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Electrorheological Properties of poly(Li-2-hydroxyethyl methacrylate)-*co*-poly(4-vinyl pyridine): The Effect of Dispersed Particle Size

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Abstract

The electrorheological (ER) effect is commonly known as a reversible increase in the viscosity of a suspension of solid particles dispersed in an insulating liquid after application of an external electric field. In this study, ER properties of poly(Lithium-2-hydroxyethyl methacrylate)-co-poly(4-vinyl pyridine), poly(Li-HEMA)-co-poly(4-VP), ionomeric salt (ionomer) was investigated. The ionomer particle sizes were characterized by dynamic light scattering, (DLS) method. Suspensions of ionomers were prepared in insulating silicone oil, at a series of concentrations (c = 5-30 %, m/m). Effects of particle size, temperature and concentration on the sedimentation stabilities of these suspensions were determined. Flow times of the suspensions were measured under no electric field (E = 0 kV/mm), and under an applied electric field (E ≠ 0 kV/mm) strength and a strong ER activity was observed for all the suspensions studied. Further, the effects of particle size, concentration, shear rate, electric field strength, frequency, promoter and temperature onto ER activities of ionomer suspensions were investigated.

Keywords: Electrorheological effect, poly(2-hydroxyethyl methacrylate)-co-poly(4-vinyl pyridine), particle size, Shear stress.

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1. Introduction

Electrorheological (ER) fluids are suspensions composed of semi-conducting solid particles and weakly conducting liquids, which show abrupt changes of rheological properties under the stimulation of an external electric field. It is generally accepted that the cause of this behavior is the dielectric polarization of suspended particles and a consequent formation of particle chains oriented in the direction of applied electric field. Due to a higher resistance of the chain structure against deformation, the viscosity increases especially at low shear rates. In some cases, polarization forces may be so high that even a static yield stress of the fluid appears. At high shear rates the particle chain arrangement is destroyed and the suspension flows as in the absence of electric field [1].

Electrorheological fluids (ERF) have attracted enormous attention because of their potential usage in devices such as valves, dampers, clutches, brakes, or robotic actuators [2]. The mechanism of the ER effect has been intensively discussed in many review articles [3,4]. A generally accepted concept for the positive ER effect is that the particles form fibrillated chains, which cause an abrupt increase in the ER parameters.

The dramatic change in the ER properties of ER fluids coupled with the fast response, offers possibilities to use this effect in a variety of technical applications such as stop valves, clutches, torque transducers, dampers, etc. However, there are still difficulties in practical engineering due to insufficient performance of ER fluids reported in the literature so far.

Since the ER effect is induced by an external electric field, the polarization is believed to play an important role; and the particle dielectric property should be dominant in the ER effect. It is well known that the crystal structure, particle size and composition of the particles, which will influence the physical and chemical natures of a material, are essential to the dielectric and polarization properties of the material. Therefore, it is possible to modify a material's dielectric and polarization properties by adjusting the synthetic procedure such that an ER material having a desired crystal structure, particle size, and composition is produced. The crystal structures of some ER materials have been reported in the literature [5].

Since the discovery of the ER effect by Winslow [6], it has been the subject of many studies and of several reviews [7,8]. A number of various materials, both inorganic and organic, have been proposed as an active particulate phase. The results obtained so far indicate that the efficiency of the ER materials at a given field strength, i.e. the relative increase in the suspension viscosity on the electric field application, depends primarily on the magnitude and the rate of polarization of suspended particles, which is controlled by their dielectric properties and conductivity. It appeared that, in suspensions of many inorganic particles, an activator, such as water [9] or other polar compound adsorbed on the particle surface, is needed to improve the ER efficiency. In our recent studies we have investigated the synhesis and characterization of poly(Li-2-hydroxyethyl methacrylate)-*co*-poly(4-vinyl pyridine) copolymeric salt [10,], and also electrorheological properties of poly(methylmethacrylate)-*block*-polystyrene [11] and poly(Li-2-hydroxyethyl methacrylate) [12] suspensions.

In this study, the effect of dispersed particle size on the ER performance of poly(Li-HEMA)-co-poly(4-VP) ionomeric salt was investigated.

