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Response time and electrorheology of semidiluted gellan, xanthan and cellulose suspensions

L. Rejón[a], A. Ramírez[b], F. Paz[c], F.M. Goycoolea[d], M.A. Valdez[b,*]

[a]Instituto de Investigaciones Eléctricas, Ap. Postal 1-475, Cuernavaca, Morelos, 62001 Mexico
[b]Departamento de Física, Universidad de Sonora, Rosales y Transversal, Ap. Postal 1626, 83000 Hermosillo, Sonora, Mexico
[c]Departamento de Geología, Universidad de Sonora, Ap. Postal 1626, 83000 Hermosillo, Sonora, Mexico
[d]Centro de Investigación en Alimentación y Desarrollo, A.C. Ap. Postal 1735, Hermosillo, Sonora, Mexico

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Abstract

Response times with electrical fields of gellan and xanthan dry powder suspensions of 25, 32 and 53 μm average diameter and concentrations of 1.0, 1.5 and 2.0% (w/w) dispersed in commercial corn oil were optically measured through a specifically designed set up. In all cases, the delay time was proportional to $1/E^a$, where E is the applied field and a is an adjustable parameter. The values of parameter a were very different from the typical value of some known electrorheolgical fluids. Response time of gellan suspensions was shorter than the one obtained for xanthan and it is comparable to the time found by using silica particles in silicon oil. Response times for cellulose were very large and the fibrillation phenomenon was negligible for $E < 1.0$ kv/mm.

Viscosity measurements of semidiluted xanthan, gellan and cellulose suspensions (1.0 and 1.5% w/w) under the influence of electrical fields, were performed in a parallel plates rheometer. Results in the range of stress <70 Pa showed that viscosity values of gellan suspensions were larger than those obtained with xanthan or cellulose under the same applied electric field at shear rates higher than 10 s^{-1}. However, cellulose suspensions showed larger viscosity values compared with the ones measured with xanthan and gellan suspensions at very low shear rates. Dielectric measurements of cellulose, xanthan and gellan 1.5% w/w suspensions were performed in the range 10^0–8×10^4 Hz. Results agree with a Maxwell–Wagner type relaxation model. © 2002 Elsevier Science Ltd. All rights reserved.

Keywords: Electrorheology; Polysaccharide; Response time; Electric permittivity

1. Introduction

Electrorheology is a term applied to the phenomenon in which the fluidity of a suspension is modified by the application of electric fields (EF) (Conrad, 1998). This phenomenon is explained by the formation of a fibrillated microstructure in a dense suspension due to dipole–dipole interactions under the influence of electrical fields. These interparticle forces result in a fluid with enhanced viscosity that exhibits large yield stress. This behavior along with the rapid time scales of the structure formation makes these fluids ideal working systems in electromechanical applications.

The shear stress of an electrorheological (ER) suspension under an EF approximates the rheology of a Bingham material, given by the equation

$$\tau \cong \tau_E + \eta_s \dot{\gamma} \tag{1}$$

Where τ_E is the electrical contribution to the total stress τ, η_s is the viscosity of the suspension at zero field and $\dot{\gamma}$ the shear rate.

A characteristic feature of an ER suspension under static conditions is the alignment of the particles into chains in the direction of the applied field. The stress τ_E represents that for the continued rupture and reformation of the chains or clusters.

The extrapolated value of τ at zero shear rate, known as yield stress τ_y, is used as a measurement of the ER response. Another parameter to characterize the ER suspension is the average viscosity with the field

$$\eta_E = \frac{\tau(E)}{\dot{\gamma}} \tag{2}$$

Yet another important parameter to characterize an ER suspension is the response time, whose definitions given in the literature differ in their criteria. One is the time interval between the application of the field and the formation of the first bridging fibril (Hill and Van Steenkiste, 1991). However, other authors using light scattering to monitor

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* Corresponding author. Fax: +52-62-592109.
E-mail address: valdez@eudeve.fisica.uson.mx (M.A. Valdez).

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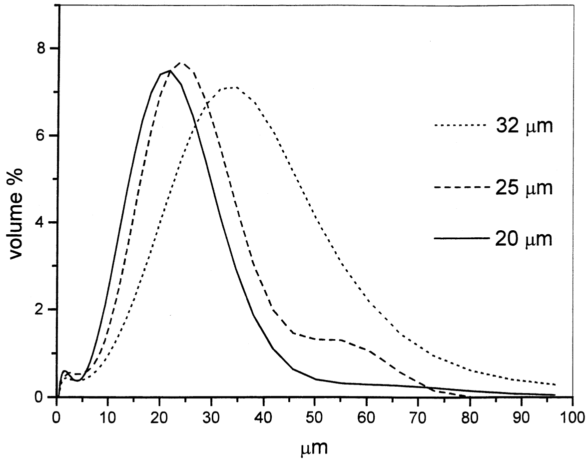

Fig. 1. Particle size distribution plot of xanthan powders using three different sieves.

induced fibrillation, consider it as 50% of the total time to produce saturation of the fibrillation phenomenon (Smith and Fuller, 1989), while other researchers have used the total time to produce saturation due to the EF (Zamudio, Nava, Rejón, Ponce, Viquez and Castaño, 1996).

