

# Oscillatory Measurements for Salad Dressings Stabilized with Modified Starch, Xanthan Gum, and Locust Bean Gum

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**ABSTRACT:** The rheological properties of low-oil-content food emulsions were analyzed with oscillatory tests within the linear viscoelastic region. The formulations of these salad dressings usually include modified starch because of its low cost and the special creamy texture that it affords. The combination of starch with other natural gums may improve the quality of the product. A reference emulsion containing 4% modified starch and four other formulations in which the starch was partially replaced by xanthan gum (0.4%), locust bean gum (0.4%), and synergistic blends of these gums (0.03 + 0.03% or 0.1 + 0.1%) were formulated. Gels before emulsifi-

cation were studied for comparative purposes. All systems showed weak gel behavior. An empirical equation fitting the storage and loss modulus dependence on the frequency was applied. The loss tangent allowed us to compare the viscoelastic character of all the systems. The complex viscosity followed the power law in all cases, and the generalized Cox–Merz rule was applied. © 2006 Wiley Periodicals, Inc. *J Appl Polym Sci* 102: 897–903, 2006

**Key words:** hydrogels; polysaccharides; viscoelastic properties

## INTRODUCTION

In oil-in-water food emulsions such as mayonnaise and salad dressings, oil droplets are usually stabilized by proteins or mixtures of proteins and emulsifiers and frequently appear suspended in a continuous gel phase containing a biopolymer that acts as a thickener.<sup>1–3</sup> On the other hand, in mayonnaises with an oil concentration of less than 60%, rheological properties can be modified by the replacement of the oil with chemically modified starch and the incorporation of a stabilizer such as a galactomannan or xanthan into the recipe.<sup>4–6</sup>

Starch is a polysaccharide of great importance because it is a major source of carbohydrates in the human diet and is widely used in the food industry as a thickening, stabilizing, and gelling agent. Starch granules are insoluble in cold water, although they swell and rupture when heated above a certain temperature. Gelatinized starch consists of a continuous phase enriched in amylose and a disperse phase (starch granules containing mainly amylopectin). With cooling and further storage, a composite gel can be formed.<sup>7,8</sup> However, uses of native starch are limited,

as pastes present the problem of retrogradation, together with syneresis and slow resistance to shear treatment, for example.<sup>9</sup> To improve the physical and chemical properties of these pastes, starches have been chemically modified. Such chemical modification includes acid hydrolysis, oxidation, etherification, esterification, and crosslinking.<sup>8,10</sup> The currently most widely used approaches in the food industry are combinations of crosslinking and chemical substitution (acetyl or hydroxypropyl groups).<sup>11</sup>

Hydrocolloids are used in starch-based products to improve the stability, modify the texture, facilitate processing, and generally improve the overall quality.<sup>7,12</sup> They have been shown to influence the gelatinization and rheological properties of starches. The addition of hydrocolloids (e.g., galactomannans and xanthan gum) increases the viscosity of starch pastes, influences the retrogradation rate, and, most importantly, prevents the syneresis of starch.<sup>4,13</sup>

In this work, we have studied the parameters obtained from dynamic viscoelasticity tests [storage modulus ( $G'$ ), loss modulus ( $G''$ ), loss tangent ( $\tan \delta$ ), and complex viscosity ( $\eta^*$ )] for low-oil-content emulsions (35%) containing different modified-starch/hydrocolloid mixtures. A reference emulsion containing only modified starch has been compared with others formulated with the substitution of 10% of the starch with xanthan gum or locust bean gum. On the other hand, taking into account the well-known synergism of these two gums,<sup>4,10,14</sup> we have also studied

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two additional formulations containing a synergistic combination (1:1) of the two, which implies a lesser amount of total gums (only 1.5 and 5% of the reference amount of starch).

In a previous article, the flow and thixotropic behavior was studied.<sup>15</sup> The aim of this work is to analyze the influence of these hydrocolloids on the viscoelastic properties of the emulsions. It is well known that these emulsions exhibit a weak, gel-like, viscoelastic behavior that can be attributed to the network of droplets of the disperse phase.<sup>16</sup> To better compare the relative effects of the gums, the gels prepared before final emulsification have also been considered.

### EXPERIMENTAL

Acetylated distarch adipate (C\*Tex 06214) was acquired from its manufacturer (Cargill, Barcelona, Spain). Xanthan gum was provided by Jungbunzlauer GmbH (Barcelona, Spain), whereas locust bean gum was obtained from Industrial Garrofera Valenciana (Valencia, Spain). Pasteurized, liquid, salted egg yolk, sunflower oil, and wine vinegar (equivalent acetic acid concentration = 10%) were kindly supplied by Hijos de Ybarra (Seville, Spain). Commercial salt and sugar, preservatives (potassium sorbate and sodium benzoate), and deionized water were also used.

