

Steady and Dynamic Shear Rheological Properties of Starch and Decolorized *Hsian-tsao* Leaf Gum Composite Systems

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ABSTRACT

Cereal Chem. 79(1):58–63

Rapid Visco Analyser (RVA) and dynamic rheological measurements were performed on a mixture of decolorized *hsian-tsao* leaf gum (dHG) and starch as a function of starch-to-gum ratio (S/G 0:6, 1:5, 2:4, 3:3, 4:2, 5:1, and 6:0), starch type (wheat, corn, and tapioca), and total solid content (2, 3, and 4%). Under S/G of 5:1, 4:2 and 3:3, dHG interacted with starch synergistically, which resulted in a marked increase in viscosity during cooling. The storage modulus of the resulting mixed gel was higher than the loss modulus, and both moduli were almost frequency-

independent throughout the frequency range tested, indicating that the composite gels could be classified rheologically as elastic gels. Mixed systems with tapioca starch showed higher pseudo-gel viscosity in the RVA test, but lower storage modulus in the dynamic test than those with wheat or corn starch. Such results implied that tapioca starch contributed a more viscous property but wheat and corn starch contributed a more elastic property to the mixed systems.

Hsian-tsao (*Mesona procumbens* Hemsl) is one of the edible plants containing polysaccharide gum. Lai et al (2001) report that crude *hsian-tsao* leaf gum is a potent polar antioxidant that interacts with a wide range of species directly responsible for oxidative damage. The unique aroma and health benefits of *hsian-tsao* make it quite popular among the Chinese. *Hsian-tsao* gum has been reported as an ionic heteroglycan consisting of galactose, glucose, rhamnose, arabinose, and uronic acid at a ratio of $\approx 2:1:1:1:2$ (Yang and Huang 1990). As compared with other commercial gums, *hsian-tsao* gum formed a low-viscosity solution with a pronounced shear-thinning characteristic (Tung 1998). Moreover, due to its ionic nature, the solution properties of *hsian-tsao* gum alone are strongly affected by salts (Lai et al 2000). *Hsian-tsao* gum reportedly interacts strongly with nonwaxy type starch without any ionic side group (Lii and Chen 1980) and forms resilient gels. The gelling behavior of *hsian-tsao* gum with starch was affected by factors such as extraction conditions, parts of plants, ionic strength, etc. (Lii and Chen 1980; Lai et al 2000). Through rheological and textural and thermal studies of *hsian-tsao* leaf gum and starch composite systems, Lai and Chao (2000a,b) and Chao and Lai (1999a–c) reported that the synergistic effect of starch and HG mixtures was enhanced with higher ionic strength.

Crude *hsian-tsao* gum is dark brown to black in color, which limits applications in foods. The dark color of *hsian-tsao* gum could be successfully removed using active carbon (Yang et al 1987; Chen et al 1996; Liao and Lai 2001). Yang et al (1987) reported that the gel strength of decolorized *hsian-tsao* gum and starch composite gel is higher than that of crude gum and starch composite gel under relevant concentrations. Liao and Lai (2001) find that decolorization treatment significantly reduces the protein content of crude gum. Significant exothermic and endothermic differential scanning calorimetry (DSC) peaks during cooling and heating were found with S/G 5:1, 4:2, and 3:3, which indicates that decolorized *hsian-tsao* leaf gum (dHG) interacts with starch synergistically. The optimum S/G depends on the unique chemistry of each starch.

Though investigators such as Yang et al (1987) Chen et al (1996) and Liao and Lai (2001) initiated research on the physical properties of dHG, systematic studies on the pasting and gelling behavior of starch and dHG mixed systems and the rheological characterization of the resulting composite gels are quite limited. Therefore, the objective of this research was to examine the rheological properties

of dHG and starch composite systems as a function of S/G, starch types, and total solids content using a Rapid Visco Analyser (RVA) and dynamic rheological measurements.

MATERIALS AND METHODS

Materials

The dried *hsian-tsao* leaves were purchased from a contracted farmer. Starches were kindly supplied by a local ingredient company (Ku-Tung Inc., Taiwan).