One of the many problems related with ER suspensions is the sedimentation problem and the use of different kinds of non biodegradable oils, such as silicon and mineral oils, among others. In case of a future extended use of these materials in different applications, special care should be taken in the use of ER suspensions. Natural polymers, such as cellulose, have been used in preparation of ER fluids and recently some authors have used chitosan sulfate suspensions in silicon oil. The biodegradable feature of polysaccharides and biodegradable oils might be an advantage for their applications in terms of environmental protection (Wu and Shen, 1999).

In this work we investigate some ER properties of commercial polysaccharides suspended in a comestible corn oil.

Three bacterial polysaccharides were used in this investigation, namely xanthan, gellan and microfibrillar cellulose. Xanthan has a linear backbone comprising of (1 → 4) linked β-D glucose residues, bearing a trisaccharide side chain attached on alternate units. The side chains contain two negatively charged substituent groups. The chemical structure of gellan is described as a neutral linear structure with a tetrasaccharide repeating unit, with one charged site per repeating unit. Microfibrillar cellulose is a linear structure of β (1 → 4)-linked groups. The three polymers have a very high intrinsic chain stiffness and exhibit crystalline order in the solid state (Robyt, 1998).

We have investigated the behavior of semidiluted suspensions of dry gellan, xanthan and cellulose of varying particle size and weight fraction by measuring the variations of transparency of the suspension when a laser beam is passed through the system due to the effect of the applied field on the fibrillation phenomenon. Viscosity measurements were performed under the influence of constant EF by using a parallel plates rheometer; complementary dielectric measurements of the polysaccharide suspensions were performed as a tool to understand the ER behavior.

In Section 2 we indicate the particles' preparation method and the equipment used, in Section 3 we discuss some results of response time, permittivity and ER measurements; finally we give some conclusions.

2. Experimental

Different particle sizes of the polysaccharides dry commercial xanthan (Aldrich), gellan (Kelco) and cellulose (Primacel, Kelco) were obtained after the materials were milled with an agate mortar and pestle and later passed through metal sieves of 25, 32 and 53 μm. The neutrality of the powder suspensions was observed by videomicroscopy when a constant EF was applied. The fibrillation phenomenon was observed to be homogeneous and the powder suspensions in corn oil did not show particles migrating to any electrode, this could guarantee that the grinding process did not contribute to put charges on the powders. The particle size distribution was measured with a light dispersion method (Coulter, LS Light Size Analyzer). In Fig. 1 we show the volume distribution of the particles for some xanthan samples. The mean size calculated from the volume distribution was 30.5 μm for the 32 μm sieve, 25.07 μm for the 25 μm sieve and 18.95 μm for the 20 μm sieve.

Commercial corn oil, 0.872 g/cm^3 density and 0.10 Pa s viscosity, was used to disperse the polysaccharide powders. The water content of these samples was 8% for xanthan, 4% for gellan and 3% for the cellulose samples.

Polysaccharide powder suspension were mixed with a Cowles type disperser at 2000 rpm for 5 min. All samples were placed into a vacuum chamber to extract air bubbles and the conditions of the experiments were around room temperature: 25.0 ± 0.5°C for response time measurements and 25 ± 1°C for ER measurements.

Response times were measured for semidiluted suspensions of 1.0, 1.5 and 2.0% w/w. None of them showed any significant sedimentation during the experiments.

Response times of the polysaccharide suspensions were determined with a method similar to the one reported by Zamudio et al. (1996). A Ne 10 mW laser light is directed through the suspension placed in a transparent capacitor-like cell with two thin copper foil electrodes (4.0×1.0 cm^2), the electrodes are parallel to the laser beam with a gap separation of 4 mm. The transmitted light is detected with an optical detector, then the image was analyzed with software developed in our laboratory. The transmitted light results in proportion to the magnitude of the applied EF and to the time it acts on the fibrillation process. The response time is equated to the total time the electric field takes to get a stable fibrillar structure in the

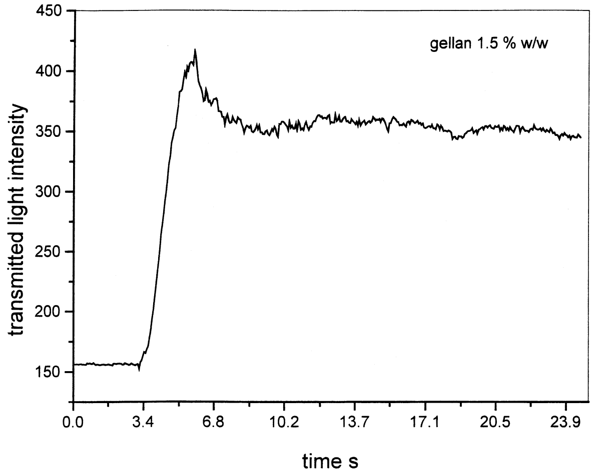

Fig. 2. Transmitted light intensity through a gellan sample when an EF is applied at about 3.4 seconds after starting the program.

system. As is shown in Fig. 2, after this time the transmitted light intensity becomes approximately steady.

Rheological measurements were made in a strain controlled ARES (2KFRTN1) rheometer using a steady rate sweep test. We used parallel plate geometry with two 25 mm diameter plates and a gap separation of 0.5 mm. DC voltages were supplied by a Treck High Voltage Amplifier model 609E-6. The method used included steady-state equilibrium conditions within a shear rate sweep procedure and the shear rate was logarithmically increased. Shear rate was varied from 0.05 to 100 s^{-1} and each shear stress and viscosity value was obtained and averaged by the instrument during a measurement time of 10 s. The delay before measure was 5 s and 10 points per decade were taken in each run.

