Rheological Behaviour in the Interaction of Lecithin and Guar Gum for Oil-in-Water Emulsions

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Abstract

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The effects of guar gum and lecithin concentrations (1, 0.75, and 0.5%wt) on the stability of oil in water emulsion were investigated. All emulsions can be stabilized at the studied concentrations of stabilizers. The samples tested by steady shear flow and dynamic viscoelasticity tests were carried out to characterize the rheological behaviour of emulsions as influenced by concentration. Emulsions presented a non-Newtonian behaviour type with shear thinning and flow curves that could be described by the Carreau model. The dynamic viscoelastic properties characterized by an oscillatory frequency sweep under small deformation conditions showed fluid-like viscoelastic behaviour. The interaction of the stabilizers in the mixture with each other at the interface appears to play a decisive role for the stabilization of emulsions.

Keywords: Carreau model; guar gum; lecithin; oil-in-water emulsion; rheology; shear thinning; viscoelasticity

Emulsions are part of a thermodynamically unstable system that consists of two immiscible liquids, the dispersed and continuous phases. To obtain kinetically stable emulsions, their formulation requires the incorporation of substances known as emulsifiers and stabilizers (García *et al.* 2014). The emulsion stability could be tuned by using different types of continuous phase (Rousseau & Hodge 2005; Rodríguez-Abreu & Lazzari 2008; Rousseau 2013) or dispersed phase (Pons *et al.* 1993). The use of stabilizers in emulsions provokes an increase in the continuous phase viscosity, and therefore improves the long-term physical stability of the emulsion, slowing the movement of the droplets.

Among polysaccharides, only a few have amphiphilic properties and these have been studied for their emulsion stabilization performance (Dickinson 2003; Nilsson & Bergenståhl 2006). In the case of formulations with polysaccharides, the efficiency depends on the concentration of hydrocolloids in the aqueous phase and on the characteristics of the structure formed by the polymer (McClements 2005). Their ability to stabilize the oil-water interface is dependent not only on the polymer backbone and type, but also on the other ingredients and process parameters. These polysaccharides form a thick adsorbed layer at the oil-water interface, which acts as a protective layer. Synergistic polysaccharide-polysaccharide interactions are attractive commercially because they offer the potential to create new textures and manipulate the rheology of products in the food industry. Also, to meet the demand for ingredients with more specific functionality, significant efforts have been devoted to find new structure-functionality issues (Hayati *et al.* 2016). Previous studies reported the behaviour characterization

of various mixtures of emulsifiers, stabilizers, and polysaccharide-polysaccharide interactions (Fitz-patrick *et al.* 2013; Hayati *et al.* 2016). To the best of our knowledge, there is no work in the literature suggesting or discussing the evolution of the interaction of selected gums, widely addressing steady and dynamic rheological parameters. The main aim of this work was to study the influence of lecithin and guar gum on the rheological and physical stability of emulsion type oil in water.

MATERIALS AND METHODS

Commercial grade sunflower oil was obtained from a local supermarket. Guar gum and lecithin were purchased from Tecnas (Colombia). Distilled water was used to prepare all solutions and emulsions.

Oil-in-water emulsions. Different oil-in-water emulsions were prepared using sunflower oil, lecithin, guar gum, and deionised water to analyse the influence of stabilizers on rheological properties. The 30%wt of sunflower oil emulsion in 70%wt of water at different concentrations of guar gum and lecithin were prepared (Table 1). Guar gum and lecithin concentrations were 0.5, 0.75, and 1%wt for all emulsions.

Solutions were prepared by thoroughly dispersing the desired amount of premix guar gum and lecithin powders in deionized water and stirring at room temperature for hydration. The samples were stirred at 2500 *g* for 20 minutes. The emulsions were prepared using the methods described by Quintana *et al.* (2015) with some modifications. Emulsion continuous phases were formed prior to addition of the oil phase to the premix and homogenization at pH 7.0 ± 0.02, using an Ultra-Turrax disperser (IKA T-25 Basic; Germany) equipped with an S25 N-10ST dispersing tool, at constant temperature of 25°C at 16 800 *g*. Total homogenization time was 30 minutes. Prior to measurements, prepared emulsions were left at room temperature for 48 h to complete the hydration.