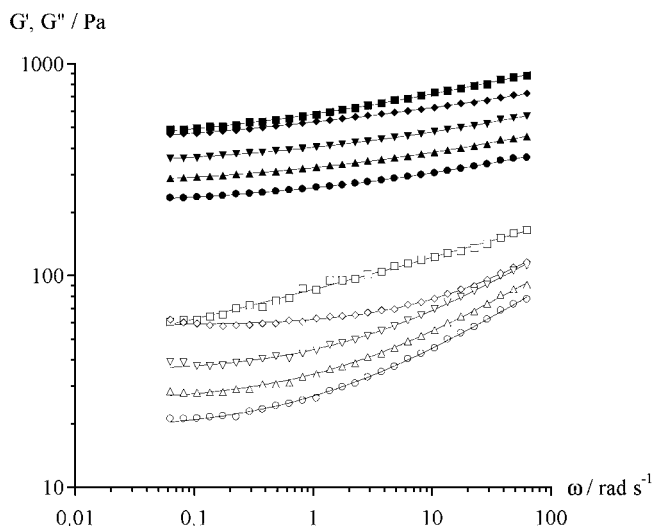
An emulsion formulation was adapted from the commercial composition of low-oil mayonnaise containing 4 wt % modified starch (MS) as a stabilizer and 34 wt % vegetable oil. The total concentration of the stabilizer was maintained at 4 wt %, the MS fraction being partially replaced by xanthan gum, locust bean gum, or a mixture of the two gums. Table I indicates the hydrocolloid contents of the different formulations tested, together with the notations used in the text to refer to each of the emulsions.

The final manufacturing method chosen comprised two stages: the first dealt with hydrocolloid gel formation and the second dealt with emulsion manufacture.

In the first stage, the solid ingredients (sugar, salt, preservatives, and hydrocolloid powders) were mixed, dispersed in water (stirring for 5 min at room temperature), and heated to 95°C with stirring for 15 min

**TABLE I**  
System Notation for Both the Gels and Emulsions and the Hydrocolloid Contents

System notation	Hydrocolloid content (wt %)
MS	4% modified starch
LBG	3.6% MS and 0.4% locust bean gum
XG	3.6% MS and 0.4% xanthan gum
003	3.94% MS, 0.03% locust bean gum, and 0.03% xanthan gum
01	3.8% MS, 0.1% locust bean gum, and 0.1% xanthan gum



**Figure 1**  $G'$  (filled symbols) and  $G''$  (open symbols) as functions of  $\omega$  for all the emulsions: (●, ○) MS, (■, □) LBG, (◆, ◇) XG, (▲, △) 003, and (▼, ▽) 01.

to ensure homogeneous gelation of the starch. After cooling to room temperature, the gels were kept at 5°C for 24 h.

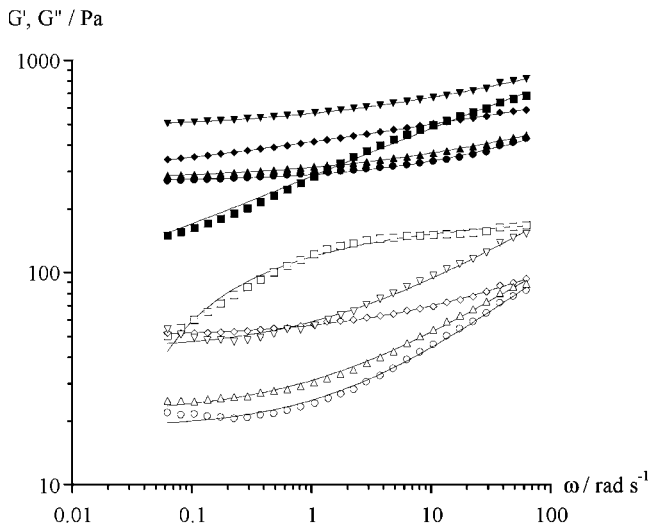
Finally, the liquid egg yolk was added to the gel, and emulsification was carried out with an Ultraturax (Ika, Germany) T-50 rotor-stator homogenizer. This process was performed at 10°C at a homogenization speed of 5000 rpm for 7 min. Oil was added slowly during the first 3 min, and vinegar was added at the end. The final emulsions thus prepared were stored at 5°C until the measurement. Three batches of each formulation were prepared.

