Effects of Cellulose Gums on Rheological Interactions in Binary Mixtures of Xanthan Gum and Locust Bean Gum

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ABSTRACT: The effects of cellulose gums (CG), such as carboxymethyl cellulose (CMC) and hydroxypropyl methylcellulose (HPMC), on the flow and dynamic rheological properties of binary mixtures of xanthan gum (XG) and locust bean gum (LBG) were examined at different XG/LBG/CG mixing ratios (50/50/0, 47.5/47.5/5.0, 45/45/10, and 42.5/42.5/15.0). All XG/LBG/CG ternary mixtures showed high shear-thinning behavior and the flow behavior index values of samples containing HPMC were lower than those of samples containing CMC. An increase in consistency index and apparent viscosity values was observed for ternary gum mixtures containing HPMC, indicating that the flow properties of the XG/LBG binary mixture were affected by the content of HPMC. Storage modulus and loss modulus values of ternary gum mixtures decreased with an increase in CG content from 5 to 15%. The maximum viscoelasticity of XG/LBG/CG mixtures was observed at a mixing ratio of 47.5/47.5/5.0. These findings suggest that the rheological properties of XG/LBG binary mixtures were strongly influenced by the addition of CMC and HPMC.

Keywords: xanthan gum, cellulose gum, rheological property, viscoelasticity

INTRODUCTION

Hydrocolloids, or gums, are high-molecular-weight hydrophilic biopolymers commonly used as functional ingredients in the food industry for the control of microstructure, texture, flavor, and shelf-life (1). They can be used as stabilizing, thickening, and gelling agents to improve and control the rheological and textural properties of food products. In particular, some gums and their mixtures have been used to develop food formulations for patients with dysphagia (2). Gums such as xanthan gum (XG), guar gum (GG), locust bean gum (LBG), and cellulose gums (CGs) [e.g, carboxymethyl cellulose (CMC) and hydroxypropyl methylcellulose (HPMC)] are most commonly used in binary or ternary gum mixture systems, especially in concentrated solution systems (≥1.0 %). These gum mixtures are used as food thickeners for safe and easy swallowing; they can improve food bolus formation and mouthfeel because of the synergistic interactions between individual gums (3-6). In particular, the synergistic interactions of the binary mixtures of XG and galactomannans (GG and LBG) have been scientifically demonstrated in various studies (7-9). However, there are no studies of ternary gum mixtures of XG/GG/CG in concentrated solution systems.

XG is known to exhibit unique flow properties with pronounced shear-thinning behavior, due to its rigid, rod-like conformation, which is more responsive than a random-coil conformation to shear. In particular, XG is the most studied gum for dysphagia diets because it has various desirable properties including a stable viscosity with a wide range of pH, temperature, and salt content; it is a clear, weak gel-like liquid that can be applied to a variety of liquid foods (3,10-12). It has also been used in diet modifications for patients with dysphagia because of its smooth texture and stable bolus formation with high viscosity and elasticity (5). Moreover, XG is often used with LBG because of their synergistic rheological properties, resulting from the intermolecular binding or cooperative interaction between XG and mannan molecules (13). CMC and HPMC, which are water-soluble derivatives of the natural polysaccharide cellulose, give clear and odorless solutions of high viscosity and are also used in food applications as viscosity modifiers or food thickeners with other gums (14,15). In aqueous solutions, CMC or HPMC represents a complex rheological system as aggregates and associations can be formed (15). At higher CG concentrations, extended CG chains start to overlap and undergo the coiling process, which can result in the formation of a network structure due to polymer-

polymer interactions (entanglements) in a concentrated regime (16).

Various studies have indicated that the synergistic interactions between XG and LBG may be mediated via the intermolecular binding mechanism between the surface of the ordered XG helix and the smooth (unsubstituted) regions of the LBG backbone (9,17-19). However, it is insufficient to assess only the rheological synergism between XG, LBG, and CG in the ternary gum mixture systems in terms of thickened fluids prepared with a food thickener for dysphagia management. Therefore, in this study, the steady and dynamic shear rheological properties of ternary mixtures of XG, GG, and CG with different contents of CMC and HPMC (mixing ratio of XG/LBG/CG: 50/50/0, 47.5/47.5/5.0, 45/45/10, and 42.5/42.5/15.0) were investigated in a concentrated solution (1% w/w). The purpose of this study was to determine the rheological properties of ternary mixtures composed of XG, LBG, and CMC or HPMC to develop formulations of food thickeners for patients with dysphagia, and to compare the rheological differences between XG/LBG/CMC and XG/LBG/HPMC ternary mixtures.