Dielectric measurements of gellan, xanthan and cellulose 1.5% w/w suspensions were performed with a dielectric analyzer (Dupont Instruments, model DEA 2970) in the range $1.0^{-8} \times 10^4$ Hz.

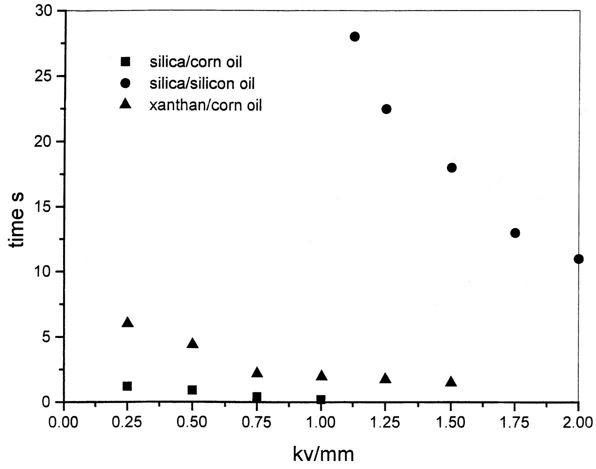

Fig. 3. Response time comparisons between silica particles and xanthan in silicon oil and corn oil, using 1.0% w/w concentrations.

3. Results

Response times of suspensions with three different concentrations (1.0, 1.5 and 2.0% w/w) and three different particle sizes (25, 32 and 53 μm) of gellan and xanthan in corn oil suspensions were determined applying a constant EF in the cell described above.

As expected, the response time of the polysaccharide materials tested is larger compared to those of concentrated typical ER suspensions. Response times obtained by Zamudio et al. (1996) with different fluids and silica particles, are shorter than 50 ms by using 10–20% w/w particle concentrations. These concentrations are about 10–20 fold higher than the one used in this work. Response times for the same silica sample and silicon oil used in that work were measured by us for 1.5% w/w suspensions. The results showed that shorter response times were obtained for silica/corn oil than for silica/silicon oil, probably due to differences in the solvent viscosity. Fig. 3 shows these comparisons along with the behavior obtained with xanthan/corn oil used in this work.

According to the available model, the response of ER fluids depends on the EF as

$$t \approx 1/E^a, \tag{3}$$

where a is a parameter approximate to 2.

This behavior was found by Klingenberg, Zukoski and Hill (1993), performing dynamic simulations and employing an electrostatic polarization model for ER suspensions considering the point dipole limit. They observed that any measurement of time characterizing the kinetics of structure formation will scale as $1/E^2$. However, as reported by Zamudio et al. (1996), this prediction does not agree with their experimental results. Using silica and silicon oil, among other fluids, they found an a parameter varying between 1.69 and 1.99.

Klingenberg, Van Swol and Zukoski (1989), defined a percolation time as the time required to form the first percolating cluster. They found for hydrated glass spheres (47 μ) in silicone transformer oil that the percolation time reduces as Φ (the weight fraction) grows, and for a $\Phi = 0.05$, equivalent to the concentration used by us, they found a percolation time of about 1 s. Experiments performed on hollow silica spheres in corn oil, display a response time dependence $\tau \approx 1/E^{2.9}$. Our results obtained with xanthan and gellan show that this parameter varied with the concentration and the size of the particles. Results of the a parameter are shown in Table 1 for xanthan and gellan suspensions.

It is noticeable that none of the values is close to 2.0. A significant difference between xanthan and gellan was observed. For xanthan suspensions, the largest a value is obtained with the 53 μm particle size; however, gellan suspensions do not show this regularity. A value of 1.37, close to our results, was obtained for the a parameter by

Table 1
Parameter *a* of the relationship between response time and electrical field, given by $t \approx 1/E^a$

% w/w Xanthan	25 μm	32 μm	53 μm
1.0	0.61	0.54	1.01
1.5	0.30	0.45	0.79
2.0	0.71	0.68	1.05
% w/w Gellan	**25 μm**	**32 μm**	**53 μm**
1.0	0.85	0.60	0.73
1.5	0.41	1.05	0.65
2.0	1.01	0.77	0.58