Extraction and Decolorization of Polysaccharide Gum

Extraction of polysaccharide gum from *hsian-tsao* leaves was performed using the method of Lai and Chao (2000a,b). *Hsian-tsao* leaves (4% dry matter) in a sodium bicarbonate solution (0.14M) were refluxed at 95°C for 4 hr. Decolorization of crude *hsian-tsao* leaf gum was performed using the method of Liao and Lai (2001). The hot polysaccharide extract of *hsian-tsao* leaves was treated with active carbon (10, 5, and 2%) followed by centrifugation (7,000 \times g, 10min) to remove active carbon consecutively. The decolorized polysaccharide gum in the extract was then precipitated with 70% ethanol, centrifuged (7,000 \times g, 15 min), and vacuum-dried (40°C, 48 hr).

Chemical Composition of dHG and Starches

The proximate compositions of dHG and starches were analyzed using an official method (AOAC 1995). Uronic acid content of dHG was analyzed using the modified carbazole assay (Bitter and Muir 1962). The amylose content of starches was analyzed using the method of Sowbhagya and Bhattacharya (Sowbhagya et al 1971).

RVA

The pasting and gelling behavior of starch and dHG mixed systems was measured using an RVA (model RVA-3C, Newport Scientific Co., Australia). Samples were prepared by mixing various concentrations of wheat, corn, or tapioca starch with dHG to S/G 0:6, 1:5, 2:4, 3:3, 4:2, 5:1, and 6:0 in 0.14M sodium bicarbonate to make a total sample size of 28 g. The mixture was first stirred at 960 rpm for 10 sec, and then 160 rpm for the remainder of the test. The temperature profile included a heating and cooling cycle. During heating, samples were kept at 50°C for 1 min, heated to 95°C at 12°C/min, and kept at 95°C for 3 min. During cooling, samples were cooled to 50°C at 12°C/min, and then kept at 50°C for 3 min. Viscosity (expressed in mPa-sec) of the mixed system was monitored throughout the entire heating and cooling cycle. All measurements were performed in triplicate. The errors of rheological measurements were within 10% of the mean values reported.

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Preparation of Composite Gels

Samples were prepared by mixing various concentrations of wheat, corn, or tapioca starch with dHG to S/G 0:6, 1:5, 2:4, 3:3, 4:2, 5:1, and 6:0 in 0.14M sodium bicarbonate to a total solids content of 2, 3, and 4%. The mixture was then poured into a mold (2.5 cm diameter, 2.5 cm height), heated in a water shaker (ZC-40000, Deng Yang Inc., Taiwan) at 95°C for 30 min, and then held at 25°C for 24 hr before dynamic rheological tests were performed.

Dynamic Viscoelasticity of Composite Gels

The starch and dHG composite gel was cut into slices (2 cm diameter, 1 mm height) with a mold and knife. The dynamic viscoelasticity of the composite gel was measured using a controlled-stress rheometer (Carri-Med CSL²100, TA Instrument Ltd., Surrey, England) equipped with a parallel plate measuring- tool (2 cm diameter, 1 mm gap size setting). Measurements were performed at 25°C from 0.1 to 10 Hz under a strain level in the linear viscoelastic range (strain amplitude 1%). The dynamic viscoelastic properties, including storage modulus (G'), loss modulus (G''), and loss factor ($\tan\delta$) were recorded as a function of frequency. All measurements were performed in triplicate. The errors of rheological measurements were within 10% of the mean values reported.

RESULTS AND DISCUSSION

Chemical Composition

Table I lists the chemical compositions of dHG. As compared with the composition of crude *hsian-tsao* leaf gum reported in the literature (Lai et al 2001, 2000; Lai and Chao 2000a,b), it was clear that decolorization significantly reduced the protein content but increased the uronic acid level in *hsian-tsao* leaf gum. Liao (2000) reported that the weight average molecular weight of HG increased from 450,000 to 550,000 and intrinsic viscosity increased from 200 to 400 mL/g after decolorization. The increase in molecular weight could be due to the removal of protein and pigments, and the increase in intrinsic viscosity could be due to an increase in uronic acid levels. On the other hand, as shown in Table II, the nonstarch components in the three starch samples used were <1% (dry basis). The amylose content was the highest in wheat starch, followed by cornstarch and tapioca starch.