2. Experimental

2.1. Materials

Poly(Li-HEMA)-co-poly(4-VP) was synthesized by free radical polymerization technique. Details of synthesis, characterization and ionomer preparation were given in our previous publication [10]

The average diameters of particles of ionomers were determined by Fraunhofer scattering using a Malvern Mastersizer E, version 1.2b particle size analyzer. The samples were dispersed in distilled water and stirred for 1 min at a constant temperature of 25ºC. The data collected were evaluated according to Fraunhofer diffraction theory by the Malvern software computer [13]. From these measurements $d_{0.5}$ = 8, 13, 19 and 23 μm average particle sizes were observed for the ionomers.

2.2. Preparation of suspensions

The base fluid was silicone oil (SO) provided with a specific gravity of $\rho = 0.97$ g/cm^3, a dielectric constant of $\varepsilon_1 = 2.61$ and a viscosity of $\eta = 150$ mPas at 25^{o}C. Prior to mixing with SO, all the ionomer particles were dried at 150 ^{o}C for 24 h and SO at 110 ^{o}C for overnight to remove any moisture present in a vacuum oven (P = 15 mmHg). Ionomer/SO suspensions were than prepared at a series of concentrations of c = 5-30 (%, m/m).

2.3. Determination of colloidal stability of ionomers

The gravitational stability of suspensions (c = 5-30 %, m/m) against sedimentation was determined at 25°C. Glass tubes containing each concentration of the suspensions were immersed into a constant temperature water bath and formation of first precipitates at the bottom of the tubes was taken to be the indication colloidal instability.

2.4. Rheometry

Rheological experiments were carried out on the suspensions, for the experimental determination of flow behavior and visco–elastic material properties, which influence processing technology and polymers stability and consistency. Flow rate measurements were carried out between two parallel plate brass electrodes. The gap between the electrodes was 0.5 cm, the width of the electrodes was 1.0 cm and the height of liquid on the electrodes was 5.0 cm. During the measurements these electrodes were connected to a high voltage dc electric source (0-12.5 kV with 0.5 kV increments, Fug Electronic HCL-14, Germany) and a digital voltmeter.

At the beginning of the experiment, the electrodes were dipped into a vessel containing the ER fluid and after a few seconds the vessel was removed and the flow time for complete drainage measured using a digital stop-watch ($E = 0.0$ kV/mm). At the second stage, the electrodes were again dipped into the same vessel containing the ER fluid and a high voltage applied. After several seconds the vessel was removed and the flow time for complete drainage measured under the applied electric field ($E \neq 0.0$ kV/mm). This procedure was repeated for each ER fluid sample under various external electric field strengths ($E = 0.5–2.0$ kV/mm with 0.5 kV/mm increments) at ambient temperature for all the ER samples.

Electrorheological properties of the ionomer suspensions were tested by Thermo-Haake RS600 rheo–stress electro-rheometer in controlled stress (CS) mode at a shear rate of 0.001-1500 s^{-1}, using a 35 mm plate to plate geometry electrode with a 1.0 mm gap. All the experiments were carried out at various temperatures ($T = 25$-125°C). The voltage used in these experiments was supplied by an external dc electric field generator (0-12.5 kV, with 0.5 kV increments) (Fug electronics HCL-14, Germany), which enabled resistivitiy to be created during the experiments.

3. Results and Discussions

3.1. Colloidal stability of ionomers

When the density of particles is not as same as that of medium, the particles with micron order size will settle down according to the Stoke's law [14]. In order to solve the traditional problem of particle sedimentation, several researchers have developed different solutions [15]. Density mismatch between dispersed and continuous phase plays an important role in sedimentation stability of an ER fluid. Before ER measurements to be carried out, colloidal stabilities and sedimentation ratio of all the particle sizes studied in this work were determined in SO at 25°C, and results obtained are tabulated in Table 1 and shown Fig. 1. As seen from the table and figure, colloidal stabilities of suspensions were decreased with increasing particle particle size and concentration. Maximum colloidal stability was found to be 19 days for c = 5 %, d_{50} = 7 μm ionomer/SO suspension. These expected results are in accordance with the earlier study reported by Zhao and Duan [16].