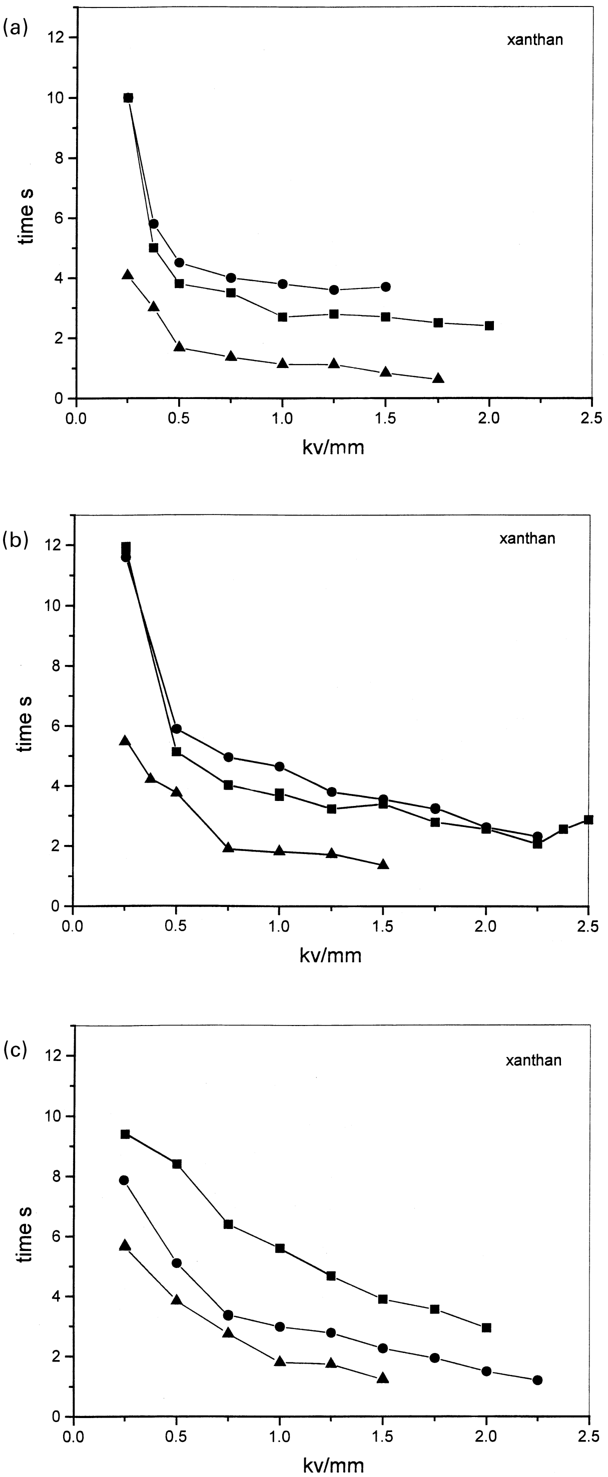

Fig. 4. Response time for xanthan suspensions for different EF and for the three different particle sizes used: 25 (■), 32 (●) and 53 (▲) μm (a) 1.0% w/w, (b) 1.5% w/w, (c) 2.0% w/w.

Wen, Zheng and Tu (1998), for volume fractions within 0.05–0.06.

Fig. 4a, b and c, show response times for different EF of nine different xanthan suspensions. It can be observed that this time is very dependent on the size of the particles and on the concentration. In all the cases, the response time is shorter for the suspensions with larger particle sizes for the same applied EF and concentration. On the other hand, inspection of these figures, show that for both the 25 and 53 μm particle size suspensions, the shortest time was obtained for the most diluted suspension (1.0% w/w).

Response times for gellan suspensions showed the opposite behavior with respect to concentration. Fig. 5a, b and c show response times for the three different particle sizes used. We observe that the response time is shorter for higher concentrations. This is more evident for the 53 and 25 μm particle sizes and for larger EF. This is in agreement with the behavior of most of the ER fluids and it is related to the interparticle average distance in the suspension; when the particle size or the concentration is increased, this distance decreases and the response time tends to be shorter. From the plots we deduce that only for higher concentration (2.0% w/w) and for the EF larger than 1 kv/mm, the response time start being independent of the particle size. A detailed comparison of the response time between xanthan and gellan suspensions, for different concentrations and particle size shows that in all cases, a shorter response time is obtained for gellan, with the same EF, particle size and concentration.

For the case of cellulose suspensions, a very negligible effect of the EF on the fibrillation phenomenon was observed for EF lower than 1 kv/mm. Significant fibrillation effects started at about 1.0 kv/mm, but the response time was much longer than any one obtained with xanthan and gellan. Another characteristic of the cellulose suspensions was the effect of the EF on the transmitted light, generating curves with several peaks where no unique response time could be obtained and consequently, we could not use the model given in Eq. (3) to obtain the corresponding *a* parameter. It seems as if the fibrillation process consisted of more than one step. In many cases, the fibrillation process was so slow, that we obtained stabilized curves with a non-monotonic behavior in a time larger than one minute.

Percolation time for these polysaccharides were measured for some samples using a videocamera adapted to a microscope. Using an EF of 0.2 kv/mm, 53 μ average