Oscillatory measurements were carried out with an RS1 rheometer (Haake, Vreden, Germany) at 25°C with serrated plates (35-mm diameter and 1-mm gap). To determine the linear viscoelastic region, stress sweeps between 0.1 and 100 Pa were performed. Sweep-frequency tests were recorded at a constant stress amplitude (3 Pa for emulsions and 0.5–2 Pa for gels) in the frequency ( $\omega$ ) range of 0.01–10 Hz. The sample was allowed to relax for 20 min before the measurement.

For the emulsions, and because the differences were not significant, the mean values of the results obtained for each prepared batch were considered. To allow a comparison, the gels before emulsification were also analyzed.

### RESULTS AND DISCUSSION

The experimental values of the mechanical spectra for all the systems studied, obtained in the region of linear behavior, are shown in Figures 1 (emulsions) and 2 (gels). In both the emulsions and gels, the behavior was predominantly more elastic because  $G'$  was greater than  $G''$  in the entire  $\omega$  interval studied.



**Figure 2**  $G'$  (filled symbols) and  $G''$  (open symbols) as functions of  $\omega$  for all the gels: (●, ○) MS, (■, □) LBG, (◆, ◇) XG, (▲, △) 003, and (▼, ▽) 01.

This could be clearly seen from  $\tan \delta$  ( $G''/G'$ ; Fig. 3) because  $\tan \delta$  was less than 1 in all cases. Because the ratio of  $G''$  to  $G'$  ( $\tan \delta$ ) was greater than 0.1, the samples were not true gels but presented a structure between those of a concentrated biopolymer and a true gel. They therefore could be characterized as weak gels, this being a typical behavior of dressings and emulsions.<sup>7,17,18</sup>

The continuous lines in Figures 1 and 2 correspond to the fits of viscoelastic moduli  $G'$  and  $G''$  to empirical functions dependent on  $\omega$ :

$$G' \text{ or } G'' = K_1 + K_2 \exp(10 - K_3 \omega^{-p}) \quad (1)$$

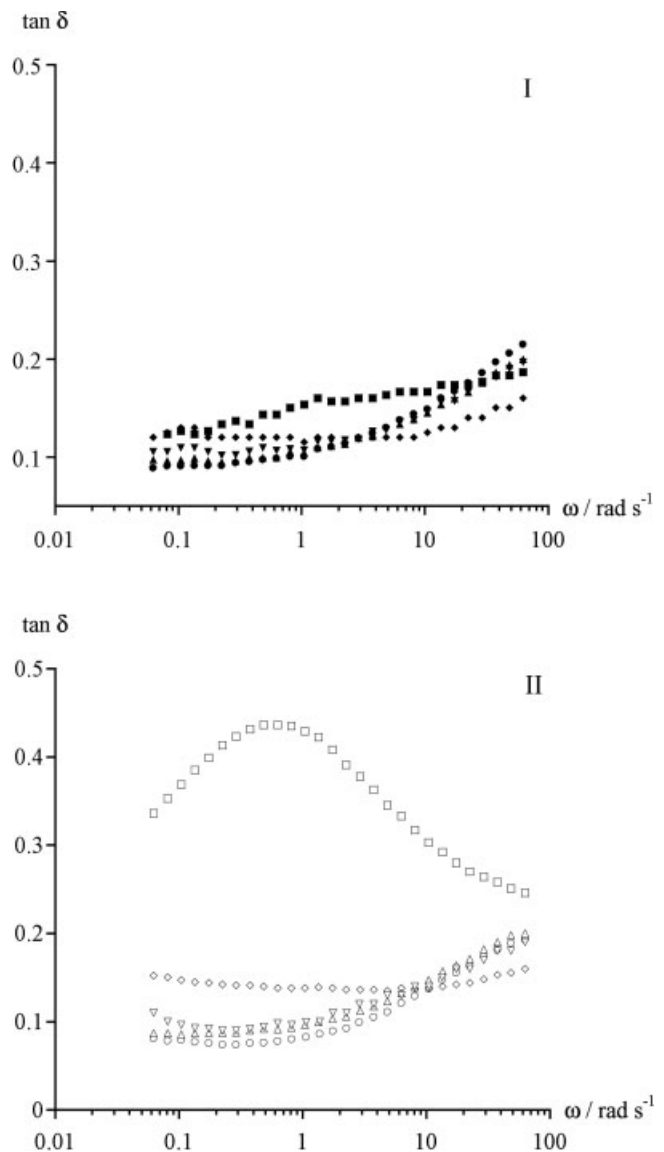
where  $G'$ ,  $G''$ ,  $K_1$ , and  $K_2$  are expressed in pascals and  $K_3$  is expressed in  $s^{-p}$  units,  $p$  being a nondimensional parameter.

