

Viscoelastic characterization of gum arabic and maize starch mixture using the Maxwell model.

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Abstract

The aim of this study was the viscoelastic characterization of dispersions of gum arabic (GA) at 25 and 35% (w/v), maize starch (MS) at 3 and 5% (w/v) and their mixtures (GA/MS) using dynamic oscillatory and creep tests. The studied dispersions presented G' values of up to 8.5, 12.7 and 3.5 greater than those of G'' in GA, MS and GA/MS dispersions, respectively, showing a predominantly elastic behavior. Creep curves revealed that in 5% (w/v) MS dispersions, recovery reached 96.7%; thus, they behaved as a viscoelastic solid. However, by adding gum arabic into the mixtures (GA/MS), no recovery was observed and they behaved as viscoelastic fluids. In relation to Maxwell model parameters, viscosity (η_0) was approximately 6 Pa s for GA and GA/MS dispersions, and 1.12×10^6 Pa s for MS dispersions. Mixture relaxation time (λ_{rel}) was between 0.023 and 220 s and instantaneous compliance (J_0) was greater than retarded compliance (J_1) regardless of MS concentration.

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1. Introduction

Gum arabic, also called gum acacia, is a complex heteropolysaccharide that belongs to the arabinogalactan family exuded by trees of the genus *Acacia*, mainly in the sub-desert regions of Africa (Sanchez, Renard, Robert, Schmitt, & Lefebvre, 2002). Structurally, gum arabic is a branched molecule with a backbone formed by β -D-galactopyranosyl units linked by 1,3 bonds, which has β -D-galactopyranosyl chains linked by 1,6 bonds. The backbone and branches are linked to α -L-arabinofuranosyl, α -L-rhamnopyranosyl, β -D-glucuronopyranosyl, and 4-O-methyl- β -D-glucuronopyranosyl molecules (Meer, 1980; Whistler, 1993). Gum arabic has been used in industry mainly because of its emulsifying, thickening and encapsulating properties (Whistler, 1993). In addition, the physical, hydrating and emulsifying properties have been reported (Biswas et al., 2000; Phillips et al., 1996; Ray et al., 1995).

Other biopolymers, such as maize starch, have been widely used in the food industry because it imports bulk and body; when mixed with hydrocolloids, they modify and control the texture and improve moisture retention of food products (Appelqvist & Debet, 1997). Changes have been observed in the melting, gelatinization, fragmentation and retrogradation in presence of hydrocolloids such as xanthan gum, gellan gum and guar gum, among others (Appelqvist & Debet, 1997; Fanta & Christianson, 1996; Rayment et al., 1998; Yoshimura et al., 1998); however, the role of these polymers in the rheology of complex mixtures remains unknown.

Research on the rheological behavior of dispersion of food gums, maize starches and their mixtures has been mainly focused on the characterization of flow and viscoelasticity by dynamic oscillatory methods (Alloncle et al., 1989; Lopes da Silva et al., 1993; Ma & Barbosa, 1996; Martínez-Padilla et al., 2004; Mothé & Rao, 1999; Sanchez et al., 2002). There are few creep/recovery studies in systems containing starch. These studies allow differentiation between the viscous and elastic response of the sample, determination of the viscosity and elasticity dependence on a given strain rate, and introduce an additional parameter of 'relaxation time' to the dependence

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on viscous and elastic behavior on stress (Schramm, 1994; Steffe, 1996). Creep studies are determined in terms of compliance $J(t)$, which is the quotient of the deformation resulting from the constant stress applied to the sample (Rayment et al., 1998). In the linear region, the magnitude of the applied stress is very small and the constant stress/recovery response is independent of this stress (Ferry, 1980; Gladwell et al., 1985). The constant stress/recovery response and its mathematical representation use different models that include springs and dashpots, which when combined in series or in parallel, characterize the viscoelastic properties of fluids and solids. Mathematical equations also correlate the applied stress with the resulting deformation (Schramm, 1994). There are three main models: the Maxwell, the Kelvin–Voigt and the Burgers model, which combines the first two models placed in series, allowing characterization of the viscoelasticity of fluids and solids. In the present study, the Maxwell model (Eq. (1)) was used since most systems behave as viscoelastic fluids (Fig. 1) (Steffe, 1996).