RVA

The steady shear rheological properties of starch and dHG composite systems were evaluated using an RVA. For many instruments, the shear rate at which the readings are taken are not known due to the complex geometry (Wood and Goff 1973). The RVA, unfortunately, is one of these instruments. If we approximate the complex geometry in RVA as a rotational viscometer with concentric cylinder unit, we could roughly estimate the maximum shear rate encountered in the measuring cup of the RVA under conditions used in this study. Specifically, if we used the Newtonian formula (Rao 1999) to get an estimate of the shear rate at the rim of the RVA paddle:

$$\dot{\gamma}_w = 4\pi N \left(\frac{\alpha^2}{\alpha^2 - 1} \right) \quad (1)$$

where N is the number of revolutions per second, and α is the radius ratio of the sample cup to the rim of the paddle. Then we could calculate the shear rate at the rim of the paddle as:

$$\dot{\gamma}_w = 4\pi \frac{160}{60} \left(\frac{\left(\frac{3.85/2}{3.49/2} \right)^2}{\left(\frac{3.85/2}{3.49/2} \right)^2 - 1} \right) \cong 188 \text{ 1/s} \quad (2)$$

As compared with the shear rate encountered in a similar instrument, the Brabender Viscoamylograph (≈ 40 1/sec) (Wood and Goff 1973), the RVA produced much higher shear than the Viscoamylograph did. This is reasonable because the common revolution speed used in the RVA is generally higher than that in the Viscoamylograph (160 vs. 75 rpm). In addition, the gap size between the wall of the sample cup and the rim of the measuring tool for the RVA is much smaller than that in the Viscoamylograph.

Samples with only dHG (S/G 0:6) showed very low viscosities (<10 mPa·sec) throughout the entire heating and cooling cycle (data not shown). For samples with only starch (S/G 6:0), under a total polymer concentration of 2%, the pasting behavior of wheat, corn, and tapioca starch did not differ significantly (data not shown). However, as the concentration was raised to 3%, the pasting behavior of tapioca starch reached the highest peak viscosity and underwent relatively greater breakdown (Fig. 1). The higher peak viscosity for tapioca starch was possibly due to higher amylopectin content enhancing molecular entanglement. The relatively greater breakdown was probably due to lower levels of amylose reinforcing the molecular network within the granules (Bahnaassey et al 1994). On the other hand, the lower peak viscosity and longer peak time for wheat and corn starch samples should be related to higher amylose content and stronger bonding forces within starch granules that restricted swelling and limited the increase in viscosity (Bahnaassey et al 1994).

When starch and dHG were mixed at different ratios, the pasting and gelling behaviors of starch were strongly affected by dHG (Fig. 1). During cooking, the peak viscosity developed by composite systems containing tapioca starch at S/G 5:1 and 4:2 was significantly higher than that developed by starch alone. The development of higher peak viscosity was much less pronounced for composite systems containing wheat and corn starch, possibly due

TABLE II
Chemical Compositions of Starches^a

Compositions (%)	Wheat	Corn	Tapioca
Crude protein	0.06(0.01)	0.04(0.02)	0.04(0.00)
Crude fiber	0.15(0.01)	0.10(0.01)	0.13(0.02)
Crude fat	0.19(0.01)	0.09(0.0)	0.28(0.01)
Ash	0.14(0.02)	0.11(0.02)	0.16(0.02)
Amylose	26.49(0.09)	24.85(0.16)	21.37(0.38)
Amylopectin ^b	72.97	74.81	78.02

^a Data expressed as mean of three replicates (standard deviation in parentheses) on dry basis.

^b Amylopectin = 100 - (crude protein + crude fiber + crude fat + ash + amylose).

TABLE I
Chemical Composition of *Hsian-tsao* Leaf Gum^a

Decolorization	Proximate Compositions (%)					
	Crude Fat	Crude Protein	Ash	Crude Fiber	NFE ^b	Uronic Acid (%)
Before ^c	0.86 (0.07)	4.56 (0.17)	26.97 (0.26)	1.07 (0.11)	66.54	31.87 (0.11)
Before ^d	0.52 (0.01)	10.04 (0.21)	26.20 (0.16)	1.47 (0.11)	61.77	19.87 (0.08)
After	0.50 (0.09)	0.70 (0.09)	23.96 (0.09)	2.67 (0.32)	72.17	48.28 (0.32)

^a Data expressed as mean of three replicates (standard deviation in parentheses) on dry basis.

^b Nitrogen-free extracts = 100 - (crude fat + crude protein + ash + crude fiber).