3.2. Electrorheology

Results obtained from parallel plate electrodes

Flow times of ionomer suspensions, prepared in SO at the range of c = 5─30 (%, m/m) concentrations, measured between the parallel plate electrodes at E = 0 kV/mm and under E = 0─1.8 kV/mm are shown in Fig. 2. As seen from the figure, first gradual increase in flow time was started at E = 0.7 kV/mm and continued until E = 1.6 kV/mm. After E_t = 1.6 kV/mm threshold energy was supplied to the suspensions, very sharp increases at the flow times was observed for all the particle sizes. This increase was highest for $d_{0.5}$ = 7 μm ionomer/SO suspensions (for E = 1.6 kV/mm, t = 105 s).

The flow times illustrated in Fig. 2 are the maximum flow times, which could be measured under E = 1.8 kV/mm. When E was further increased, flow of the liquid between the electrodes was completely stopped and the measurement could not be made even after several hours waiting.

The maximum flow times of ionomer suspensions were increased with increasing E and decreasing ionomer particle size and varied in the following order: 7 μm (105 s)>13 μm (97 s)>19 μm (87 s)>23 μm (75 s). Similar behavior was reported for poly(Li-2-acrylamido-2-methyl propane sulfonic acid) and poly(styryllithium)-block-polyisoprene ionomeric salt suspensions in silicone oil [17,18].

3.3. Results obtained from electro- rheometer

Effect of particle concentration

The effect of concentration of the ionomer particles on the viscosity and shear stress is given in Fig. 3 and Fig. 4, respectively. The electric field viscosity (η_E) increases with increasing particle concentration for all the particle sizes (Fig. 3). Highest electric field viscosity was observed for the smallest ($d_{0.5}$ =7 μm) ionomer as η_E = 281 Pas. This trend is due to the enhanced magnitude of polarization forces, acting between the ionic particles, with increasing particle concentration and E. The magnitude of this polarisation force (F) in the direction of applied electric field (E) is

$$F = \frac{6\varepsilon_2 r^6 E^2}{\rho^4} \tag{1}$$

Where ε_2 is the dielectric constant of the particle; ρ is the distance between particles, r is the radius of particle [19].

The effect of ionomers concentration on the shear stress is given in Fig. 4. As reflected from the figure, the ER activity of ionomers increased with increasing ionomer concentration, and decreasing particle size of the ionomers. For d_{50} =7 µm ionomer/SO system, we obtained τ = 22.43 kPa shear stress (for c = 30 %, m/m), which is an extremely high ER response from industrial point of view. Under an external applied E, organization of ionomer particles take place in SO and stronger chain formations take place. Similar behavior was observed in the studies of polyaniline/SO suspensions by Lengalova et.all., but they have reported maximum τ = 158 Pa shear stress under E = 2.0 kV/mm condition [20].

Effects of electric field strength on viscosity

Electric field strength (E) against electric field viscosity (η_E) obtained at c = 20 % particle concentration is shown in Fig. 5. The decrease in the particle size of ionomers obviously enhanced the ER activity of the ionomers suspensions and we obtained the highest η_E for $d_{0.5}$ =7 µm ionomer/SO system as 278 Pas.

The η_E of ionomer suspensions was increased with increasing E and decreasing ionomers particle size and varied in the following order: 7 µm (278 Pas)>13 µm (263 Pas)>19 µm (253 Pas)>23 µm (247 Pas). During the measurements, the leak current density was measured to be $J<0.35 mA/mm^2$.

When E was applied to the ER suspensions, the polarization forces caused the aggregation of particles and fibril (or chain) formation between the parallel plates occurred. This fibrillar structure is across the direction of the shear field and leads to an increased electric field induced viscosity. Under an applied shearing force, the ionomeric particles are also acted on by the effects of viscous forces, which is due to the hydrodynamic interactions of particles in the suspension.

The formation of particle chain structures in the electric field is reflected by the dependence of viscosity on E. Thus, when E was increased, the apparent viscosity increased at least up to E = 1.5 kV/mm and then this increase was slightly slowed down, which may be attributed to an electrical saturation of the ionomeric suspensions. Similar behavior was reported by Lengalova et al. [21].