MATERIALS AND METHODS

Materials and preparation of ternary gum mixtures
XG, LBG, CMC, and HPMC were purchased from Sigma-Aldrich Co. (St. Louis, MO, USA). The stock solutions (1% w/w) were prepared by mixing the individual gum (1.0 g) with de-ionized water (100 mL) under continuous stirring with a magnetic stirrer at room temperature for 8 h. For the preparation of LBG solution, it was heated for 1 h at $85^{\circ}C$ in a water bath after dispersion at room temperature. All solutions were kept overnight (16 h) at room temperature to completely hydrate the gums. Based on XG-LBG binary mixtures, which were mixed half and half, the ternary mixtures of XG, GG, and CG were blended at the following XG/LBG/CG ratios: 50/50/0, 47.5/47.5/5.0, 45/45/10, and 42.5/42.5/15.0. Subsequently, they were left to stand for 1 h before measuring the rheological properties.

Determination of steady and dynamic shear rheological properties
Flow and dynamic rheological measurements were performed using a controlled stress rheometer (HAAKE RheoStress 1, Gebrüder Haake GmbH, Karlsruhe, Germany). The plate-plate geometry was used (diameter, 35 mm). Each sample was loaded between the parallel plates at $25^{\circ}C$ and compressed to obtain a gap of 500 μm. Each sample was transferred to the rheometer plate and equilibrated at $25^{\circ}C$ for 5 min before steady and dynamic shear rheological measurements were taken. All rheolo-

gical measurements were performed in triplicate.

Steady shear flow tests were performed over a shear rate range of $0.1 \sim 100$ s^{-1}. The shear stress against shear rate data were fitted to the power law model (Eq. 1) according to the following equation:

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

where σ (Pa) is the shear stress, $\dot{\gamma}$ (s^{-1}) is the shear rate, K (Pa·s^n) is the consistency index, and n is the flow behavior index. The apparent viscosity ($\eta_{a,50}$) at a specific shear rate of 50 s^{-1} was calculated using the values of K and n.

Small-amplitude oscillatory rheological measurements were performed to obtain dynamic rheological data. Frequency sweep tests were carried out over a range of $0.63 \sim 62.8$ rad·s^{-1} at a constant strain of 2% (within the linear viscoelastic region). The measurement temperature was maintained at $25^{\circ}C$. Dynamic viscoelastic parameters including the storage (or elastic) modulus (G'), loss (or viscous) modulus (G''), and tan δ (G''/G') were determined with HAAKE RheoWin data manager software (Gebrüder Haake GmbH). To relax the samples prior to flow and dynamic rheological measurements, each measurement was taken at $25^{\circ}C$ after a 5 min rest.

Statistical analysis
The results are presented as the mean±standard deviation (SD) of triplicate analyses for each sample. Analysis of variance (ANOVA) and Duncan's test were used to establish the significance of differences among the mean values at the 0.05 significance level. Statistical analyses were performed using the Statistical Analysis System program (version 9.2) (SAS Institute, Cary, NC, USA).