The stability of the emulsions was evaluated by transferring 25 ml of the freshly prepared emulsions to cylindrical tubes that were capped and stored at room temperature for 48 hours. After 48 h, the stability of emulsions was evaluated based on emulsification efficiency (E%), which is the ratio between the volume of the emulsified dispersed phase (V_e) (read 90 min after preparation) and the initial volume of the dispersed phase (V_o), calculated by Equation (1) (Castro 2007):

$$E\% = V_e/V_o \tag{1}$$

Rheological evaluations. Steady shear and small deformation oscillatory measurements were carried out using a Modular Advanced Rheometer System MARS 60, HAAKE (Thermo-Scientific, Germany), equipped with a coaxial cylinder (inner radius 12.54 mm, outer radius 11.60 mm, cylinder length 37.6 mm). The temperature was fixed, using a Peltier system, at 25°C and each sample was equilibrated at 600 seconds before the rheological test to have the same recent past thermal and mechanical history.

The continuous shear test was performed at 25°C, over a shear rate between 0.001 and 1000 s^{-1} to measure the apparent viscosity (η). Stress sweeps from 0.01 Pa to 1000 Pa at 1 Hz were performed for all systems to estimate the dynamic linear viscoelastic range. A frequency sweep test from 0.01 rad/s to 100 rad/s was performed selecting a stress well within the linear range. All measurements were done in duplicate.

Statistical analysis. All measurements per batch were done in duplicate. A one-way ANOVA, using Minitab 18 software (Minitab Inc., USA), was applied to study the effect of the interaction of lecithin and guar gum on the rheological parameters. The significance level was set at 95%.

RESULTS AND DISCUSSION

Standardisation of emulsions. The emulsions were prepared using different concentrations, and mixing lecithin and guar gum at pH 7.0 ± 0.02 while applying the same levels of emulsification velocity and using a homogenizer to reduce particle size and entanglement for emulsion stability (Quintana *et al.* 2015). Nine formulations were used that are shown in Table 1. The emulsions obtained after the homogenization processes showed high emulsification efficiencies (E%) with a value to 100% for all samples, which demonstrates the high stability of the system until 48 h after preparation and how the samples maintained their physical characteristics until rheological analysis.

Steady-state viscous flow. Figure 1 shows experimental steady shear flow curves of emulsions prepared with lecithin and guar gum in the studied shear range. Emulsions showed a strong shear thinning behaviour, with a variation in η about three

Table 1. Steady shear rheological parameters of emulsions and the yield stress values

Sample	guar gum (%wt)	lecithin (%wt)	η_0 (Pa·s)	η_∞ (Pa·s)	λ_c (s)	α	η	R^2	τ_c
					Carreau model				
F1	1.0	1.0	164.448 ± 0.229	1.80E-5 ± 0.007	12.144 ± 0.452	1.231 ± 0.017	0.266 ± 0.017	0.999	33.073
F2	1.0	0.75	62.660 ± 0.307	0.002 ± 0.135	8.222 ± 0.311	0.968 ± 0.033	0.335 ± 0.023	0.999	32.107
F3	1.0	0.5	143.089 ± 0.327	2.06E-8 ± 0.308	12.204 ± 0.280	1.215 ± 0.049	0.298 ± 0.024	0.999	32.693
F4	0.75	1.0	42.997 ± 0.160	9.75E6 ± 0.062	6.044 ± 0.444	0.807 ± 0.017	0.322 ± 0.018	0.999	19.555
F5	0.75	0.75	61.299 ± 0.489	1.29E-41 ± 0.154	8.056 ± 0.113	0.833 ± 0.037	0.304 ± 0.035	0.999	19.865
F6	0.75	0.5	54.564 ± 0.169	1.51E-5 ± 0.079	7.162 ± 0.405	1.008 ± 0.022	0.296 ± 0.018	0.999	20.187
F7	0.5	1.0	14.566 ± 0.223	1.70E-2 ± 0.066	1.269 ± 0.615	0.511 ± 0.033	0.236 ± 0.100	0.998	7.6731
F8	0.5	0.75	12.326 ± 0.174	1.49E-17 ± 0.060	1.213 ± 0.563	0.527 ± 0.033	0.247 ± 0.098	0.998	11.171
F9	0.5	0.5	10.591 ± 0.047	1.223 ± 0.024	1.630 ± 0.224	0.651 ± 0.017	0.295 ± 0.032	0.999	11.499