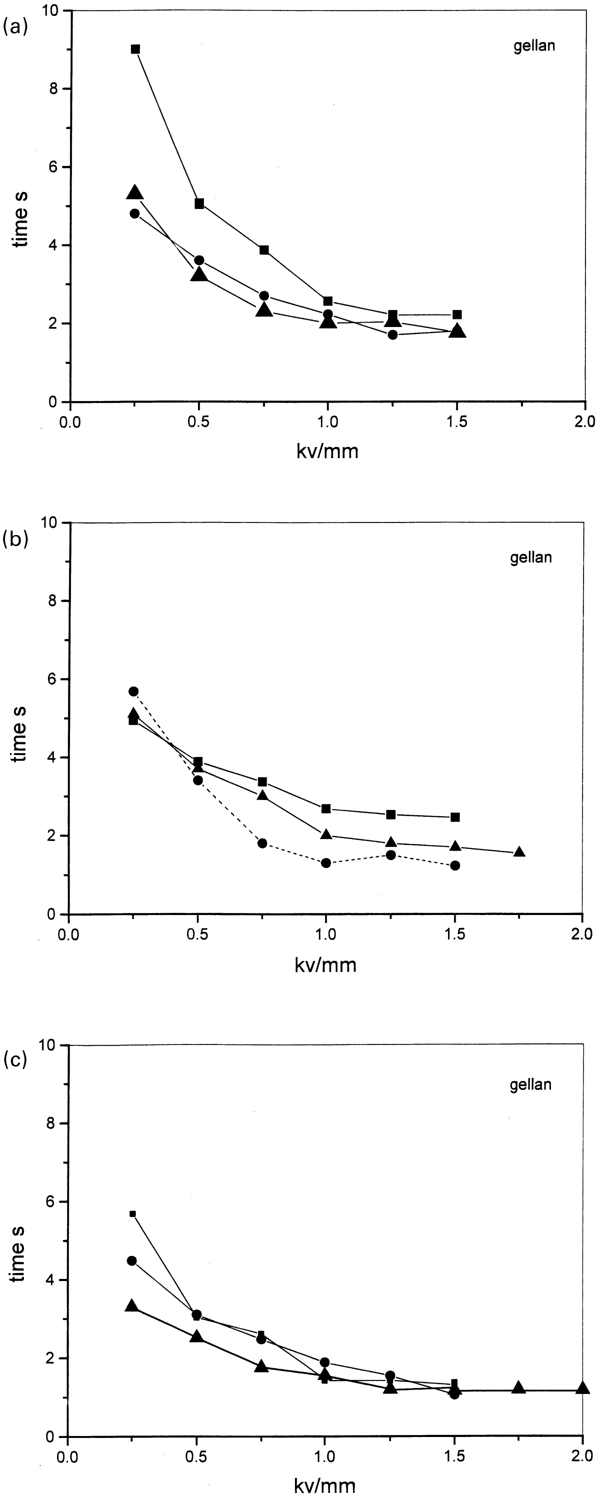

Fig. 5. Response time for gellan suspensions for different EF and for the three different particle sizes used: 25 (■), 32 (●) and 53 (▲) μm). (a) 1.0% w/w, (b) 1.5% w/w, (c) 2.0% w/w.

particle diameter and 1.0% w/w suspensions, we obtained average 180 ms for xanthan, 100 ms for gellan and 1.70 s for cellulose. This result qualitatively agrees with the response times obtained with the method described above. Measurements of the percolation time were performed using silica particles in corn oil at 1.0% w/w concentration, showing an average time of 300 ms for the same electric strength.

Now we discuss the dielectric properties of the polysaccharide suspensions obtained for samples of 1.5 w/w concentration.

It is well known that the electrical properties of the host oil, the particles, and consequently of the suspension, are very important for the dielectric behavior. The four types of polarization that exist in order of increased relaxation times are: electronic, atomic, dipole (Debye) and interfacial (Maxwell–Wagner) polarization. From the known dielectric properties of many materials, we know that relaxation times (τ) normally observed for ER suspensions ($\geq 10^{-3}$ s) suggests that the major source of polarization in most of ER suspensions is the Maxwell–Wagner interfacial polarization (van Beek, 1967). The model proposed by Wagner and later developed by von Hipel (Barnikas and Eichorn, 1983) describes the behavior of the complex dielectric permittivity as a function of the EF frequency $w(w = 2\pi f)$, $\epsilon(w) = \epsilon'(w) + i\epsilon''(w)$, where ϵ' is termed the permittivity and ϵ'' is termed the dielectric loss factor. The permittivity and the loss factor are given by the equations

$$\epsilon'(w) = \epsilon'_\infty + (\epsilon'_s - \epsilon'_\infty)/(1 + w^2\tau^2), \tag{4}$$

$$\epsilon'' = \epsilon'_\infty \tau/(w\tau_1\tau_2) + w\tau(\epsilon'_s - \epsilon'_\infty)/(1 + w^2\tau^2), \tag{5}$$

where ϵ'_∞ and ϵ'_s are the permittivity at very high and very low frequencies, respectively. τ is the relaxation time of the suspension, given as $\tau = 1/w_{\max}$. Here $w_{\max}$ is the frequency value where the loss factor ϵ'' reaches a relative maximum. In Eq. (5), τ_1 and τ_2 are relaxation time parameters given in the model developed by von Hipel.

The results of permittivity and loss factor versus frequency of gellan, xanthan and cellulose suspensions are shown in Fig. 6a and b. From these curves we obtain the loss tangent defined as $\tan\delta = \epsilon''/\epsilon'$. The maximum of the $\tan\delta$ for the suspensions analyzed are: 0.21 for gellan, 0.19 for xanthan and 0.14 for cellulose. The τ for cellulose was 5×10^{-3} s and for gellan and for xanthan, we obtained approximately the same value 5×10^{-4} s, both corresponding to typical ER suspensions (Conrad, 1998).

Using Eqs. (4) and (5), we find good agreement for the three polysaccharide suspensions in the range 10^0–8×10^4 Hz as can be observed in Fig. 6a and b. The shapes of the ϵ'' vs frequency curves are common for a system with a dominant Maxwell–Wagner polarization process. The behavior of ϵ'' obtained at low frequencies corresponds to conductive effects and its contribution is given by the first term in Eq. (5) (Parthasarathy and Klingenberg, 1996). For higher frequency values, the Debye type behavior is found for the three suspensions, but the location of the peaks ($w \lesssim 10^5$ Hz) reinforces the idea of a interfacial polarization process for the three suspensions. It is noticeable that around the peak of each experimental ϵ'' plot, the adjustment of the