RESULTS AND DISCUSSION

The shear stress versus shear rate data for XG/LBG/CMC and XG/LBG/HPMC ternary mixtures at different mixing ratios are presented in Fig. 1. The effect of CMC and HPMC content ($0 \sim 15\%$) on the flow properties of ternary gum mixtures was determined by the power law model (Eq. 1) with high determination coefficients ($R^2 = 0.98 \sim 0.99$). The results are summarized in Table 1. All samples exhibited a non-Newtonian (pseudoplastic) nature, showing high shear-thinning behavior with flow behavior index (n) values of as low as $0.13 \sim 0.21$. The n values ($0.13 \sim 0.17$) of XG/LBG/HPMC ternary mixtures were lower than that (0.18) of the XG/LBG binary mixture, and the n value (0.13) of the sample with 5% HPMC was the smallest compared with those ($0.14 \sim 0.21$) of other samples. It is known that liquids with lower n val-
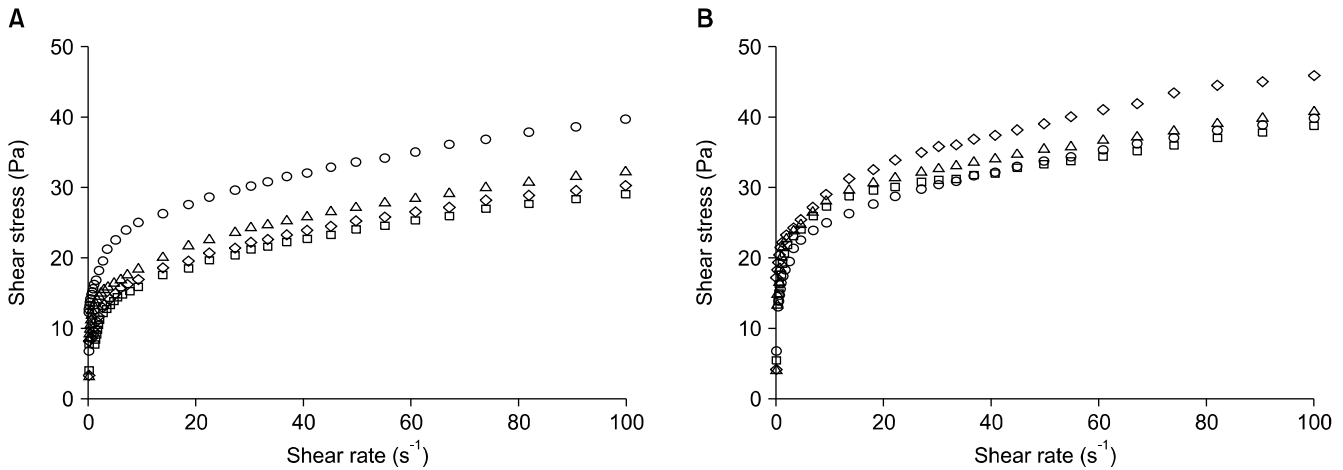
Fig. 1. Plots of shear stress versus shear rate of (A) xanthan gum (XG)/locust bean gum (LBG)/carboxymethyl cellulose and (B) XG/LBG/hydroxypropyl methylcellulose mixtures at different mixing ratios (50/50/0, 47.5/47.5/5.0, 45/45/10, and 42.5/42.5/15.0). , 50/50/0; , 47.5/47.5/5.0; , 45/45/10; , 42.5/42.5/15.0.

Table 1. Apparent viscosity ($\eta_{a,50}$), consistency index (K), and flow behavior index (n) of XG/LBG/CMC and XG/LBG/HPMC ternary mixtures at different mixing ratios

Mixing ratio	XG/LBG/CMC				XG/LBG/HPMC			
	$\eta_{a,50}$ (Pa s)	K (Pa s^n)	n (−)	R^2	$\eta_{a,50}$ (Pa s)	K (Pa s^n)	n (−)	R^2
50/50/0	0.68 ± 0.01^a	17.0 ± 0.18^a	0.18 ± 0.00^c	0.99	0.68 ± 0.00^d	17.0 ± 0.18^b	0.18 ± 0.00^a	0.99
47.5/47.5/5.0	0.47 ± 0.00^d	11.1 ± 0.14^c	0.19 ± 0.00^b	0.98	0.69 ± 0.00^c	20.4 ± 0.30^a	0.13 ± 0.00^c	0.98
45/45/10	0.55 ± 0.00^b	12.6 ± 0.17^b	0.20 ± 0.00^b	0.99	0.71 ± 0.01^b	20.5 ± 0.30^a	0.14 ± 0.00^c	0.98
42.5/42.5/15.0	0.50 ± 0.00^c	11.1 ± 0.16^c	0.21 ± 0.00^a	0.99	0.79 ± 0.00^a	20.4 ± 0.43^a	0.17 ± 0.01^b	0.99

Values are means of three measurements± SD.
Means with different letters (a-d) within each column are significantly different ($P<0.05$).
XG, xanthan gum; LBG, locust bean gum; CMC, carboxymethyl cellulose; HPMC, hydroxypropyl methylcellulose.

ues could provide good mouthfeel (20). A study (6) has also reported that higher shear-thinning behavior allows thickened liquids to be swallowed easily and reduces organoleptic sliminess during swallowing, resulting in a pleasant and light mouthfeel. Therefore, the addition of a small amount of HPMC to the XG/LBG binary mixture may reduce the organoleptic sliminess of thickened liquids. In addition, the n values of the XG/LBG/HPMC mixtures were much lower compared with those of the XG/LBG/CMC mixtures, indicating that the addition of HPMC decreased the shear-thinning behavior of XG/LBG binary mixtures. Therefore, the addition of HPMC may reduce the organoleptic sliminess of thickened liquids prepared with gum-based thickeners.