Change of shear stress with particle size and electric field strength

The suspension of ionomer particles was expected to become an excellent model system for studying the effect of the particle size on the ER response, and the relationship between the particle size and the shear stress behavior under dc electric field strength is shown in Fig.6. The yield stress (τ_0) can generally be obtained by extrapolating the shear rate to zero. The changes of τ_0 with particle size are as follows: 7 μm (τ_0 = 1216 Pa)<13 μm (τ_0 = 960 Pa)<19 μm (τ_0 = 759 Pa)<23 μm (τ_0 = 546 Pa). These τ_0 values are referred to as the parameter incorporating the interparticle interactions, especially in the fluid under direct current fields [22]. As the particle size of ionomers decreased, the interparticle interactions between the ionomeric particles increased and lead to the shear stress increase. The results in Fig. 6 indicate that the shear stress is proportional to the square of the electric field, i.e. $\tau \propto E^2$. The polarization model of ER fluids can be described by the equation given below;

$$\tau_E \propto \varphi K_f E^2 \beta^2 \qquad (2)$$

where, φ is the volume fraction of particles, K_f is the dielectric permittivity of the base fluid, E is the electric field and β is the relative polarizability at dc or low-frequency ac fields given by Conrad [23] and Davis [24]. It follows from the fact that the interaction force for dipole in an electric field is proportional to the electric field intensity. The net effect will be that the interaction force and consequently the strength of the fluid are dependent on the square of the electric field [25].

Effect of temperature on ER activity

The temperature dependence of the shear stress and log shear stress versus 1/T are shown in Fig. 7 and 8, respectively. The results were obtained at the temperatures of 25, 50, 75, 100 and 125°C. As shown in Fig. 7, the shear stress decreases with increasing particle size and the temperature due to the increasing polarization of the ionomer particles. As reflected from the graph, we obtained a very high shear stress (τ = 19.52 kPa) for d_{50} = 7 μm particle sized ionomer/SO system at T = 25°C and this τ value slowly decreased with increasing temperature and reached to τ = 19.12 kPa at T = 125°C. The loss at the τ of d_{50} = 7 μm particle size ionomer/SO system for ΔT = 100°C temperature change is $\Delta\tau$ = 0.40 kPa (approximately 2%), which is an extremely small τ loss in terms of potential high temperature application areas. The Arrhenius plot of the data in Fig. 8 reveals the average activation energies of about E_a = 2.36 kcal mol^{-1} in this temperature range for all the particle sizes examined.

The variability of ER activity with temperature and moisture content is known to be a major problem with most conventional ER fluids and can limit their high temperature use [26]. The observation that the ionomer system investigated in the present work is not affected from high temperature could prove particularly important from an application stand point [27]. Similar behavior was reported for chitosan/SO suspension [19].

Change of ER efficiency with shear rate

For the possible industrial application of ER suspensions, it is necessary to attain the highest possible electric field viscosity (η_E) at the lowest field-off viscosity (η_0) of the suspension. It is clear that the ER efficiency expressed as the ratio $(\eta_E - \eta_0)/\eta_0$ can be significantly affected by the flow properties of each ionomer suspension particles in addition to particle polarisability.

Change of ER efficiency of ionomer/SO suspensions with shear rate is shown in Fig 9. As is evident, with an applied E (Fig.9a), viscosity of suspensions decreases sharply with increasing shear rate up to $\dot{\gamma}$ = 10 s^{-1}, gives a typical curve of shear thinning non-Newtonian visco-elastic behavior, and then become shear rate independent (Newtonian). For example for d_{50} = 7 μm average particle sized ionomer/SO suspension system, the on field viscosity was observed to be η_E = 136 Pas and the off field viscosity (Fig. 9b) was η_0 = 4 Pas, which is a desired low viscosity in terms of potential industrial applications.

The ER efficiency of ionomer/SO suspension system, which is an extremely important parameter for future design of highly effective ER fluids, characterized by a pseudoplastic decrease of the viscosity with the shear rate is given in Fig.9c [28].

Effect of promoter on ER activity

To investigate the influence of type of additive on the ER activity of ionomer suspensions we tried water and ethanol as promoters. We had electrical break down problems with water but obtained successful ER data with ethanol. In this article just the results obtained for d_{50} = 7 μm particle sized ionomer/SO system containing 1000 ppm ethanol is presented (Fig. 10). It was observed that, the ionomer system studied in the present work was almost insensitive to the amount of moisture present over the range studied. The electric field viscosity of the d_{50} = 7 μm particle size ionomer/SO system was increased from η_E = 278 Pas to η_E = 302 Pas after the addition of 1000 ppm ethanol. The most likely explanation is that the inherent polarisability of the ionic cores was sufficient to overweight the effect of any adsorbed moisture. We observed similar behaviors in our previous studies [14,29].