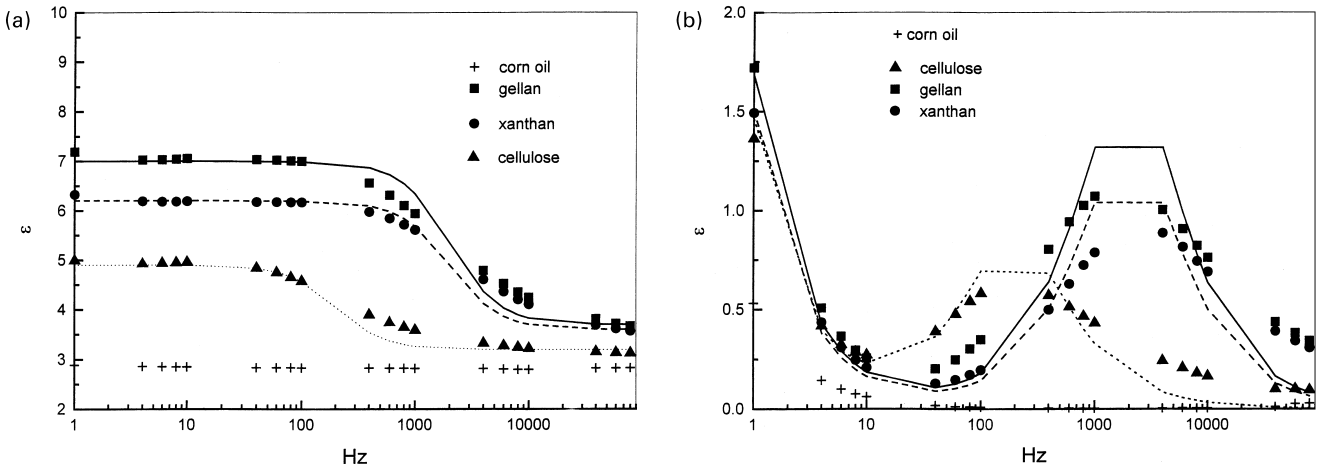

Fig. 6. Dielectric behavior of 1.5% w/w polysaccharide suspensions in the range 10^0–8×10^4Hz. (a) ϵ' vs. frequency. Solid line, dash line and dot line correspond to plots given by Eqs. (4) and (5) for gellan, xanthan and cellulose, respectively.

experimental points with Eq. (5) is similar in the three cases and it is not a very good fit. This has been explained by some authors as being due to the presence of a multiple set of relaxation times for some ER suspensions (David and Misra, 1999). The best fitted values taken for the product of $\tau_1\tau_2$ in Eq. (5) were 0.011 for cellulose suspensions and 0.0011 for gellan and xanthan suspensions.

According to the generalized idea (Conrad, 1998) that the strength of ER fluids increases with the difference $\epsilon'_s - \epsilon'_\infty$ for the suspensions, which is a measure of the degree of polarization, we obtain from Fig. 6a, 1.6 for cellulose, 2.6 for xanthan and 3.3 for gellan. This would mean that gellan suspensions should have a better ER behavior than the others. As we will see in the next section of the rheological results, this is partially true. On the other hand, the values of tan δ for the suspensions analyzed are close to the theoretical value predicted by a model based on the Wagner model for an ER fluid (0.10) (Hao, Xu and Xu, 1997).

The three polysaccharide suspensions studied showed a shear thinning behavior in the range $0.1–100\ \mathrm{s}^{-1}$ under applied EF up to 2 kv/mm. In Fig. 7a, b and c we show the effect of the shear rate on the viscosity for different applied EF. For relative low shear rates, cellulose suspensions show the highest viscosity, close to three orders of magnitude with respect to the viscosity at zero EF. However, at higher shear rates ($\gtrsim 10\ \mathrm{s}^{-1}$) gellan suspensions show greater viscosity values than cellulose and xanthan for the same EF applied. It is interesting to notice that in spite of the fact that cellulose maintains a higher viscosity at lower shear rates, it decreases faster with the shear rate. On the contrary, for xanthan and gellan suspensions, their viscosity values decrease more slowly with the shear rate, showing a plateau-like curve in the middle of the shear rate range for EF $\gtrsim$ 1 kv/mm. This means that the fibrillar structure, due to the particle polarization, is maintained for higher shear rates than for the case of cellulose suspensions. For shear rates higher than $10\ \mathrm{s}^{-1}$, the effect of the EF on the viscosity is different for each suspension. It seems that the largest permittivity value of gellan suspensions is responsible for maintaining larger viscosity values at higher shear rates. On the contrary, for the case of cellulose suspensions the viscous forces dominate the electric ones responsible for the particle polarization achieving lower viscosity and saturation for EF $\geq$ 1.5 kv/mm. However, this explanation is not complete, as is well known (van Beek, 1967) that the behavior of the polarization process at constant EF and low frequencies is not dominated by primitivities, but by conductivities of the particles and the solvent.

Fig. 8a, b and c show the behavior of stress for the three polysaccharide suspensions studied and different EF applied. The three suspensions show Newtonian behavior in the whole range. Cellulose suspensions show typical Binham flow curves, only for 2 kv/mm the flow curve shows a plateau at a relatively low shear rate. This behavior has also been observed by Rejón (1998). At higher shear rates; the curves are almost linear and saturation is observed for EF $\geq$ 1.5 kv/mm. Fig. 8b and c show similar behavior, both suspensions show an initial linear region at low shear rates, then a plateau is present for the four applied EF values. The height of the plateau is larger as the EF increases, and the corresponding stress value is taken as the yield stress. This yield stress is a measure of the stress needed to apply in order that the fibrillar structure begins to be broken in smaller chains. It is clear from Fig. 8 that the yield stress values are larger for gellan in all cases. At higher shear rates, stress grows very slowly for gellan suspensions and saturation is not observed in the range of EF measured. For the flow curves of xanthan suspensions we observe a decreasing slope around $80\ \mathrm{s}^{-1}$ and for 2 kv/mm, in agreement to the abrupt decreasing viscosity observed in Fig. 7b.