^c Data taken from Lai et al (2000).

^d Data taken from Lai and Chao (2000a).

to higher amylose content and stronger bonding forces within starch granules that restricted swelling, solubilization, and the interaction with dHG at this stage. The imposed high shear and large deformation conditions in the RVA may even make the detection of such weaker and slower development in viscosity more difficult. As the S/G changed to 3:3, 4:2, and 1:5, the peak viscosity was significantly lower than that of starch alone. Such results could be due to the fact that the concentration of starch in these systems was too low for a positive interaction with dHG. In addition, the dHG may also compete for water with starch granules and affect the swelling and breakdown of starch granules as well as the amount of solubilized starch.

After cooking, lowering temperature drove the mixed macromolecules to a more ordered conformation through formation or association of ordered macromolecular chains, which resulted in a marked increase in viscosity. Looking at the data for the tapioca starch and dHG mixed systems with a total concentration of 3% (Fig. 1), we could see that 3% starch alone (S/G 6:0) had a pseudo-gel viscosity of ≈ 200 mPa·sec (Fig. 1 and Fig. 2) and, as mentioned above, 3% dHG alone (S/G 0:6) showed a viscosity value <10 mPa·sec. However, 2.5% tapioca starch and 0.5% dHG mixture (S/G 5:1) showed a pseudo-gel viscosity >800 mPa·sec. Because the viscosity of dHG alone is much lower than the gelatinized starch under comparable concentration, the significant increase in viscosity of starch and dHG mixtures can not simply be explained by the swelling of starch and, hence, increase the gum concentration. In a DSC study, Liao and Lai (2001) reported that dHG or gelatinized starch alone did not show exothermic peaks during cooling, but starch and dHG mixtures at 5:1 and 4:2 showed significant exothermic peak during cooling, indicating the mixed systems showed changes from a disordered state to an ordered state, in other words, the formation of junction zones. Therefore, our results implied that there were interactions between soluble starch (amylose or low molecular weight amylopectin) and dHG, and resulted in an increase viscosity significantly (Lai and Chao 2000a). Because the large stirring action exerted by the RVA stirrer might break the composite paste and gel structure, the viscosity of composite paste and gels may decrease after reaching maximum viscosity values.

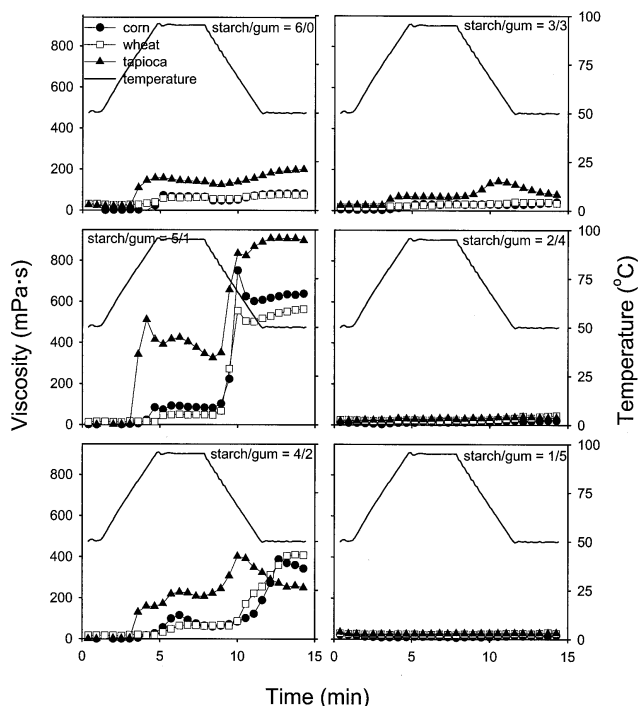


Fig. 1. Viscosity profiles of starch and decolorized *hsian-tsao* leaf gum mixed systems (total solid concentration 3%).