Effect of frequency on ER activity

The external stress frequency is an important factor for characterizing the dynamic visco-elastic properties of ER fluids [30]. Fig. 11 shows the relationship of electric field (E = 2.0 kV/mm) induced shear stress and frequency. The setting shear stress for this measurement was τ = 10 Pa, which can ensure the measurements are conducted in the small strain region. As seen from the figure, ER activity of all the particle sizes were not affected from the frequency increase up to f = 20 Hz, and then shown sharp decreases, which may be attributed to the dielectric loss and low polarizability of the ionomer suspension [28]. The same features were reported by many other investigators [31,32].

4. Conclusions

1. Colloidal stability of ionomeric salt in silicone oil was found to be 19 days at d_{50} = 7 μm particle size and c = 5 % suspensions concentration.
2. Flow time of suspensions was observed to increase with increasing electric field strength and suspension concentration. The highest flow time was obtained from $d_{0.5}$ = 7 μm particle size and 30% suspension concentration at E = 1.6 kV/mm as 105 s, further increase in E caused solid-like structure.
3. ER activity of ionomer suspensions was observed to increase with increasing field strength and decreasing shear rate and giving a typical shear-thinning non-Newtonian viscoelastic behavior.
4. ER activity of ionomeric salt suspensions was observed to increase with decreasing particle size.
5. It was found that the ionomeric salt system studied in the present work was not sensitive to high temperature and moisture within the limits studied.
6. ER activity of ionomers was found to sharply decrease applied external frequency.

Acknowledgements

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Figure Captions

Figure 1 Sedimentation ratio of ionomer suspensions, (◇) 5 %, (□) 10 %, (△) 15 %, (◆) 20 %, (■) 25 %, (▲) 30 %.

Figure 2 The change of flow time with electric field strength, T = 25°C, c = 30 %. (△) 7 μm, (□) 13 μm, (▲) 19 μm, (■) 23 μm)

Figure 3 The effect of concentration of the particles in silicone oil on the electric field viscosity, E = 2.0 kV/mm, $\dot{\gamma} = 1\ s^{-1}$, T = 25°C, (△) 7 μm, (□) 13 μm, (▲) 19 μm, (■) 23 μm)

Figure 4 Effect of particle concentration on shear stress, E = 2.0 kV/mm, $\dot{\gamma} = 1.0\ s^{-1}$, T = 25°C %, (△) 7 μm, (□) 13 μm, (▲) 19 μm, (■) 23 μm)

Figure 5 The change of viscosity with electric field strength, T = 25°C, c = 20 %, $\dot{\gamma} = 1.0\ s^{-1}$, %. (△) 7 μm, (□) 13 μm, (▲) 19 μm, (■) 23 μm)

Figure 6 Effect of squared electric field on the shear stress, T = 25°C, c = 20 %, $\dot{\gamma} = 1.0\ s^{-1}$, (△) 7 μm, (□) 13 μm, (▲) 19 μm, (■) 23 μm.

Figure 7 Effect of temperature on the shear stress. E = 2.0 kV/mm, c = 20 %, $\dot{\gamma}=1.0\ s^{-1}$, (△) 7 μm, (□) 13 μm, (▲) 19 μm, (■) 23 μm)

Figure 8 Arrhenius plot of log shear stress vs. 1/T derived from Fig. 7. (△) 7 μm, (□) 13 μm, (▲) 19 μm, (■) 23 μm)

Figure 9 Change of electric field viscosity of suspensions with shear rate, T = 25°C, E = 2.0 kV/mm, c = 20 %, d_{50} = 7μm.

Figure 10 Effect of ethanol promoter on ER activity, T = 25°C, E = 2.0 kV/mm, c = 20 %, $\dot{\gamma} = 1.0\ s^{-1}$, d = 7μm (◆) promoter free, 1000 ppm ethanol promoted (△) 7 μm, (□) 13 μm, (▲) 19 μm, (■) 23 μm).

Figure 11 Frequency dependence of storage modulus, (△) 7 μm, (□) 13 μm, (▲) 19 μm, (■) 23 μm), T = 25°C, E = 2.0 kV/mm, c = 20 %.