Equation (1) is valid in the  $\omega$  interval studied (0.01–10  $s^{-1}$ ), and in principle it would not be extrapolable to other  $\omega$  values. In all cases, we considered  $K_2 = 1$  Pa, except for the LBG gel, because  $G''_{\text{LBG}} = f(\omega)$  presents a convex curvature, in contrast to the rest. Therefore, in the latter case, we considered  $K_2 = -1$  Pa. In this way, all fits made by the least-squares method yielded a correlation coefficient ( $r$ ) greater than 0.997. The values of  $K_1$ ,  $K_3$ , and  $p$  for all the gels and emulsions studied, together with the corresponding uncertainties, are reported in Table II.  $K_1$  is a parameter providing information on the value of both moduli for low  $\omega$  values. For interpretation, it is clearly necessary to take into account that eq. (1) would not be extrapolable below  $\omega = 0.06$  rad/s. On the other hand,  $K_3$  and  $p$  provide information on the variation of the moduli with  $\omega$ .

The replacement of some of the starch by gums always increased the value of both moduli,  $G'$  and

$G''$ , this increment increasing with the proportion of gum added. This agrees with the observations of other authors.<sup>12,18</sup>

In the case of synergic mixtures, with a lower proportion of hydrocolloids, the emulsion spectra are parallel to those of the systems containing only starch. This is clearly reflected by the values of  $K_3$  and  $p$  in the table, which were very similar for these three types of systems; that is, the dependence on  $\omega$  was very similar. The effect was more accentuated in the emulsions but was also present in the gels. It therefore can be deduced that the emulsification process does not modify the viscoelastic behavior, which is dominated by the continuous phase. However, the values of  $K_1$  increased with increasing hydrocolloid concentration:  $K_1(\text{MS}) < K_1(003) < K_1(01)$ .



**Figure 3**  $\tan \delta$  as a function of  $\omega$  for (I) emulsions (filled symbols) and (II) gels (open symbols): (●, ○) MS, (■, □) LBG, (◆, ◇) XG, (▲, △) 0.03(LB + XG), and (▼, ▽) 0.1(LB + XG). For comparative effects, the same scale has been used.

TABLE II  
Values of the Parameters for the Dynamic Moduli Fitted to  $G'$

	$G'$ (Pa)			$G''$ (Pa)		
	$K_1$ (Pa)	$K_3$ ( $s^{-p}$ )	$p$	$K_1$ (Pa)	$K_3$ ( $s^{-p}$ )	$p$
Emulsion						
MS	214 ± 2	6.18 ± 0.04	0.052 ± 0.001	18.9 ± 0.3	7.92 ± 0.04	0.071 ± 0.001
LBG	412 ± 9	4.88 ± 0.06	0.059 ± 0.002	30 ± 5	6.0 ± 0.1	0.037 ± 0.002
XG	391 ± 8	5.05 ± 0.06	0.045 ± 0.002	58.6 ± 0.4	8.77 ± 0.09	0.094 ± 0.003
003	264 ± 2	5.91 ± 0.04	0.053 ± 0.001	25.7 ± 0.4	7.85 ± 0.05	0.073 ± 0.002
01	329 ± 3	5.68 ± 0.04	0.056 ± 0.001	35.5 ± 0.5	7.78 ± 0.05	0.078 ± 0.002
Gel						
MS	273 ± 2	7.1 ± 0.1	0.087 ± 0.004	18.9 ± 0.5	8.18 ± 0.08	0.083 ± 0.002
LBG	65 ± 2	4.62 ± 0.07	0.064 ± 0.001	181 ± 5	5.87 ± 0.09	-0.053 ± 0.004
XG	228 ± 8	4.77 ± 0.04	0.036 ± 0.001	50.6 ± 0.4	7.82 ± 0.07	0.066 ± 0.002
003	277 ± 2	6.40 ± 0.04	0.065 ± 0.001	22.2 ± 0.6	8.21 ± 0.06	0.074 ± 0.002
01	487 ± 4	5.66 ± 0.06	0.073 ± 0.002	44 ± 2	7.3 ± 0.1	0.079 ± 0.003

$G'' = K_1 + K_2 \exp(10 - K_3\omega^{-p})$ ;  $K_2 = 1$  in all cases, except for the LBG gel ( $K_2 = -1$ ).

