%]	Swelling power [g/g]
0 (Control)	17.2
10	18.0
20	16.1
30	15.6

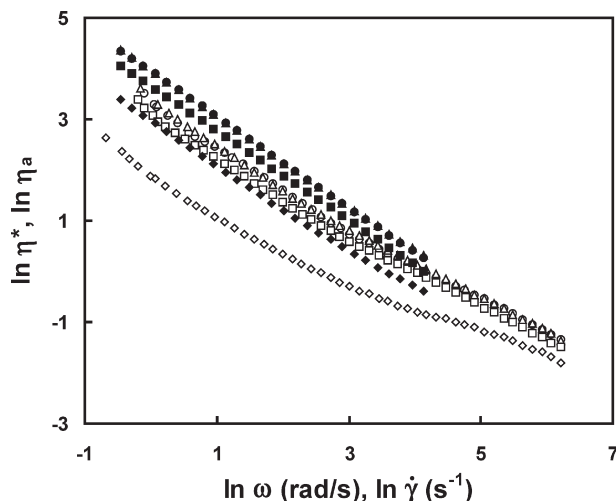
### 3.4 Applicability of Cox-Merz rule

*Cox* and *Merz* [26] proposed an empirical correlation between the apparent viscosity ( $\eta_a$ ) of polymers as a function of shear rate ( $\dot{\gamma}$ ) and the complex dynamic viscosity ( $\eta^*$ ) as a function of frequency ( $\omega$ ):

$$\eta^*(\omega) = \eta_a(\dot{\gamma})|_{\omega = \dot{\gamma}} \quad (5)$$

The Cox-Merz rule (Eq. 5) is an empirical relationship that equates  $\eta^*$  measured with dynamic oscillatory rheometry to  $\eta_a$  measured in shear flow, where  $\omega$  is taken as  $\dot{\gamma}$  in the oscillatory tests. The Cox-Merz superposition rule has been found to apply to a number of starch pastes [16, 27–29]. When the Cox-Merz rule is followed, it may be used to predict the steady-shear properties of a material from the dynamic rheological properties obtained without extensive alteration of structures [28].

In order to examine the applicability of the Cox-Merz superposition rule,  $\eta_a$  and  $\eta^*$  of rice starch-sucrose composites are plotted versus  $\dot{\gamma}$  and  $\omega$ , respectively (Fig. 4). It was observed that the magnitudes of  $\eta^*$  were higher than those of  $\eta_a$ , as is mostly the case, because most departures from the Cox-Merz rule can be attributed to structure decay due to the effect of the strain deformation applied to the system (i.e. low in oscillatory shear and high in steady shear) [29]. Therefore, the Cox-Merz rule was



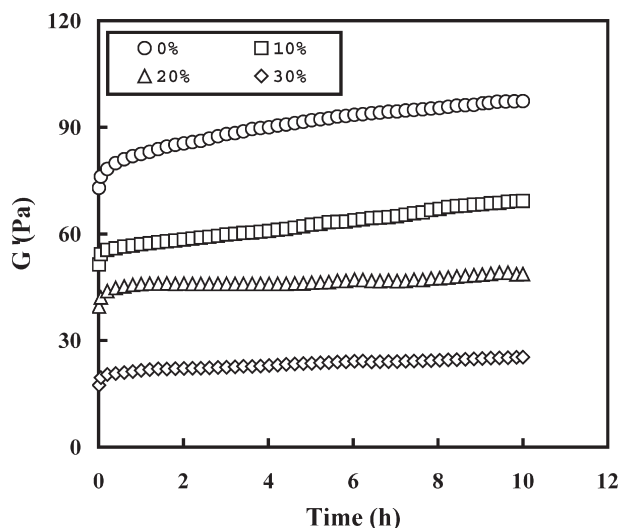
**Fig. 4.** Comparison of steady flow viscosity ( $\eta_a$ ) and complex viscosity ( $\eta^*$ ) for rice starch pastes (5%, w/w) with different sucrose concentrations at 25°C: 0% sucrose (●, ○), 10% sucrose (▲, □), 20% sucrose (■, □), 30% sucrose (◆, ◇). closed symbol:  $\eta^*$ , open symbol:  $\eta_a$ .

not applicable to rice starch-sucrose composites, but the dynamic viscosity provided reasonable estimates of the apparent viscosity. This behavior is in good agreement with those found for corn starch-sucrose composites [16], modified corn starch pastes [27, 29], and pectin-sucrose composites [18]. Such failure of the Cox-Merz rule to correlate the steady and dynamic shear data also reflects the weak structure build-up of the rice starch-sucrose composite samples.

### 3.5 Effect of sucrose concentration on $G'$ during aging

Fig. 5 shows changes in storage moduli ( $G'$ ) as a function of aging time (10 h) for rice starch-sugar composites at 4°C. From the time-dependent curves of all samples except for 10% sucrose, it was obvious that  $G'$  values increased rapidly with time for the first few hours of aging. Later, the increase of  $G'$  became slower until a pseudo-plateau value is approached at long aging, indicating that the aggregation speed for amylopectin appears to be slowed down by the addition of sucrose. Therefore, the changes in  $G'$  on aging of starch has been suggested to consist of two different rates of structure development. The rapid increase and pseudoplateau of  $G'$  can be due to the rapid aggregation of amylose chains at the early stage and the slow aggregation of amylopectin chains at long-term storage, respectively, as described by several researchers [4, 5, 21]. This trend was reported with other starch pastes containing sucrose [16, 20, 30]. The pseudoplateau values of  $G'$  after a long aging time were





**Fig. 5.** Changes in  $G'$  during aging at 4°C for 10 h for rice starch-sucrose composites as a function of sucrose concentration.

reduced by the sucrose addition, and decreased with the increase in sucrose concentration. Such reduction of  $G'$  values in the presence of sucrose appeared to be due to the prevention of molecular chain rearrangement [20, 22]. Therefore, the effect of sucrose on changes in  $G'$  values of rice starch-sucrose composite systems appears to depend on the sucrose concentration.