As shown in Fig. 2, for a given total solid content and starch type, the maximum viscosity developed during cooling (pseudo-gel viscosity) was highest for composite systems with S/G 5:1, followed by S/G 4:2. In addition, when comparing two mixtures at 2% starch with different gum concentrations, we noticed something interesting. As shown in Fig. 2, mixtures of 2% starch and 1% gum (total conc. 3%, S/G 4:2) showed higher viscosity than mixtures of 2% starch and 2% gum (total conc. 4%, S/G 3:3). This feature implied excess distribution of hydrocolloid in the intergranular spaces may reduce the interparticle contacts or interrupt the amylose and amylose and gum networks. Optimum interactions occurred when S/G was relevant. It also suggested that the significant increase in viscosity of starch and dHG mixtures was not simply due to the swelling of starch (that resulted in an increase in gum concentration) but also due to interactions between starch and dHG that played a key role for significant synergistic effects. Composite systems with tapioca starch always showed higher pseudo-gel viscosity than with wheat or corn starch. Interactions between starch and dHG were enhanced with increasing total solid content for a given S/G.

Various workers have studied hydrocolloid interactions with starch. The cause of the synergistic effect of hydrocolloids on starch paste viscosity has been attributed to various factors, including a complex reaction between starch exudates and gum, an increase in gum concentration in the continuous phase of the medium due to loss of water into swollen starch granules, and the influence of gum on the physical properties of starch granules such as shape, granule integrity, and amount of exudates from starch granules (Lai and Chao 2000a,b; Christianson 1982; Alloncle and Doublier 1991; Annable et al 1994; Eidam et al 1995). The dHG effects on different types of starch could be explained on a similar basis. Because this study was performed on relatively diluted systems (total polymer concentration $\leq 4\%$), it was believed that much of the amylose could leach out of the starch granules during heating if S/G were relevant. The leached out amylose (and perhaps the low molecular weight amylopectin also) may then rearrange itself and form a coupled network with dHG, if the S/G were relevant (Lai and Chao 2000a).

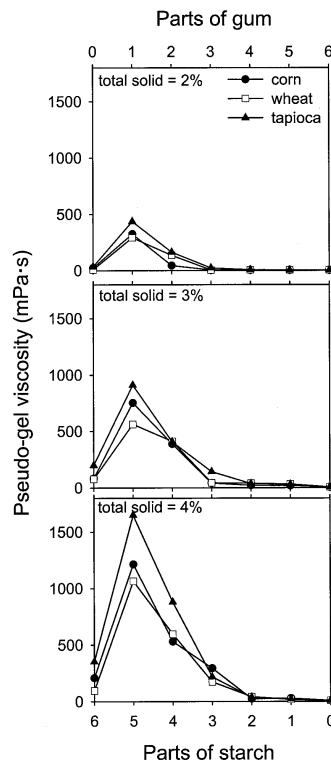


Fig. 2. Pseudo-gel viscosity of starch and decolorized *hsian-tsao* leaf gum mixed systems.

Dynamic Viscoelasticity

RVA is a type of large deformation measurement. Under the action of the imposed shear in such measurement, a competition between gel formation and structure breakdown during cooling could occur. In addition, the formation of a delicate, easily disrupted network at intermediate stage during cooling could not be detected (Lai and Chao 2000b). Therefore, in this study, small deformation rheological properties were also measured using dynamic rheological test to compare the findings in the RVA.

Figure 3 showed the frequency dependence of storage modulus (G') and loss modulus (G'') for dHG and starch alone as a function of concentration. The G' represents the elastic property and is a measure of the magnitude of the recoverable energy that is stored in the materials per cycle of dynamic deformation (Rao 1999). The G'' , on the other hand, is a measure of the energy that is lost as viscous dissipation per cycle of dynamic deformation (Rao 1999). For dHG alone, both G' and G'' showed a strong dependence on frequency. Within the frequency range studied, the G'' values were greater than G' values for all three concentrations tested, and the slopes of the lines were somewhat low. Such results indicated that

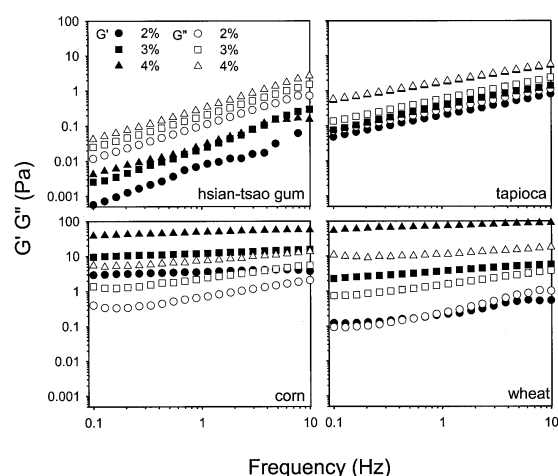


Fig. 3. Frequency sweeps of decolorized *hsian-tsao* leaf gum or starches.