$$J(t) = J_0 + \frac{\eta}{t} + \sum_{i=1}^n J_i \left(1 - e^{-\frac{t}{\lambda_{rel}^i}}\right) \quad (1)$$

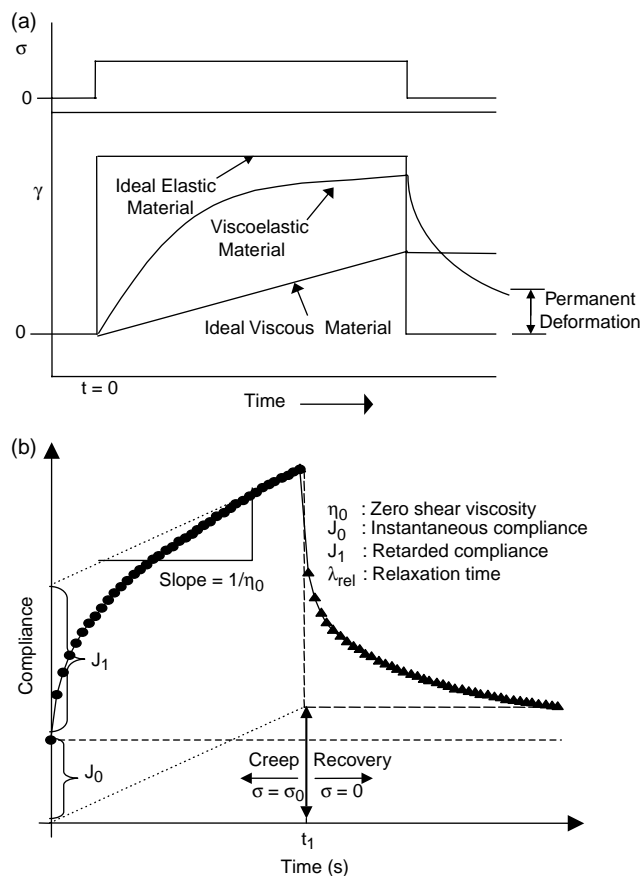


Fig. 1. Graphic representation. (a) Typical creep and recovery curves. (b) Maxwell model parameters.

The aim of this study was to characterize the viscoelasticity of dispersions of gum arabic, maize starch, and their mixtures applying the Maxwell model.

2. Materials and methods

2.1. Materials

Food-grade gum arabic (GA) was purchased at La Farmacia París (México); the preparation contained 11.52% moisture, 83.94% carbohydrate, 1.39% protein, 2.81% ash and 0.34% fat. Food-grade maize starch (MS) was also purchased at La Farmacia París (México) and contained 16.15% moisture, 82.24% carbohydrate, 1.04% protein, 0.09% ash and 0.48% fat.

2.2. Preparation of the dispersions

GA dispersions at 25 and 35% concentrations and MS dispersions at 3 and 5% were prepared with distilled water on a hot plate stirrer model 524C (Barnstead Int., USA) with vigorous stirring for 15 min; GA dispersions were prepared at 37 °C and MS dispersions at 90 °C, holding the temperature for 5 min and subsequently leaving them to cool and rest for 24 h. The GA/MS mixture dispersions were prepared at 25 and 35% concentrations of GA, and 3 and 5% concentrations of MS, respectively, in the same manner, slowly adding the MS first and then the GA to avoid lump formation; these mixtures were heated to 90 °C for 5 min, and subsequently left to cool and rest for 24 h at room temperature before the rheological determinations were performed in duplicate.

2.3. Dynamic rheological test

Determinations were performed at 25 ± 0.1 °C in a low stress rheometer LS 100 (Paar Physica, Germany), which included a circulating water bath (Julabo F10, Germany) with ± 0.1 °C accuracy. Geometries used were parallel plate (PP20) of 20 mm diameter for shear rates between 5.0×10^{-5} and 1.0×10^3 s⁻¹ and shear stress between 6.37×10^{-1} and 6.37×10^3 Pa for 5% MS dispersion, and concentric cylinder (CC18) with 18.0 mm diameter and 27.0 mm length for shear rates between 6.16×10^{-5} and 1.23×10^3 s⁻¹ and shear stress between 6.04×10^{-2} and 6.04×10^2 Pa. The Paar Physica ver. 2.06—E software was used for the calculation of dynamic and Maxwell model parameters. An amplitude scan was performed with a 1×10^{-3} – 1×10^1 mN m torque to determine the linear viscoelasticity region (LVR), which is obtained when the storage modulus (G') and loss modulus (G'') become independent of amplitude; subsequently, a frequency scan was performed within the 6–60 rad s⁻¹ interval to determine the behavior of elastic and viscous components related to frequency.