η_0 – zero shear rate viscosity; $\eta\infty$ – infinite shear rate; λc – time constant of Carreau; α – > 0; η – behaviour index of power law; R^2 – correlation coefficient; τ_c – yield stress

orders of magnitude. From viscosity curves, it can be seen that an increased gum concentration resulted in increased η, similar to results found in the literature (Seyed *et al.* 2016). Higher solids contents generally cause an increase in viscosity, mainly due to increased molecular entanglements and interfacial film formation (Maskan & Göğüş 2000). Small shear rates and η exhibited a Newtonian plateau followed by a shear-thinning zone. In this region of concentration, the degree of shear thinning was increased in relation to the concentration of guar gum (1 > 0.75 > 0.5%wt), quantified by the slope of the viscosity/shear rate curve, which exhibited a concentration dependence.
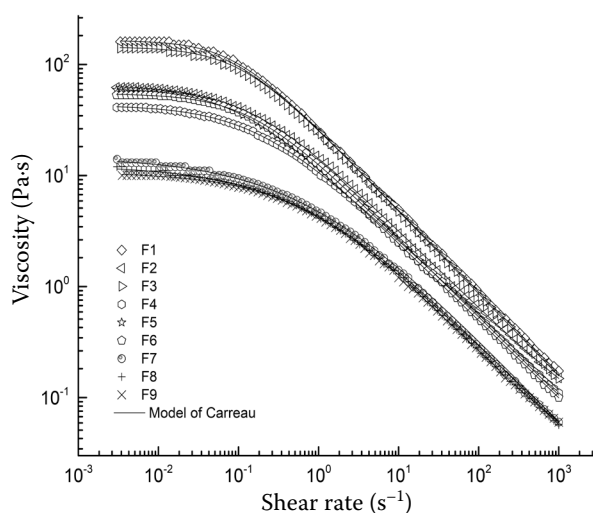

Figure 1. Apparent viscosity of emulsions stabilized with mixing guar gum and lecithin at different concentrations (1, 0.75, and 0.5 wt%) after 48 h of storage adjusted to the Carreau model

Apparent viscosity of dispersions in semi-dilute conditions is therefore related to the molecular structure that influences possibilities of interactions (Kapoor *et al.* 1994; Morris *et al.* 1981; Rinaudo 2001). As a typical behaviour of polysaccharide systems, the shear-thinning behaviour is in agreement with previous reports for galactomannans (Nwokocha & Williams 2012 ;Albuquerque *et al.* 2014; Hussain *et al.* 2015). At small shear rates, long chains of the galactomannan molecules tend to present a coiling structure; the relatively high η may contribute to the development of an interlocking between the polymer chains composed of these coiling structures. Thus, at high shear rates, the induced successive increasing force might result in interlocking deformation, linkage breakdown, and consequently, an η drop (Jian *et al.* 2014). The lecithin did not have a high influence on shear thinning behaviour due to a superposition of the curves that can be observed; a further increasing lecithin concentration raises yield stress and does not lead to a further reduction of viscosity (Nebesny & Żyżelewicz 2005; Schantz & Rohm 2005; Karnjanolarn & McCarthy 2006; Sokmen & Gunes 2006; Afoakwa *et al.* 2007). A common explanation for the lecithin-induced decrease of yield stress and η in oil-based suspensions is that the surface active components adsorb at the surface of suspended particles (Weyland & Hartel 2008), thus causing a smoothing of the particle surface and reduced friction between particles (Dedinaite *et al.* 1998).