Table 1 Colloidal stability of ionomeric particles in insulating silicone oil (T = 25°C)

		Concentration					
		5 %	10 %	15 %	20 %	25 %	30 %
Particle size	23μm	15 days	14 days	13 days	11 days	9 days	8 days
	19μm	17 days	15 days	14 days	12 days	11 days	9 days
	13μm	18 days	16 days	15 days	13 days	12 days	11 days
	7μm	19 days	17 days	16 days	14 days	13 days	12 days

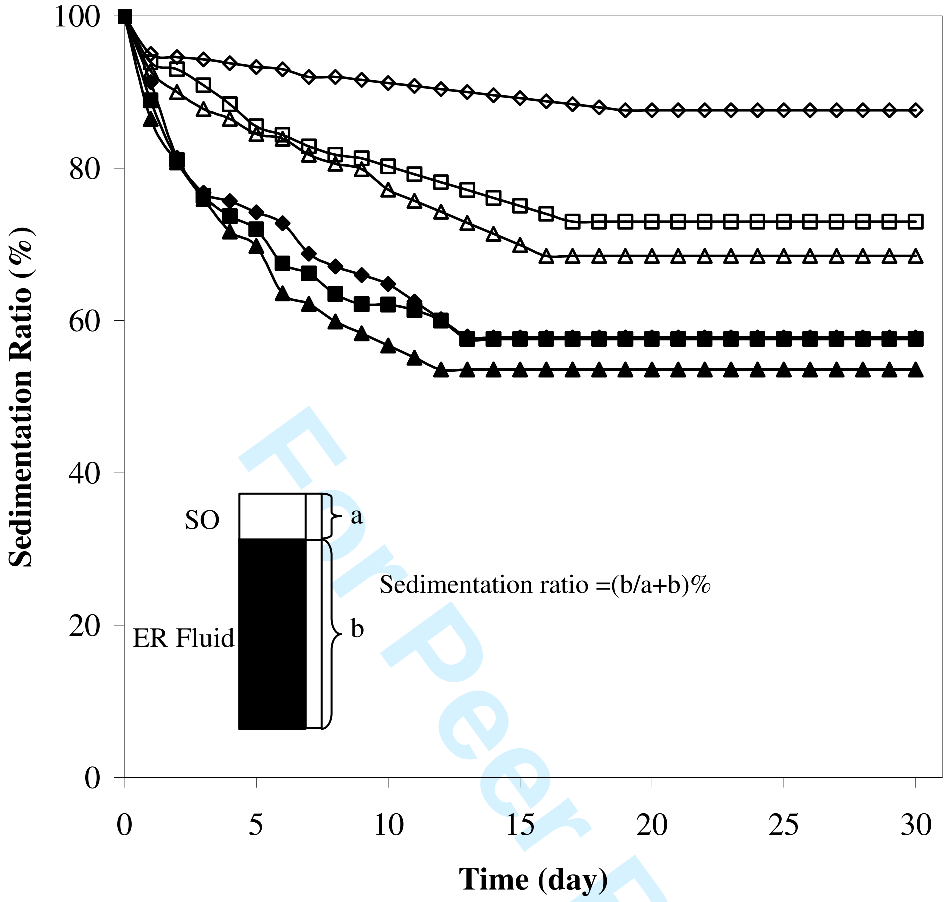
100
80
60
40
20
0
Sedimentation Ratio (%)
0
5
10
15
20
25
30
Time (day)
SO
ER Fluid
a
b
Sedimentation ratio =(b/a+b)%