Yield stress measurements were directly obtained from the flow curves for each constant EF as the stress value at the

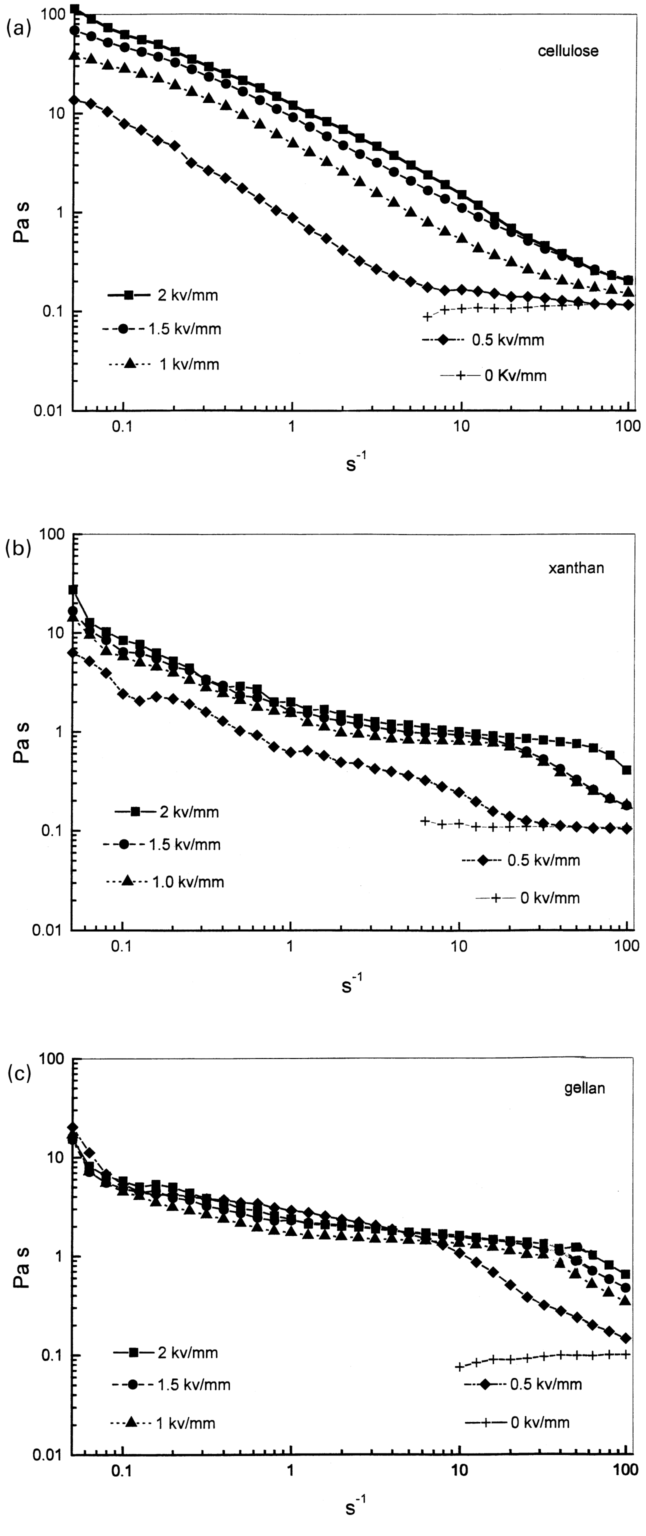

Fig. 7. Viscosity measurements for different EF and the three 1.5% w/w suspensions: (a) cellulose, (b) xanthan, (c) gellan.

plateau, extrapolated to zero shear rate. Different authors have experimentally and theoretically obtained that the yield stress scales with E^2 for low EF (Kim and Klingenberg, 1996). Our results for the yield stress and for the three polysaccharide suspensions, with the two concentrations used, are shown in Fig. 9 for different EF. For the 1.0% w/w suspensions, the yield stress scales as $\tau_y \sim E^m$, were $m = 2.9$ for cellulose, 2.1 for xanthan and 1.5 for gellan. For the 1.5% w/w suspensions, the yield stress scales as $\tau_y \sim E^2$ for cellulose and xanthan and $\tau_y \sim E^{1.2}$ for gellan. From these results, we observe that only for gellan suspensions the result is close to the behavior found by Davis (1997). Using a conductivity model and cellulose suspensions, but with a higher volume fraction ($\Phi = 0.2$), he found the dependence $\tau_y \sim E^{1.5}$. The results for xanthan and cellulose are closer to the predicted $\tau_y \sim E^2$ than to the one found with the conductivity model.

4. Conclusions

In this work we have compared the response time and viscosity measurements, applying different electrical fields, for three different semidiluted polysaccharides suspensions. As expected, response time for these suspensions is not as short as for a more concentrated ER suspension, but response time comparisons with silica particles suggest that gellan and xanthan might show faster response times at higher concentrations. Our measurements determined significant differences for the polysaccharides studied and from these results, it is possible to infer that besides the influence of different water content and permittivity of the samples, charges associated to the two negative charged substituent groups in the xanthan molecule could induce dipoles with the applied EF not oriented to the principal chain; this could explain that the gellan molecule, with one charged site per repeating unit, produced a shorter response time. On the contrary, neutral cellulose molecules induce very low polarization and consequently long response times with the EF. The observed phenomenon for this material was probably due to the water content. Our measurements of the percolation time again suggests the possibility that especially the polysaccharide gellan can be used for future ER characterizations and can compete with other non-biodegradable materials.