The Newtonian plateau region occurred at low shear rates and has a constant viscosity value, followed by the shear-thinning region and infinite shear

rate viscosity at a high shear rate; generally, this behaviour could be described using the model of Carreau (1972) that represents the fluid viscosity as a function of shear rate γ as shown in Equation (2), where η_∞ is the infinite shear rate viscosity, η_o, is the zero shear rate viscosity, λ_c is the time constant of Carreau, a > 0, and *n* is the behaviour index of power law.

$$\eta = \eta_\infty + (\eta_o - \eta_\infty)\,[1 + (\lambda_c \times \gamma)^a]^{[(n-1)/a]} \qquad (2)$$

Fitting parameters are shown in Table 1. In the case of the zero shear rate viscosity η_o, it increased proportionally to the increase of guar gum and lecithin concentration. The time constant of Carreau (λ_c) increased with the percentage of guar gum, but no relationship with lecithin was found for concentrations of 0.75 and 1%, although in the case of 0.5%, the similar value with a variation of SD ± 0.22 was present. The behaviour index (n) decreased with the increase of guar gum concentration, but in the case of lecithin, it was similar. The correlation coefficients (R^2) between the viscosity predicted by the Carreau model and the experimentally measured viscosity were higher than 0.999 for all samples, indicating that this model was a good fit to the experimental results obtained. Thus, the shear thinning parameters of emulsions present a relation with the concentration of gums.

According to the ANOVA results, the effect of lecithin on the rheological parameters depends on the guar gum concentration in emulsion formulations, as indicated by the significant interaction between the effects (Table 2). The infinite shear rate viscosity, η_o (time constant, λ_c) and behaviour index; *n*-values increased when the guar gum proportion increased and tended to be constant with different percentage of lecithin. In the case of the zero shear rate viscosity, η_∞, the behaviour was the same with the values decreased with the increase of lecithin and guar gum. According to the results, when the

guar gum concentration in the emulsion increased, the sample presented more resistance to flow and the reduction in viscosity took place at lower shear rates and was more pronounced.

Stress sweep. Many materials found in daily life exhibit properties characteristic of either solids or liquids, depending on the imposed stress. At small stresses, these materials deform essentially in an elastic manner, but flow once a critical stress is exceeded; this critical value is called the yield stress (τ_c), and materials exhibiting a yield stress are called yield stress materials (Dinkgreve *et al.* 2016). Many methods have been proposed for determining the yield stress; it has been demonstrated that variations of more than one order of magnitude can arise, however, are dependent on the method used and the handling of the sample. In this case, we used the method described by Rouyer *et al.* (2005) to determine the intersection of the horizontal line representing the behaviour of elastic modulus (G') well below the yielding point, with the power law equation representing the behaviour of G' well above the yielding point. Figure 2 shows the amplitude performed over the stress range of 0.01 Pa to 1000 Pa, at 1 Hz of frequency and 25°C to determine the linear viscoelastic region, where two different regions were observed: (I) linear viscoelastic region where G' and loss modulus (G") were almost constant and (II) non-linear region in which G' and G" started to decrease. In the linear viscoelastic range, G' was greater than G' at all samples, also, both moduli increase with increasing the percentage of guar gum. The linear viscoelastic range can be considered as a measure of gel strength. In the amplitude test, stronger gels have a more ex-
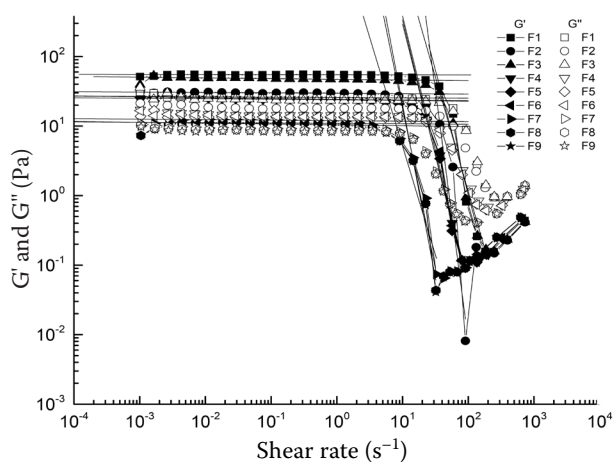
Table 2. ANOVA of two factors with the interaction for the Carreau model parameters

Parameter	A – guar gum		B – lecithin		Interaction A × B	
	F	*P*	*F*	*P*	*F*	*P*
η_0	243 437.02	0.001	19 158.39	0.001	29 346.98	0.001
η_∞	27.28	0.001	26.19	0.001	26.62	0.001
λ_c	426.56	0.001	6.85	0.007	16.42	0.001
α	704.59	0.001	76.35	0.001	18.52	0.001
η	0.79	0.470	1.82	0.192	1.16	0.364