2.4. Creep test

This test is divided in two phases: creep and recovery. The method consists of the application of stress and its subsequent suspension to evaluate the recovery of the dispersion and the characteristic parameters that represent this behavior. Four curves were determined by applying different stress levels within the LVR interval for 240 s and dispersion recovery was determined when stress was suspended. Results are in compliance terms adjusted to the Maxwell model to obtain the parameters: limiting viscosity at zero shear rate (η_0), instantaneous compliance J_0 (Pa^{-1}), retarded compliance J_1 (Pa^{-1}) and relaxation time λ_{rel} (s). Determinations were performed at 25 ± 0.1 °C in a Paar Physica LS 100 low stress rheometer using parallel plates for 5% starch dispersions and concentric cylinders for the others.

3. Results and discussion

The storage modulus (G'), loss modulus (G'') and phase angle (δ) are among the parameters that characterize a system in a dynamic rheological study. G' is a measure of the energy temporarily stored in a material; G'' is a measure of the energy used to activate a flow, energy that is dissipated and transformed into heat, and δ represents the phase difference between stress and deformation,

dependent and independent variables, in an oscillatory system.

3.1. Dynamic rheological test

Fig. 2 shows the variation of storage and loss moduli (G' and G'') with frequency in 25% GA dispersion, 3 and 5% MS dispersions and their mixtures (GA/MS). In GA dispersions, the higher the frequency, the more the dispersion elasticity ($G' > G''$) and in 3% MS dispersions the behavior was more elastic than viscous ($G' > G''$) in the studied frequency interval. In dispersions containing GA/MS mixtures (25/3%) at low frequencies, G' was slightly smaller than G'' , in contrast to that at high frequencies where a predominantly elastic behavior was observed; in these mixtures, the elastic component (G') showed a viscoelastic behavior similar to that observed in dispersions containing GA and the viscous component (G'') presented a magnitude intermediate between 25% GA and 3% MS dispersions. G' and G'' showed no differences in the 5% MS mixture in contrast to the mixture of lower MS concentration (3%), where G' exhibited a behavior similar to that obtained in 25% GA dispersions. In dispersions of 3% MS, the elastic component (G') presents a zone Plateau, where G' is independent of the frequency studied interval, to low frequencies (6–15 rad/s), what indicates a behavior viscoelastic with solid character although the appearance of the sample is liquid (Rodríguez, 2004). In other studies (Closs et al., 1999) also, $G' > G''$,