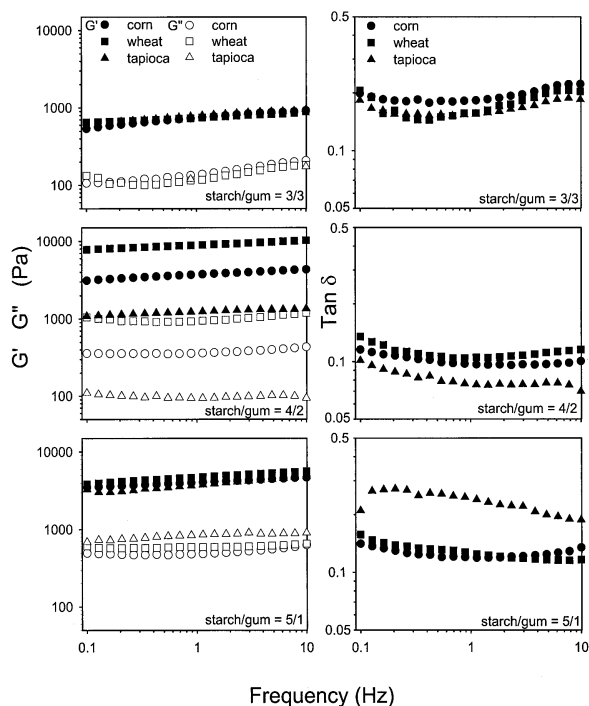


Fig. 4. Frequency sweeps of starch and decolorized *hsian-tsao* leaf gum composite gels (total solid concentration 3%).

dHG alone could be classified rheologically as a semidiluted solution (Clark and Ross-Murphy 1987). Both the G' and G'' values of dHG alone increased with increasing gum concentration due to enhanced molecular entanglement.

Rheologically, the 2–3% tapioca starch alone also belonged to the semidiluted solution category (Clark and Ross-Murphy 1987); G'' values were greater than G' values and the slopes of the lines were somewhat low. However, as the concentration of tapioca starch increased to 4%, the G' and G'' values were almost identical. Such results implied that the gelling concentration for tapioca starch was $\approx 4\%$ (Nishinari 1997). On the other hand, rheologically, 2–3% corn and wheat starch alone systems belonged to the category of weak gel (Clark and Ross-Murphy 1987) as G' values were higher than G'' values and G'' showed stronger frequency dependence than G' . However, as the concentration increased to 4%, the G' values were higher than G'' values, and both moduli were almost frequency-independent. Such results indicated that 4% wheat or corn starch can be classified rheologically as an elastic gel (Clark and Ross-Murphy 1987). It was also noted that, the G' of wheat starch is lower than that of corn starch at 2 and 3% but higher at 4%, though the amylose content of both starches were comparable. This is possibly due to the lower swelling and solubility behavior of wheat starch as compared with corn starch when the concentration is lower than 4% (Ward et al 1994; Zobel 1984).

The dynamic rheological test was not performed on composite systems with S/G 2:4 and 1:5 due to severe syneresis and phase-separation phenomena observed. Figure 4 showed the frequency dependence of G' , G'' , and loss tangent ($\tan\delta$) for composite gels with S/G 3:3, 4:2, and 5:1, where the loss tangent is the ratio of the energy dissipated to that stored per cycle of deformation. As compared to the results shown in Fig. 3, the G' and G'' values of these composite gels were significantly higher than those of starch or dHG alone due to the constructive interactions between starch and dHG. In addition, rheologically these composite gels could be classified as weak to strong gels due to the fact that the G' values were much greater than G'' values, and $\tan\delta$ values were much < 1 over the frequency range tested.