Figure 2. Stress sweep of lecithin and guar gum emulsions
G' – storage modulus; G" – loss modulus

tensive linear viscoelastic region in comparison with weak gels (Steffe 1996). In Table 1, the yield stress (τ_c the stress in which G' decreases with an increase of stress sharply) is shown and depends on the molecular architecture of the food polymer molecules (Heldman & Lund 2007). The τ_c increased with the guar gum concentration for all samples. For the samples F1, F4, and F7, when the concentration of lecithin was 1.0%wt and the guar gum concentration decreased (1.0, 0.75, and 0.5%wt), the τ_c values were 33.07, 19.56, and 7.67 Pa, respectively, showing a significant decrease. Thus, lecithin does not influence in the cases when the gum concentration was 1.0%wt and decreased concentrations of lecithin (1.0, 0.75, and 0.5%wt) had the τ_c values of 33.07, 32.10, and 32.69 Pa, respectively, with a variation of ± 0.486. The interaction with guar gum changed the τ_c, and consequently increased the time required for new entanglements to replace those disrupted by externally imposed deformation in a small amplitude oscillatory test.

Frequency sweep. The amplitude test performed over the studied stress range determined the linear viscoelastic region, where two different regions were observed: a linear viscoelastic region where G' and G" were almost constant and a nonlinear region in which G' and G" started to decrease. In the linear viscoelastic range, G' was greater than G" for all samples, also, both moduli increased with increasing the percentage of guar gum.

Mechanical spectra obtained from the frequency sweep test for emulsions at different lecithin and guar gum concentrations (0.5, 0.75, and 1%wt) prepared at room temperature are shown in Figure 3. The viscoelastic behaviour of emulsions is also influenced by the structure of polymers. At low oscillation frequencies, G" is higher than G', whereas the reverse is observed at higher frequencies with the crossover observed depending on the concentration of gums. Beyond the shift point, the oscillation frequency becomes too high which may result from the higher entanglement structural strength, thus galactomannan chains cannot be dissociated. The intersection between G' and G" curves increased in relation to the percentage of guar gum (1 < 0.75 < 0.5%wt) and the corresponding frequency is called the relaxation rate denoted as ω (G' = G") in the Maxwell model (Wayne *et al.* 2005). This means that it quickly had a viscosity dominance behaviour generated by entanglements and hyper-entanglements that dissociate less easily. The inverse of the crossover frequency (G' > G") can be regarded as the relaxation time, denoted as T (G' = G") of the network. The relaxation time indicates the elas-
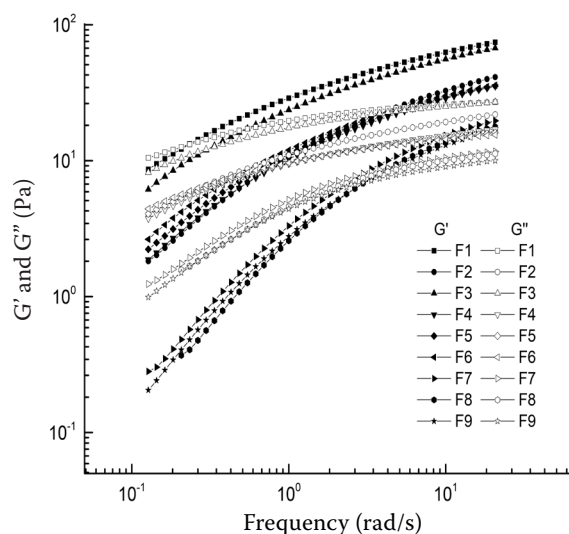

Figure 3. The storage modulus (G') and loss modulus (G") as a function of frequency for oil-in-water emulsions stabilized with different concentrations of mixing guar gum and lecithin

ticity of these fluids. This effect may be attributed to modifications in the interactions among protein side chains and polysaccharide molecules. Thus, the relaxation time can be used as an index for the number and strength of the temporary crosslink junctions. When the network quickly relaxes, a proponent suspended in the fluid can settle more frequently based on the viscous region of the fluid (Loveless *et al.* 2011). Similar results were obtained by (Wang *et al.* 2014). Other food emulsions (Quintana *et al.* 2002; Bengoechea *et al.* 2006) or aqueous dispersions of protein-polysaccharide systems (Aguilar *et al.* 2011) also showed this entanglement region. The interactions of hydrocolloids with other components coexisting in food matrices allow increased flexibility for food research development. The stabilizers are indispensable in the design of food structure and functionality that are demanded by the consumer in modern society.