The K and $\eta_{a,50}$ values of the XG/LBG/HPMC mixtures were much higher than those of the XG/LBG binary mixture (0% CG) (Table 1), whereas those of the XG/LBG/CMC mixtures were much lower, indicating that the addition of HPMC and CMC greatly affected the viscosity of XG/LBG mixture. In addition, the K and $\eta_{a,50}$ values of ternary mixtures containing HPMC were higher compared with those of ternary mixtures containing CMC. This suggests that HPMC had a more pronounced effect on the flow properties of the XG/LBG mixture due to its

higher water-binding capacity and thickening properties. The $\eta_{a,50}$ values of ternary gum mixtures decreased with an increase in CG content from 5 to 15%. On the other hand, there were no differences in K values among the ternary gum mixtures except for the mixture with 10% CMC. Based on the results, the ternary gum mixtures containing HPMC were more efficient than the XG/LBG binary mixture in improving flow properties, indicating that the flow properties of XG/LBG mixture can be affected by the addition of CMC and HPMC, the gum mixing ratio, and the type of CG.

Generally, viscoelastic properties play an important role in the swallowing of food bolus (5,6,10). The dynamic moduli (G' and G") can be used to predict the degree of swallowing the food bolus with ease. The magnitudes of G' and G" at 6.28 rad·s^{-1} for ternary gum mixtures with different CG contents at 25°C are shown in Table 2. Their G' and G" values, except for samples with 10 and 15% CMC, were higher compared with those of the XG/LBG binary mixture, and the values also decreased with an increase in the CG content from 5 to 15%, indicating that the viscoelastic properties of the ternary mixtures decreased with increasing CG content. The ternary gum mixtures with 5% CG had the highest G' and G" values.

Table 2. Storage modulus (G'), loss modulus (G"), and tan δ of XG/LBG/CMC and XG/LBG/HPMC ternary mixtures at different mixing ratios

Mixing ratio	XG/LBG/CMC			XG/LBG/HPMC		
	G' (Pa)	G" (Pa)	tan δ	G' (Pa)	G" (Pa)	tan δ
50/50/0	40.0± 0.39[b]	11.9± 0.14[c]	0.30± 0.00[d]	40.0± 0.69[c]	11.9± 0.14[b]	0.30± 0.00[a]
47.5/47.5/5.0	40.9± 0.21[a]	14.2± 0.03[a]	0.35± 0.00[c]	47.2± 0.70[a]	13.7± 0.18[a]	0.29± 0.00[b]
45/45/10	33.8± 0.23[c]	13.5± 0.13[b]	0.40± 0.00[b]	42.0± 0.57[b]	12.1± 0.16[b]	0.29± 0.00[b]
42.5/42.5/15.0	27.8± 0.36[d]	12.0± 0.10[c]	0.43± 0.00[a]	40.4± 0.27[bc]	11.9± 0.11[b]	0.30± 0.00[a]

Values are means of three measurements± SD.
Means with different letters (a-d) within each column are significantly different (P<0.05).
XG, xanthan gum; LBG, locust bean gum; CMC, carboxymethyl cellulose; HPMC, hydroxypropyl methylcellulose.

These higher G' and G" values could be explained by the formation of a greater viscoelastic weak gel network with low CG content. In particular, the G' values of the XG/LBG/HPMC mixtures were higher than those of the XG/LBG/CMC mixtures, indicating that the elastic properties of the XG/LBG mixture may be more pronounced in the presence of HPMC. The improved elastic properties of the XG/LBG/HPMC mixtures may be attributed to the higher elastic properties of the added HPMC. The HPMC solution has been reported to have higher elastic properties than the CMC solution because of its high-water-binding capacity and thickening properties (21). Therefore, the addition of HPMC to the XG/LBG mixture appears to contribute synergistically to the elastic properties of the XG/LBG mixture. The observed synergism can be explained by complex mutual exclusion effects between HPMC and the binary gum mixture as a result of polymer incompatibility. Incompatible polymers can also exhibit synergy by naturally concentrating each other in a single-phased system via excluded volume effects (22, 23).