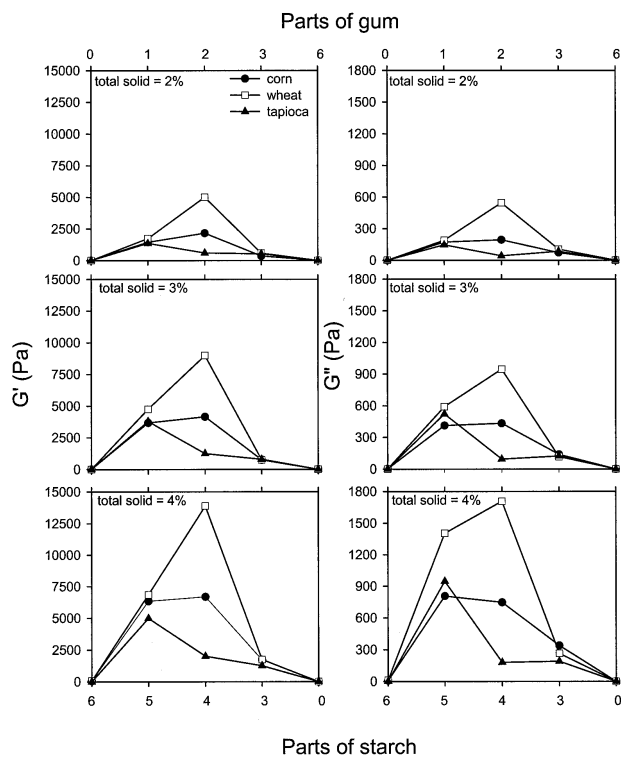


Fig. 5. Dynamic viscoelastic parameters of starch and decolorized *hsian-tsao* leaf gum mixed systems at 1 Hz.

As shown in Fig. 5, for a given total solid content, composite gels containing wheat or corn starch generally showed higher G' and G'' than composite gels containing tapioca starch, and showed the highest G' and G'' at S/G 4:2, followed by S/G 5:1 and 3:3 in decreasing order. Nevertheless, composite gels with tapioca starch showed the highest G' and G'' values at S/G 5:1 followed by 4:2 and 3:3. Such results implied that the S/G for optimum interactions depended on the unique chemistry of each starch, such as the shape, size, average chain length, chain length distribution, and degree of polymerization of amylopectin and amylose molecules, deformability, and the volume fraction occupied (Doublier et al 1987; Ring 1987; Clark et al 1989; Jane and Chen 1992). Similar to what was observed in the RVA measurements, when comparing two mixtures at 2% starch with different gum concentrations, mixtures of 2% starch and 1% gum (total conc. 3%, S/G 4:2) showed higher G' than mixtures of 2% starch and 2% gum (total conc. 4%, S/G 3:3). Again, this feature implied that optimum interactions could occur only when S/G were relevant. When keeping the S/G constant, interactions between starch and dHG increased with increasing total solid content.

Steady and Dynamic Shear Rheological Properties

The RVA results indicated that composite system containing tapioca starch at S/G 5:1 showed the highest pseudo-gel viscosity. Dynamic results indicated that composite gel containing wheat starch at S/G 4:2 showed the highest G' and G'' values. For a gel system, the G' is generally thought to be related to parameters such as the effective chain stiffness, the number and bonding energy of the junction zones, and the mean end-to-end distance between junction points (Oakenfull and Scott 1988; Clark 1992). The G'' is generally thought to be related to parameters such as the flowability of small molecules, mobility of molecular chain, frictional forces between molecular chains, and vibration and rotation of functional groups (Oakenfull and Scott 1988; Clark 1992). In addition, Liao and Lai (2001) report that tapioca starch and dHG composite gel showed less orderly network structure than did wheat starch and dHG composite gels. Therefore, we believed that in the RVA test, the viscous nature of tapioca starch (due to its higher amylopectin content) and the less orderly network structure formed with dHG (Liao and Lai 2001) would make a composite system showing higher resistance to the imposed large deformation that would result in higher pseudo-gel viscosity. In dynamic testing, the number and bonding energy of the junction zones contributed to the magnitude of G' (Oakenfull and Scott 1988; Clark 1992). The higher amylose content and less viscous nature of wheat starch seemed to assist interaction with dHG more effectively, which resulted in the formation of a more ordered network structure and higher G' values. Such results implied that tapioca starch contributed more viscous properties, but wheat and corn starch contributed more elastic properties to the mixed systems and were consistent with the informal sensory findings.

ACKNOWLEDGMENTS

This research was supported by National Science Council, Taiwan (NSC89-2313-B-126-010). Their support is greatly appreciated.

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[Received May 7, 2001. Accepted September 26, 2001.]