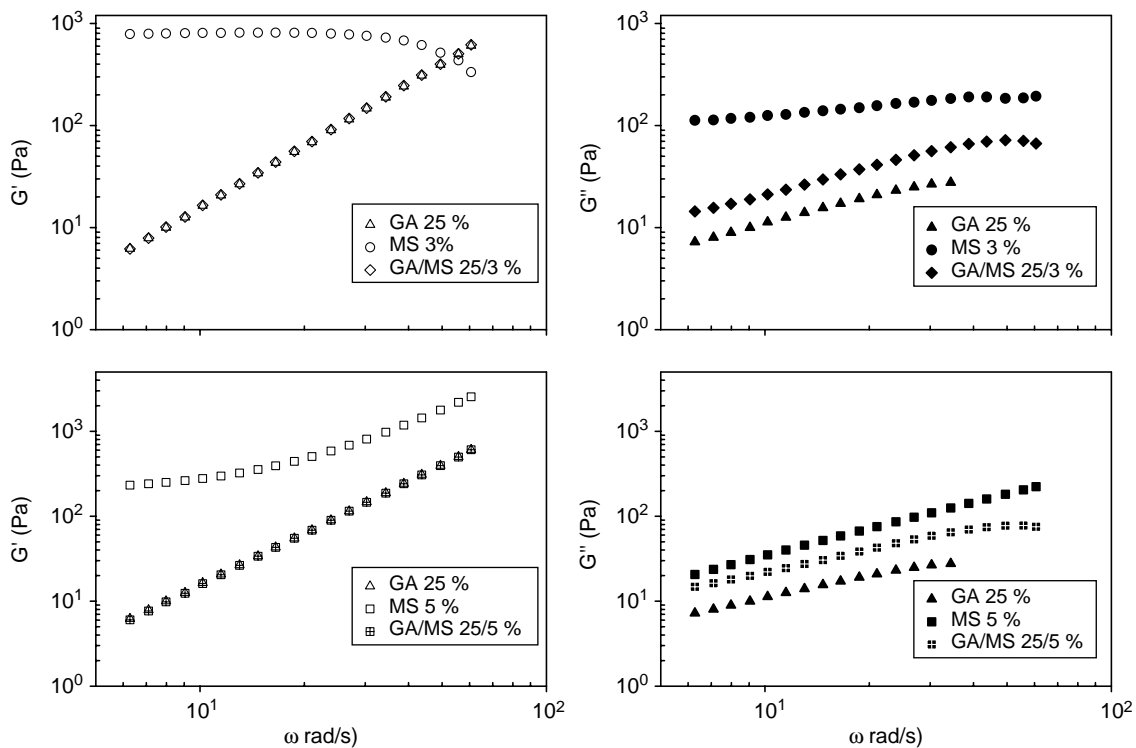


Fig. 2. Behavior of dynamic moduli (G' , G'') in relation to frequency in dispersions of gum arabic (GA) at 25% (w/v), maize starch (MS) at 3 and 5% (w/v) and gum arabic/maize starch mixtures (GA/MS).

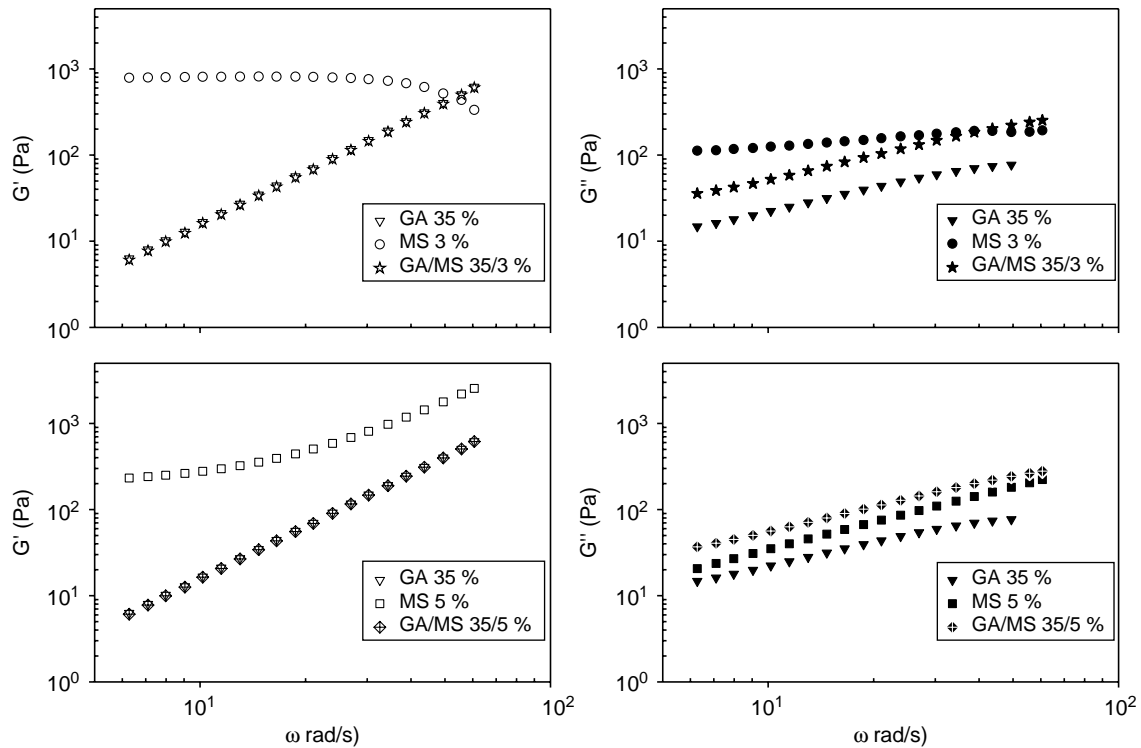


Fig. 3. Behavior of dynamic moduli (G' , G'') in relation to frequency in dispersions of gum arabic (GA) at 35% (w/v), maize starch (MS) at 3 and 5% (w/v) and gum arabic/maize starch mixtures (GA/MS).