Thus, it can be stated that synergistic mixtures of LBG and XG added to starch do not modify the structure of the system significantly, and only its consistency increases. This is clearly reflected by the  $\tan \delta$  values, which are practically coincident in all these systems, both emulsions and gels.

However, this is not the case for the systems with greater xanthan gum and locust bean gum concentrations. In these systems, a modification of the internal structure may indeed be presumed because the spectra exhibited different evolutions with  $\omega$ . In the case of the XG emulsion,  $G'$  was almost constant until about 6 rad/s. This would represent a more structured system, as can be clearly seen from the spectra of the gels, for which  $G'$  and  $G''$  were more parallel than in the case of the starch pastes, with behavior more typical of xanthan gels.<sup>7</sup>

In contrast,  $G''$  of the LBG emulsion showed practically constant growth over the logarithmic scale; that is, the increase proved to be nearly a power law with  $\omega$ . The influence of the structure of the corresponding gel is apparent in Figure 2. In this case,  $G'$  presents a much steeper slope than the other gels, and  $G''$  appears convex in shape. This change in the curvature of  $G''$  in the LBG gel is reflected by the negative sign of  $p$ , which is shown in Table II. This drastic change in the spectrum of the gel upon the replacement of some of the starch with LBG was already studied by Kulicke et al.,<sup>19</sup> with the obtainment of totally analogous results at similar proportions.

These differences in the curvatures of  $G''$  are reflected by parameter  $p$  in Table II. The value corresponding to the LBG emulsion was the smallest of all, giving an idea of its less concave nature. In contrast, the  $p$  value for XG was greatest, thus implying increased concavity (greater variation at the higher  $\omega$  values).

Figure 3 shows these differences in the viscoelastic behavior more clearly. The value of  $\tan \delta$  is more constant with  $\omega$  over the entire interval in both the

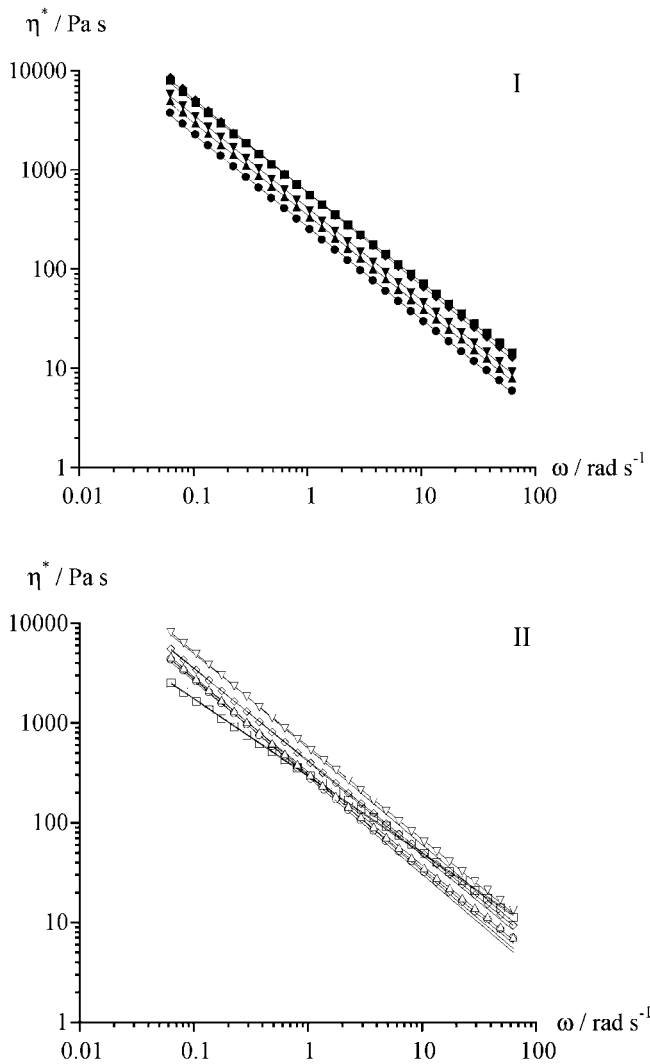
gels and emulsions of the XG system. This indicates the predominance of xanthan gum in the observed viscoelastic behavior. However, the concavity of  $G''$  gives rise to a maximum  $\tan \delta$  value for the LBG gel.<sup>19</sup> Likewise, it is clear that the LBG gel is markedly more fluid in behavior than the rest, as can be observed from the greater dependence of  $G'$  on  $\omega$  and the clearly greater values of  $\tan \delta$ .