According to Evageliou et al. [30], the stability in starch-sugar composite systems during aging are commonly attributed to modification of water structure, with the degree of modification induced by small carbohydrate solutes (sugars) being determined by their steric compatibility with the pattern of hydrogen bonding between water molecules. From the various studies based on rheological and thermal properties of starch-sugar composites, it is also obvious that the retrogradation in the composite system is related to the compatibility of the solute with the water structure [20, 21, 30–33]. Katsuta et al. [32, 33] found that the viscosity of bulk water around the sugars increased due to the decrease of the thermal motion of the stabilized water in the starch-water-sugar systems, and thus the motion of the starch chain was suppressed by sugars, indicating that the rearrangements of starch chains were inhibited. Prokopowich and Biliaderis [21] and Biliaderis and Prokopowich [31] also suggested that sugars, which fit well in the hydrogen bonded structure of water, have a stabilizing effect on polymer chains, thus retarding chain reordering occurred during aging. This indicates that the compatible sugars, that greatly fit the structure of water in starch, result in the inhibition of retrogradation. Therefore, in our study the

observed effect of sucrose on the retrogradation of rice starch paste during aging can be explained by the compatibility of sucrose with the water structure.

Tab. 4 also shows the magnitudes of slopes ( $n'$  and  $n''$ ) and intercepts ( $K'$  and  $K''$ ) from plot of  $\ln \omega$  versus  $\ln G'$  and  $\ln G''$  at the end of aging at 4°C, as described earlier using Equations (3) and (4). From these dynamic rheological data,  $K'$  values are always much greater than those of  $K''$ , showing an independency ( $n' < 0.1$ ) on frequency as characteristic of an elastic network. This indicates that the rice starch-sucrose composites at the end of aging at 4°C display true gel-like behavior in comparison with those at 25°C (Tab. 2). This tendency is similar to those found in corn starch-sucrose composites [4] after 10 h aging at 4°C. A reduction in  $K'$  and  $K''$  values in the presence of sucrose was observed with the predominantly elastic character ( $K' > 10 K''$ ). The magnitudes of  $K'$  and  $K''$  also decreased with the increase in sucrose concentration. Such sugar concentration dependence can be explained by recognizing that as the sucrose concentration increases, the rate of chain rearrangement decreases, resulting in the inhibition of retrogradation [20, 21, 31, 33, 34]. From these results, it was concluded that the changes in dynamic moduli due to the crystallization during aging greatly depended on the sucrose concentration.

**Tab. 4.** Slopes ( $n'$ ,  $n''$ ) and intercepts ( $K'$ ,  $K''$ ) of  $\ln(G', G'')$  versus  $\ln \omega$  (frequency, rad/s) data of rice starch-sucrose composites with different sucrose concentration [%] after aging for 10 h at 4°C.

Sucrose conc. [%]	$G'$			$G''$		
	$n'$	$K'$	$R^2$	$n''$	$K''$	$R^2$
0 (Control)	0.06	97.5	0.99	0.26	6.09	0.99
10	0.06	70.8	0.99	0.29	4.82	0.99
20	0.07	48.1	0.99	0.35	3.79	0.99
30	0.10	21.4	0.97	0.51	2.20	0.99

## 4 Conclusions

Rice starch pastes (5%, w/w) with different sucrose concentrations (0–30%) were shear-thinning fluids with a yield stress. Steady-shear properties of rice starch-sucrose composites apparently depended on sucrose concentration. The rice starch paste at 30% sucrose concentration was found to show the greatest reduction in magnitudes of rheological parameters ( $\eta_{a,100}$ ,  $K$  and  $\sigma_{oc}$ ). Rice starch-sucrose composites at sucrose concentrations of 0, 10 and 20% displayed a highly thixotropic behavior as compared to that at sucrose concentrations of 30%, indicating that there was a low degree of structural breakdown during shearing at high

sucrose concentration. The storage modulus ( $G'$ ) dominated the loss modulus ( $G''$ ) for all rice starch-sucrose composites with positive slopes at 25°C, indicating that the rice starch-sucrose composites exhibited rheological behaviors similar to those of weak gels. The Cox-Merz superposition rule was not applicable to rice starch pastes with different sucrose concentrations, indicating that the complex viscosity ( $\eta^*$ ) values were higher than those of the apparent viscosity ( $\eta_a$ ). The values of  $G'$  during aging for 10 h at 4°C were reduced in the presence of sucrose, indicating that the sucrose inhibited retrogradation. The rice starch-sucrose composites at the end of aging at 4°C also displayed true gel-like behavior, with  $G'$  much greater than  $G''$ , being independent on frequency. The inhibition of retrogradation in the starch-sucrose composite system can be attributed to the compatibility of sucrose with the water structure. In the rice starch-sucrose composite system, the changes in  $G'$  values due to the crystallization during aging greatly depended on the sucrose concentration.

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