the rheological characterization revealed the greater structural rigidity, indicated by the increase in measured G' and G'' values, and marked frequency dependence on the dynamic moduli was found for all samples of aqueous starch/galactomannan system.

Fig. 3 shows the variation of G' and G'' with frequency in dispersions of 35% GA, 3 and 5% MS and their mixtures (GA/MS). In dispersions of GA/MS mixtures containing 35% GA and 3% MS, the elastic component showed a behavior similar to that of 35% GA dispersions, while the viscous component, at low frequencies, presented a magnitude intermediate between 35% GA and 3% MS dispersions; however, at high frequencies, the viscous character is greater than that shown by 3% MS dispersions. In GA/MS dispersions with the higher MS concentration (5%), G' presented a viscoelastic behavior similar to that of 35% GA dispersions, while G'' presented a magnitude

intermediate between 35% GA and 5% MS dispersions within the studied frequency interval. In dispersions of 5% MS, it does not present a zone Plateau; it is observed that G' is bigger than G'' and a minimal dependence of these moduli (G' , G'') with the frequency, characteristic behavior like gel (Giboreau et al., 1994).

Table 1 depicts the dynamic parameters for dispersions of GA, MS and their mixtures. Torque amplitude was within the 0.007 and 3.094 mN m interval; in all cases, G' was greater than G'' ($\tan \delta < 1$), hence dispersions presented a predominantly elastic behavior. G' values in GA dispersions presented no significant differences in relation to the studied concentrations, but a dependence was observed with frequency greater than that of G'' , according to the power law ($G' \propto \omega^{2.01, 2.02} > G'' \propto \omega^{0.82, 0.84}$, at 25 and 35%, respectively). The calculated exponents were approximately $\cong 2$ and < 1 , which generally represent dispersions with

Table 1
Material functions of dispersions of gum arabic (GA), maize starch (MS) and gum arabic/maize starch mixtures (GA/MS)

Dispersions, % (w/v)		Torque amplitude (mN m)	G' (Pa)		G'' (Pa)		$\tan \delta$
GA	25	3.094	145	$\alpha \omega^{2.01}$	17	$\alpha \omega^{0.82}$	0.12
	35	2.090	147	$\alpha \omega^{2.02}$	45	$\alpha \omega^{0.84}$	0.30
MS	3	0.070	803	$\alpha \omega^{0.03}$	148	$\alpha \omega^{0.30}$	0.18
	5	0.007	472	$\alpha \omega^{0.42}$	37	$\alpha \omega^{0.40}$	0.08
GA/MS	25–3	1.033	147	$\alpha \omega^{2.02}$	42	$\alpha \omega^{0.77}$	0.29
	25–5	1.599	144	$\alpha \omega^{2.03}$	45	$\alpha \omega^{0.79}$	0.31
	35–3	1.750	144	$\alpha \omega^{2.02}$	118	$\alpha \omega^{0.90}$	0.82
	35–5	2.390	146	$\alpha \omega^{2.03}$	128	$\alpha \omega^{0.92}$	0.88

structurally fluid characteristics (Sanchez et al., 2002). The viscous component (G'') was greater at the maximum GA concentration, about 700% in the 25% dispersion. Sanchez et al. (2002) studied the behavior viscoelastic on dispersions GA with concentrations between 3 and 32% (w/v), finding that the loss modulus G'' was higher than the storage modulus G' , throughout a wide frequency range. Above $\sim 30 \text{ rad s}^{-1}$, G' became larger than G'' at the highest frequency of the range. In this study, after 24 h at rest, dispersions GA with 25 and 35% also presented $G' > G''$.