The tan δ values of all samples were in the range of 0.29~0.43 (<1.0), indicating that the elastic nature prevailed over the viscous nature. The tan δ values of XG/LBG/CMC increased with an increase in CMC content from 0 to 15%, indicating a decrease in the elastic characteristics of the ternary mixtures. In contrast, there were no noticeable changes in the tan δ values among the XG/LBG/HPMC mixtures. These results demonstrated that

G" increases much more than G' in response to the addition of CMC, and the effect of CMC on the viscous properties of XG/LBG mixture was greater than that of HPMC. The increased viscous properties in the presence of CMC indicated that the modification of the dynamic rheological properties might be attributed to an incompatible network structure in which the effective concentration of both polymers would be increased in their own microdomains (24). In particular, the tan δ values (0.29~0.30) of the XG/LBG/HPMC mixtures were lower than those (0.35~0.43) of the XG/LBG/CMC mixtures. These findings suggest that the addition of a small amount of CMC to the XG/LBG binary mixture can substantially reduce the elastic properties of the ternary gum mixture system. The higher tan δ value of ternary mixtures containing CMC seems to be strongly dependent on the viscoelastic characteristics of CMC which is more viscous than elastic compared with other gums (21).

In the ternary mixture system, log (G' and G") versus log ω were also subjected to linear regression; the magnitudes of the slopes and the coefficients of determination (R^2) were determined (Table 3). The slopes of G' (0.18~0.27) and G" (0.16~0.24) were positive with a high R^2 (0.98~0.99). The slopes of G" increased with an increase in CG content from 5 to 15% and the slopes of G' of the XG/LBG/CMC mixtures were also increased, indicating that the dynamic moduli were greatly dependent on ω at a higher CMC content. However, there was no difference in the slopes of G' between the XG/LBG/

Table 3. Slope values of the log (G' and G") versus log ω (frequency, rad s[−1]) of XG/LBG/CMC and XG/LBG/HPMC ternary mixtures at different mixing ratios

Mixing ratio	XG/LBG/CMC				XG/LBG/HPMC			
	Slope of G'	R^2	Slope of G"	R^2	Slope of G'	R^2	Slope of G"	R^2
50/50/0	0.19± 0.00[d]	0.998	0.18± 0.01[b]	0.99	0.19± 0.00[a]	0.998	0.18± 0.01[c]	0.99
47.5/47.5/5	0.22± 0.00[c]	0.997	0.16± 0.00[c]	0.99	0.18± 0.00[b]	0.998	0.18± 0.00[c]	0.99
45/45/10	0.24± 0.00[b]	0.998	0.18± 0.00[b]	0.99	0.18± 0.00[b]	0.998	0.21± 0.01[b]	0.98
42.5/42.5/15	0.27± 0.00[a]	0.999	0.23± 0.00[a]	0.99	0.18± 0.00[b]	0.997	0.24± 0.01[a]	0.98

Values are means of three measurements± SD.
Means with different letters (a-d) within each column are significantly different (P<0.05).
XG, xanthan gum; LBG, locust bean gum; CMC, carboxymethyl cellulose; HPMC, hydroxypropyl methylcellulose.

HPMC mixtures. The slopes of G'' were higher than those of G' at a higher HPMC content (>5%), indicating that the addition of HPMC (>5%) to the XG/LBG binary mixture increased the viscoelastic properties of XG/LBG/HPMC ternary mixtures. In particular, the G' slopes (0.18) of XG/LBG/HPMC mixtures were relatively lower than those (0.22~0.27) of the XG/LBG/CMC mixtures, suggesting that the XG/LBG/HPMC mixtures were more elastic than the XG/LBG/CMC mixtures. Furthermore, the G' values of the XG/LBG/CMC mixtures showed a greater dependency on ω compared with those of the XG/LBG/HPMC mixtures. There were also large differences between the slopes of G' of the XG/LBG binary mixture and XG/LBG/CMC ternary mixtures, demonstrating that the elastic properties of the XG/LBG binary mixture can be reduced by the addition of CMC. The result is consistent with the more pronounced effect of HPMC on the elastic properties of the XG/LBG binary mixture, which was observed at different mixing ratios. These findings suggest that the changes in the viscoelastic properties of XG/LBG/CG ternary mixtures were greatly affected by the addition of CG, and these changes were dependent on the type and content of CG.

In conclusion, ternary gum mixtures containing HPMC were more efficient in terms of improving flow properties, showing higher $\eta_{a,50}$ and K values compared with those of ternary gum mixtures containing CMC. In addition, XG/LBG/CG ternary mixtures with 5% CG were more efficient in improving the viscoelastic properties with relatively higher G' values and lower slope values, indicating an elastic weak gel-like structure. The results in this study suggest that the changes in the flow and dynamic rheological properties of XG/LBG/CG ternary mixtures were greatly affected by the addition of CG, and these changes were dependent on the type and content of CG. Knowledge of the specific rheological properties of ternary gum mixtures could be useful for developing formulations of gum-based food thickeners for patients with dysphagia.

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AUTHOR DISCLOSURE STATEMENT

The authors declare no conflict of interest.

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