On the other hand, in these two types of systems, the values of  $K_1$  are greater than those of the mixtures, except in the case of  $K_1$  for  $G'$  of the LBG gel. This is due to the greater total amount of starch replaced, indicating that the expected synergic effect is not obtained when these gums act without interacting with the rest of the components present in the systems studied.

Equation (1), together with Table II, allows us to determine the values of  $G'$  and  $G''$  of the different systems studied, at any value of  $\omega$  between 0.01 and 10 Hz, in a relatively simple manner. The literature offers very complicated reiterative fits to the generalized Maxwell model that lead to relaxation spec-

TABLE III  
Power Fits for  $G'$  and  $\eta^*$  as Functions of  $\omega$

	$G' = K\omega^{n'}$			$\eta^* = M\omega^{n-1}$		
	$K$ (Pa)	$n'$	$r$	$M$ (Pa/s <sup>n</sup> )	$n$	$r$
Gel						
MS	335.7	0.059	0.946	302.7	0.061	0.999
LBG	428.9	0.234	0.996	298.1	0.227	0.999
XG	483.5	0.080	0.999	421.6	0.081	0.999
003	361.5	0.062	0.974	324.9	0.064	0.999
01	658.6	0.068	0.978	584.5	0.070	0.999
Emulsion						
MS	299.9	0.066	0.985	267.7	0.068	0.999
LBG	697.8	0.089	0.993	598.6	0.091	0.999
XG	607.4	0.065	0.994	594.5	0.061	0.999
003	374.1	0.067	0.987	350.1	0.069	0.999
01	466.9	0.067	0.987	415.6	0.069	0.999



**Figure 4**  $\eta^*$  as a function of  $\omega$  for (I) emulsions (filled symbols) and (II) gels (open symbols): (●, ○) MS, (■, □) LBG, (◆, ◇) XG, (▲, △) 0.03(LB + XG), and (▼, ▽) 0.1(LB + XG).

tra<sup>20,21</sup> for simple empirical power equations.<sup>1,2,22–24</sup> In our case, these latter equations do not respond to the behavior of  $G''$ , although they provide acceptable fits for  $G'$  ( $\geq 0.98$ ). The values of the fit parameters are shown in Table III. The exponents are similar for all the emulsions ( $0.066 \pm 0.001$ ), except in the case of LBG, which shows increased dependence on  $\omega$  ( $0.09$ ). Comparing them with the gels, we can see that the exponents for MS and the mixtures are similar ( $0.06 \pm 0.01$ ), whereas for XG, the value is slightly greater ( $0.08$ ), and in the case of LBG, it is completely different, with a much greater slope ( $0.23$ ) for  $\log G' = f(\log \omega)$ .

On the other hand, from the values of  $G'$  and  $G''$ , we obtained  $\eta^*$  [ $f(\omega)$ ] for all the gels and emulsions studied. Figure 4(I,II) plots  $\eta^* = f(\omega)$  for the emulsions and gels, respectively.

Of note in all cases is the decrease in  $\eta^*$  with increasing  $\omega$ , this being characteristic of systems

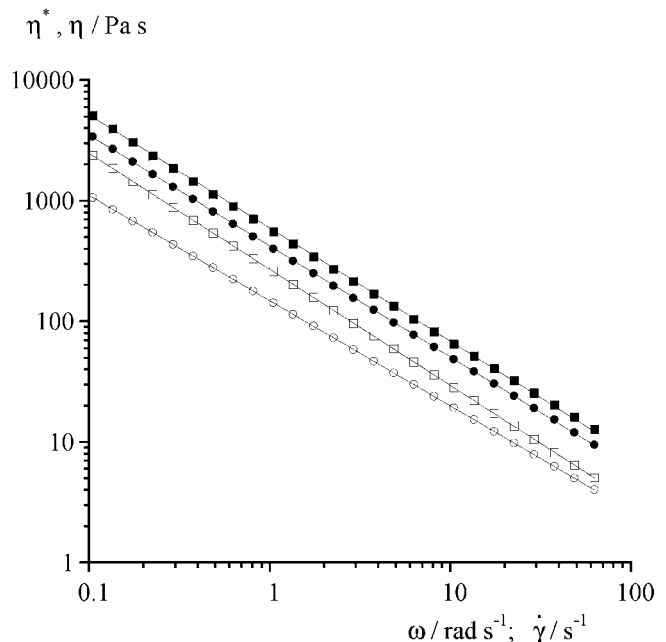
showing shear-thinning behavior. The values of  $\eta^* = f(\omega)$  for all the systems have been perfectly fitted by power functions of the following form (Table III):<sup>25</sup>