Dynamic parameters in MS dispersions presented differences with the two studied concentrations of the order of 70 and 300%. According to the power law, in 3% MS dispersions, G' practically presented no frequency dependence ($G' \propto \omega^{0.03}$) while G'' presented more dependence ($G'' \propto \omega^{0.30}$); 5% dispersions practically presented the same frequency dependence ($G' \propto \omega^{0.42}$ and $G'' \propto \omega^{0.40}$). Yoshimura et al. (1998) determined, in MS dispersions (2.1–3.5%), that G' was approximately 10 times greater than G'' . Likewise, they found that G' presented a lower frequency dependence than G'' . In the present study, in 3% MS dispersions, G' was 8.5 times greater than G'' , while in 5% dispersions, G' was three times greater than G'' . Yoshimura et al. (1988) showed that G' presented a smaller frequency dependence than G'' in MS dispersions; what indicates that behaved as gels, where G' is much bigger than G'' and shows a minimal or null dependence with the frequency (Giboreau et al., 1994).

In GA/MS dispersions with 25/3, 25/5, 35/3 and 35/5% concentrations, G' presented values between 144 and 147 (Pa) and a frequency dependence of $G' \propto \omega^{2.02-2.03}$, while G''

values were greater at higher concentrations both in GA and MS (up to 280%); frequency dependence of G'' presented values of $G'' \propto \omega^{0.77-0.92}$. These exponents were, as those presented in GA dispersions, $=2$ or <1 ; thus these dispersions presented structurally fluid characteristics (Sanchez et al., 2002). Another author (Rodríguez, 2004), mixing MS (3–7%) with gellan gum (0.1–0.3%), found that by increasing the gellan gum concentration, the magnitude of G' and G'' components increased, but these were not higher in gellan gum dispersions. In the present study, after mixing GA with MS, G' values were similar to those of GA dispersions and G'' increased up to three times, which indicates the predominance of a viscous-type viscoelastic behavior. Alloncle and Doublier (1991), when studying mixtures maize starch with guar gum and locust bean gum, found a smaller values of the dynamic moduli, G' and G'' , with regard to dispersions of maize starch. On the other hand (Annable et al., 1994), they found a small value of the elastic modulus (G') in mixtures of potato maltodextrins (15–25%) adding with 10 and 20% GA. As these authors mentioned, an antagonistic effect was probably observed; the hydrocolloid probably acts as a place of discontinuity in the net of amylose, what causes a decrease in the elastic component of mixtures starch–hydrocolloid.

3.2. Rheological creep test

Fig. 4 represents the creep and recovery curves of GA dispersions at 25 and 35% concentrations and MS dispersions at 3 and 5% concentrations and their mixtures. In GA dispersions, at the two studied concentrations, no