It is important to mention that our results for the *a* parameter in Table 1, for the behavior of the response time and the EF, were very different from the value 2 expected for a coulombic interaction between the suspended particles, this opens the question about the nature of the particle interaction between these kind of materials.

The three polysaccharide suspensions showed viscosity values at least two orders of magnitude larger than the viscosity at zero EF and low shear rates. This viscosity increment is comparable to the one obtained for other ER fluids. The dielectric characterizations of these materials indicate too, a good ER behavior for more concentrated suspensions.

In the future, we intend to characterize the ER behavior of these polysaccharides increasing the concentration of the suspensions.

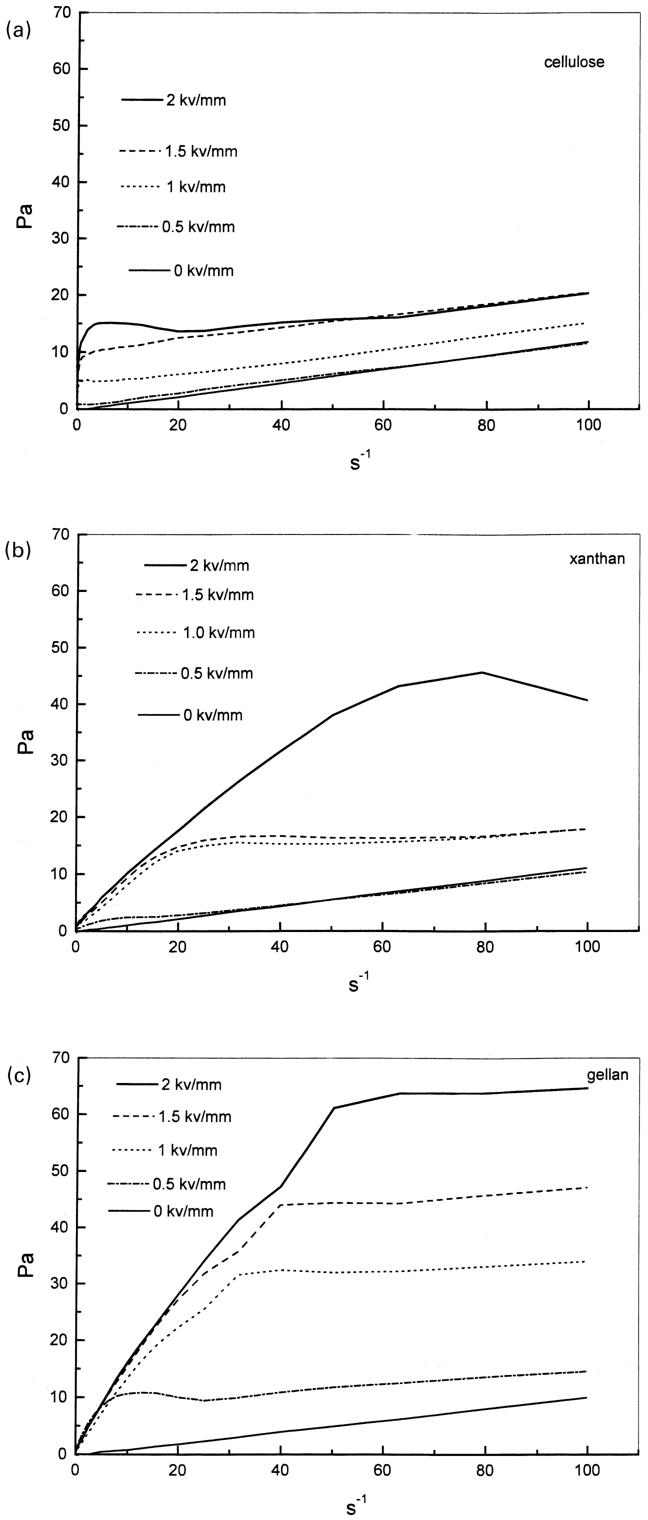

Fig. 8. Flow curves for different EF and the three 1.5% w/w polysaccharide suspensions: (a) cellulose, (b) xanthan, (c) gellan.

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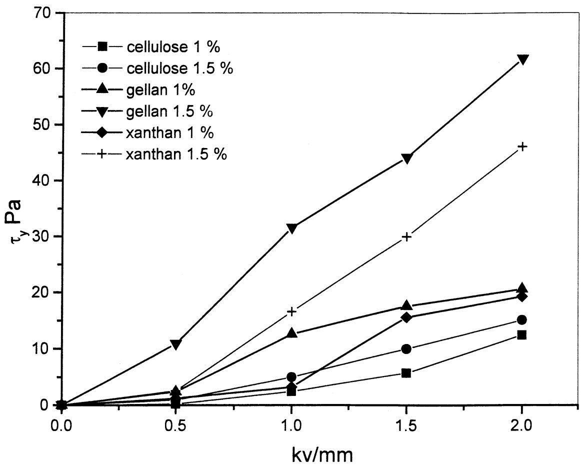

Fig. 9. Yield Stress of 1.0 and 1.5% w/w suspensions for different EF.

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