$$\eta^* = M\omega^{n-1} \quad (2)$$

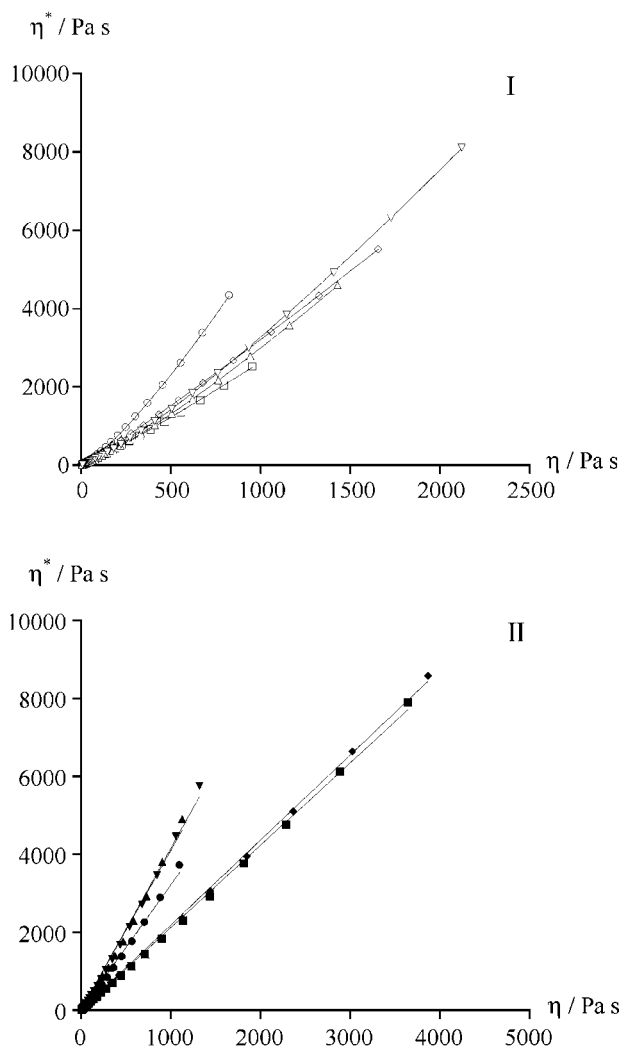
where  $M$  and  $n$  are parameters determined experimentally. All the tracings are parallel, thus indicating that the  $\eta^*$  values of all the systems are similarly sensitive to changes in the oscillation frequency, with a mean value of  $(n - 1) = -0.928 \pm 0.005$ . The sole relevant exception corresponds to the LBG gel, the tracing of which presented a lesser absolute slope:  $(n - 1)_{\text{LBG}} = 0.775 \pm 0.004$ , this indicating a lesser sensitivity to the variation of  $\eta^*$  with  $\omega$ .

Interestingly, the exponent of  $G'$  in the power law,  $n'$ , coincided with the value of  $n$  for  $\eta^*$ . This finding coincides with the observations of other authors<sup>22</sup> and is logical because, when considering that  $G'' \ll G'$ , we can affirm that  $\eta^* \cong G'/\omega$ .

The fact that all the systems except the LBG gel presented the same dependence of  $\eta^*$  on  $\omega$  (similar value for  $n - 1$ ) made it possible for us to study the effects of the different gums on the viscosity of the system, comparing the values of  $M$  (Table III), which were closely correlated to parameter  $K$  of the elastic modulus. Thus, in the case of the gels, an increase was noted in  $M$  and, therefore, in  $\eta^*$  when some of the starch was replaced with XG or XG and LBG. This effect was enhanced in the greatest concentration synergistic mixture (01).



**Figure 5** (■, □)  $\eta^*$  as a function of  $\omega$  and (●, ○)  $\eta$  as a function of  $\dot{\gamma}$  for XG systems: emulsions (filled symbols) and gels (open symbols).



**Figure 6**  $\eta^*$  as a function of  $\eta$  (for  $\omega = \dot{\gamma}$ ): (I) gels (open symbols) and (II) emulsions (filled symbols).