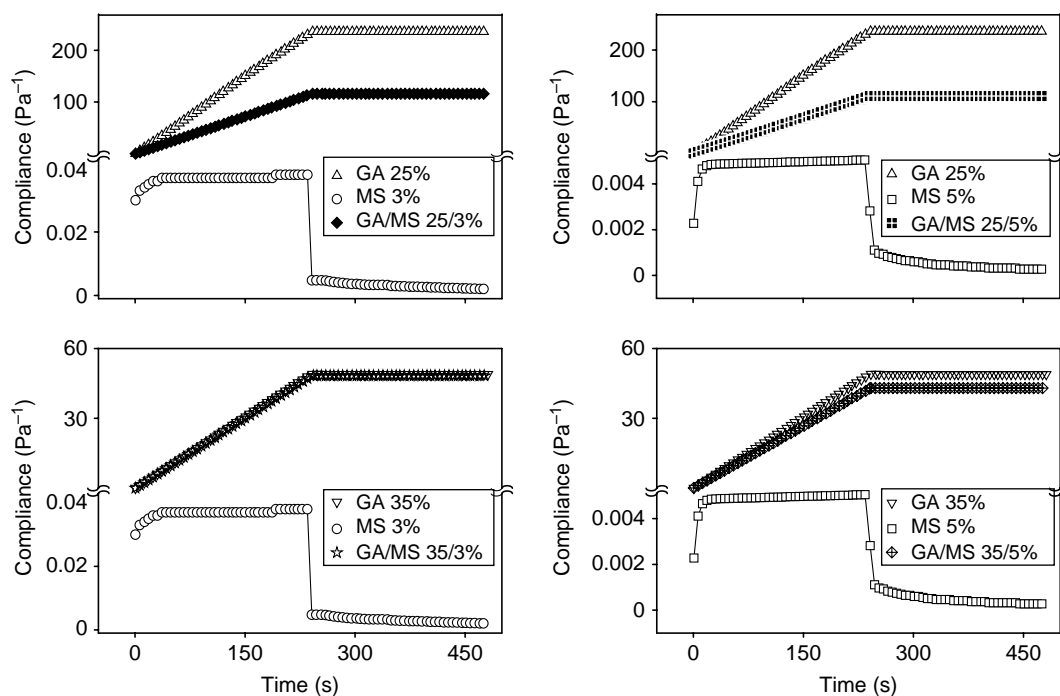


Fig. 4. Creep and recovery curves in dispersions of gum arabic (GA) at 25 and 35% (w/v), maize starch (MS) at 3 and 5% (w/v) and gum arabic/maize starch mixtures (GA/MS).

Table 2

Rheological parameters of the Maxwell model of dispersions of gum arabic, maize starch and gum arabic/maize starch mixtures

Dispersions, % (w/v)		Torque (mN m)	Viscosity, η_0 (Pa s)	Instantaneous compliance, J_0 (Pa ⁻¹) $\times 10^{-2}$	Retarded compliance, J_1 (Pa ⁻¹) $\times 10^{-3}$	Relaxation time, λ_{rel} (s)	Recovery (%)
GA	25	0.0043	1.14	213	56,510	345	0
		0.0069	1.08	93	0.001	47	0
		0.0113	1.20	81	26,430	28	0
		0.0183	1.13	71	8094	20	0
	35	0.0070	6.19	15	3669	183	0
		0.0113	5.91	21	4369	257	0
		0.0184	5.09	32	129	2.34×10^{-2}	0
		0.0298	4.88	33	213	2.35×10^{-2}	0
	MS	0.0016	3.28×10^4	3.98	0.001	5.85	85.25
		0.0026	85×10^4	3.02	7.04	16	94.68
		0.0043	48×10^4	2.97	5.04	21	92.72
		0.0070	26×10^4	3.11	1.79	41	93.15
	5	0.0043	112×10^4	0.18	3.03	450	94.65
		0.0045	40×10^4	0.07	3.63	2.94	88.72
		0.0047	221×10^4	0.09	3.48	3.94	95.54
		0.0050	1688×10^4	1.06	3.35	4.35	96.67
GA/MS	25–3	0.5440	2.22	116	0.001	220	0
		0.7544	2.10	21	0.001	26	0
		1.0423	2.09	19	0.001	125	0
		1.4400	2.08	17	0.001	14	0
	25–5	1.4400	2.17	90	0.001	58	0
		1.6930	2.10	12	23.07	26	0
		1.9904	2.13	14	712.5	134	0
		2.3400	–	–	–	–	0
	35–3	0.8860	5.24	52	0.001	90	0
		1.2247	4.98	3.8	0.001	37	0
		1.6929	5.00	3.2	32.4	25	0
		2.3400	5.13	4.9	644.4	141	0
	35–5	0.5460	5.86	35	0.001	122	0
		1.0415	5.61	2.7	0.001	13	0
		1.9868	5.57	3.6	0.001	2.38×10^{-2}	0
		3.7900	5.55	4.3	37.2	43	0

–, No calculated.

recovery was observed when the applied stress was suspended, while in the MS dispersions, recovery was up to 96.7% at the maximum studied concentration (5%). The GA/MS mixtures at 25/3 and 25/5% presented compliance values intermediate (100 Pa^{-1}) between GA and MS dispersions, while GA/MS mixtures at 35/3 and 35/5% presented compliance values similar to those of GA dispersions, probably as a result of the increase of GA concentration in the mixture that may provide more viscoelastic characteristics.