Ascertainment of the Cox-Merz rule. The Cox-Merz rule (Equation 3) is applied to correlate dynamic and steady shear properties of the solutions, and the magnitude of complex viscosity (η^*) and apparent shear viscosity (η) is compared at equal values of shear rate and frequency (Barnes *et al.* 1989; Steffe 1996).

$$\eta^* = \eta \times (\gamma) \times (\omega = \gamma) \qquad (3)$$

This rule has been studied for many polymers, solutions, and complex food systems (Rao & Steffe 1992; Tiziani & Vodovotz 2005; Yasar *et al.* 2009)
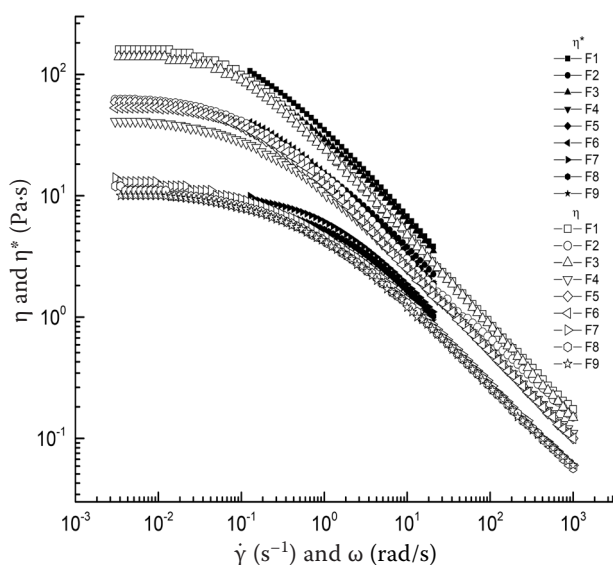
Figure 4. Comparison of complex (η^*) and apparent viscosity (η) in the function of angular frequency (ω) and shear rate ($\dot{\gamma}$)

and provides a sensitive practical test to detect the presence of aggregates in the solutions. To examine the applicability of the Cox-Merz rule (Equation 3), the η and complex viscosity (η^*) of emulsions were plotted against the shear rate (γ) and angular frequency (ω), respectively (Figure 4).

It was observed that the magnitudes of η were equal to η^* within the whole angular frequency or shear rate ranges. The values of η^* can be observed at intermediate angular frequency or shear rate ranges. This result is reported by Martín-Alfonso *et al.* (2018), where the guar gum solutions obeyed the Cox-Merz rule, although a divergence in behaviour was observed at high rates for the more concentrated solutions, probably due to entanglements. The behaviour is related to the elastic gel-like structure, which is not affected during oscillatory measurements.

CONCLUSIONS

Emulsions were stabilized with mixing of guar gum and lecithin at a percentage less than 1%wt. The studied emulsions exhibited non-Newtonian behaviour at the experimental concentration and all the systems showed a shear thinning behaviour that fit the Carreau model. The dynamic viscoelastic properties characterized by an oscillatory frequency sweep under small deformation conditions showed fluid-like viscoelastic behaviour. The yield stress

depended on the molecular architecture of gums, showing an increase with the guar gum concentration although lecithin did not exert any influence. The interaction with the guar gum changed the yield stress and consequently increased the time required for new entanglements to replace those disrupted by externally imposed deformation in the small amplitude oscillatory test when their storage modulus and loss modulus increased at angular frequency. At low frequency, emulsions presented a viscous behaviour, whereas the reverse was observed at higher frequencies. Complex viscosity decreased as the angular frequency decreased, whereas it increased with gum concentrations. The correlation between dynamic and steady shear properties with the Cox-Merz rule presented that the magnitudes of apparent viscosity were equal to complex viscosity within the whole angular frequency or shear rate ranges. The interaction of hydrocolloids with other components coexisting in food matrices allows increased flexibility for food research development. The stabilizers are indispensable in the design of food structure and functionality that are demanded by the consumer in modern society.

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