However, this effect was masked when the emulsions were formulated. In fact, an examination of the values of  $M$  obtained for the emulsions has allowed us to deduce that when the percentage of gum in substitution of starch is increased, an increase in the  $\eta^*$  values of the emulsions results (XG and LBG systems). As can be seen in the table, the synergistic effect of the mixtures of LBG and XG at the percen-

tages considered does not increase  $\eta^*$  of the resulting emulsion to the same extent as LBG and XG separately (these intervening in greater proportions than in the synergistic mixtures). Logically, the least effect is recorded for starch replacement by synergistic mixture 003.

Another particularly interesting aspect of studies of this kind is the comparison of the  $\eta^*$  values obtained by oscillatory analysis and the viscosities obtained from the flow curves. To this effect, we obtained the flow curve of each of the samples within the same shear velocity interval corresponding to  $\omega = [0.063, 63]$  rad/s. In all the systems, the complex velocity was greater than the apparent viscosity ( $\eta$ ), as is most often found to be the case.<sup>26</sup>

On the other hand, both  $\eta^*$  and  $\eta$  of the emulsions were greater than those of their corresponding gels. As an example, Figure 5 shows  $\eta^* = f(\omega)$  and  $\eta = f(\dot{\gamma})_{\dot{\gamma}=\omega}$  for the XG gel and emulsion. The nonoverlapping nature of the tracings shows that the Cox–Merz function<sup>27</sup> for homogeneous systems, expressed by

$$\eta^*(\omega) = \eta(\dot{\gamma})_{\dot{\gamma}=\omega} \quad (3)$$

is not applicable to other more complex systems such as those studied in this work. In these cases, we usually resort to the generalized Cox–Merz rule<sup>25</sup> in the following form:

$$\eta^*(\omega) = C \left\{ [\eta(\dot{\gamma})]^\alpha \right\}_{\dot{\gamma}=\omega} \quad (4)$$

where  $C$  and  $\alpha$  are parameters to be determined on an experimental basis for each system.

Figure 6 presents  $\eta^*(\omega) = f(\eta)_{\dot{\gamma}=\omega}$  in the previously indicated angular frequency interval and shear velocity range. The continuous lines are correspond to least-squares fits via eq. (4). The values of parameters  $C$  and  $\alpha$  and the corresponding  $r$  values are reported in Table IV. All the values of  $r$  are greater than 0.997, and this indicates that the fits are satisfactory.

The calculations corresponding to the emulsions were made on the basis of linear fits ( $\alpha = 1$ ) because the obtained equations were simpler and the  $r$  values of the fits were acceptable [see Fig. 6(II)].

The fact that the  $\alpha$  values for the gels were greater than those for the emulsions indicated that in the

**TABLE IV**  
Values of Parameters  $C$  and  $\alpha$  for the Generalized Cox–Merz Rule and  $r$

	Gels			Emulsions		
	$C$	$\alpha$	$r$	$C$	$\alpha$	$r$
MS	$0.96 \pm 0.01$	$1.253 \pm 0.002$	0.999	$3.21 \pm 0.04$	$1.00 \pm 0.06$	0.997
LBG	$1.27 \pm 0.05$	$1.105 \pm 0.006$	0.999	$2.11 \pm 0.01$		0.999
XG	$1.89 \pm 0.02$	$1.076 \pm 0.001$	0.999	$2.18 \pm 0.01$		0.999
003	$0.77 \pm 0.01$	$1.196 \pm 0.002$	0.999	$4.10 \pm 0.05$		0.997
01	$0.79 \pm 0.01$	$1.206 \pm 0.002$	0.999	$4.15 \pm 0.04$		0.998

case of the gels, the obtained systems presented greater bonding density between polymers than in the case of the emulsions. This was because the presence of the rest of the components, added to form the emulsion, partly masked the effects of bonding between polymers. In any case, the fact that, in both the gels and particularly in the emulsions,  $\alpha$  differed very little from unity showed that both systems approached the behavior proposed by Cox and Merz for homogeneous systems.<sup>28,29</sup>

On the other hand, the values of parameter  $C$  of the emulsions were greater than those of the gels corresponding to the formulations with starch alone and (particularly) synergic mixtures 003 and 01 because  $C$  for emulsions  $\approx 4C$  for gels (Table IV). This appears to indicate that some added component favored the interactions between XG and LBG, inducing an increased bond density that in turn led to increased differences in the system behavior versus the behavior proposed by Cox and Merz.

Interestingly, from eq. (4), and taking into account the values of  $C$  and  $\alpha$  provided in Table III, we can obtain the function  $\eta^* = f(\eta)$  for each system; that is, it is easy to determine  $\eta^*$  once  $\eta$  is known when the measurements are made with a common viscometer or  $\eta$  from the oscillatory measurements that allow the determination of  $\eta^*$ .

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