Table 2 shows the rheological parameters of the Maxwell model for dispersions of 25 and 35% GA, and 3 and 5% MS and their mixtures from four curves for each dispersion obtained with the LVR (0.007–3.79 mN m). GA dispersions (25 and 35%) presented values of limiting viscosity at zero shear rate (η_0) between 1.08 and 6.19 Pa s, respectively, while MS dispersions (3 and 5%) presented very high viscosity values (between 3.28 and 85×10^4 as well as from 40 to 1688×10^4 Pa s). The GA/MS mixtures (25 and 35% GA) presented viscosities between 2.08 and 5.86 Pa s, i.e. viscosities presented by MS dispersions decreased remarkably by adding GA, indicating that the gum may be

preventing the gelatinisation, swelling and disintegration of starch granules (Rodríguez, 2004). Other authors (Tester & Somerville, 2003) studied how different gums (arabic, carrageenan, guar, pectin and xanthan) interact with maize and wheat starches during the gelatinisation process; they conclude that it is apparent that soluble non-starch polysaccharides restrict the gelatinisation and swelling of maize and wheat starches in both a non-starch polysaccharide and concentration-dependent fashion; the effect is most marked as the ratio of solution to starch is decreased; whilst the non-starch polysaccharide probably restrict gelatinisation by reducing hydration of the amorphous regions.

Practically all GA and MS dispersions presented a retarded compliance greater than instantaneous compliance ($J_1 > J_0$), which was contrary to mixtures ($J_0 > J_1$); they need to deform initially before a flow is established, and deform again later. GA dispersions (25 and 35%) presented instantaneous compliance values (J_0) between 71 and 213×10^{-2} and 15 – $33 \times 10^{-2} \text{ Pa}^{-1}$, while values in the MS dispersions (3 and 5%) ranged between 3.02 and 3.98×10^{-2} and from 0.07 to $1.06 \times 10^{-2} \text{ Pa}^{-1}$. J_0 values are inversely proportional to η_0 values in the studied dispersions because

compliance is the opposite of the stress applied. In the GA/MS mixtures, J_0 was greater than J_1 , up to 4000% in 25% GA dispersions; in those with no recovery, J_0 was greater than J_1 , and in MS dispersions, where recovery was present, J_1 was greater than J_0 .

The stress relaxation time (λ_{rel}) for dispersions varied widely between 0.023 and 450 s; this is the time required for the applied stress to decrease to 1/e (approximately 36.8%) of its initial value under constant deformation. Higher values were obtained in dispersions (MS) presenting recovery (2.94–450 s) than in those (GA and GA/MS) without recovery (0.023–345 s). Rayment et al. (1998) performed creep tests in guar gum rice starch mixtures, reporting that as galactomannan concentration increased, λ_{rel} also increased. This tendency was not found in the present study probably because of the purity of the samples used.

4. Conclusion

All the studied dispersions presented a viscoelastic-type rheological behavior; the elastic component was greater than the viscous component ($G' > G''$), which indicates that these are systems with predominantly elastic characteristics. The dependence of dynamic parameters on frequency was greater for the elastic component (G'). The elastic component values were not modified in the mixtures by the presence of MS in contrast to those of the viscous component. The limiting viscosity at zero shear rate (η_0) in the GA/MS mixtures was lower in comparison to MS dispersions, probably because GA prevented complete MS gelatinization. Dispersions of GA and GA/MS mixtures behaved as viscoelastic fluids, in contrast to MS dispersions, which presented a behavior characteristic of viscoelastic solids. The viscosity (η_0) was approximately 6 Pa s for GA and GA/MS dispersions, and 1.12×10^6 Pa s for MS dispersions. Mixture relaxation time (λ_{rel}) was between 0.023 and 220 s and instantaneous compliance (J_0) was greater than retarded compliance (J_1) regardless of MS concentration.

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Glossary

e : logarithm

GA : gum arabic

GA/MS : gum arabic/maize starch mixture

$J(t)$: shear creep compliance

J_0 : instantaneous compliance (Pa^{-1})

J_1 : retarded compliance (Pa^{-1})

J_i : Kelvin element compliance

LVR : linear viscoelasticity region (mN m)

MS : maize starch

n : Kelvin element number

G' : storage modulus (Pa)

G'' : loss modulus (Pa)

t : time elapsed (s)

δ : phase angle

γ : shear strain

i : nth event

λ_{rel} : relaxation time (s)

η_0 : limiting viscosity at zero shear rate (Pa s)

\sum : summation

σ : shear stress (Pa)

σ_0 : initial shear stress (Pa)

ω : angular velocity (Hz or rad s^{-1})