Figure 1

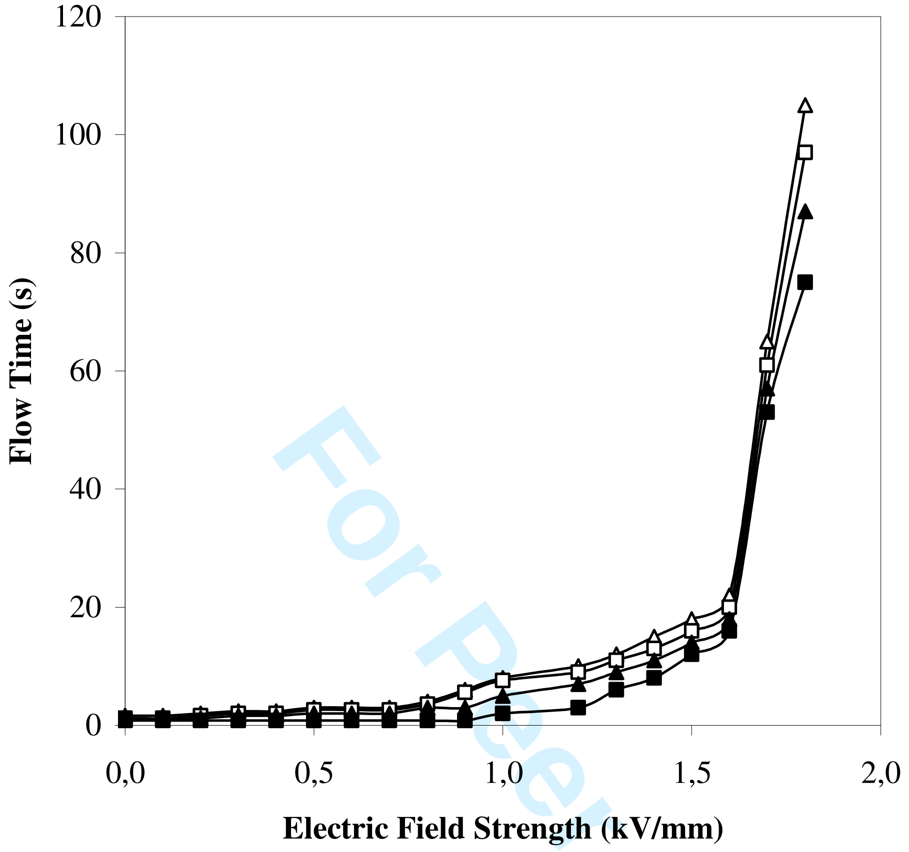
Flow Time (s)
120
100
80
60
40
20
0
0,0
0,5
1,0
1,5
2,0
Electric Field Strength (kV/mm)

Figure 2

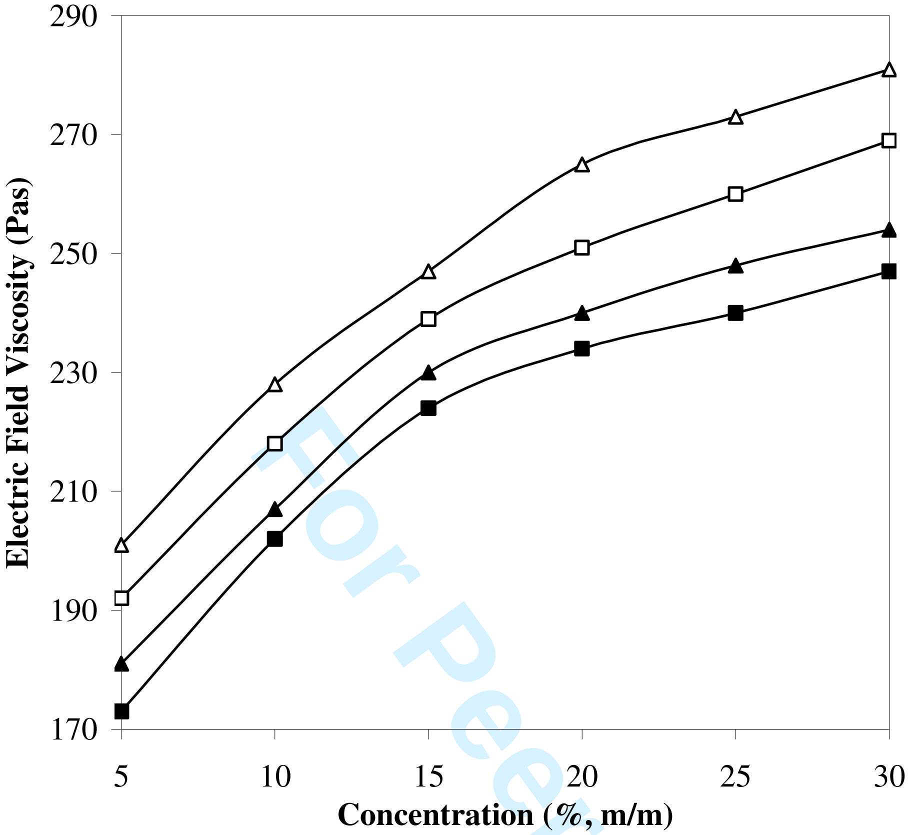
290
270
250
230
210
190
170
5
10
15
20
25
30
Electric Field Viscosity (Pas)
Concentration (%, m/m)

Figure 3

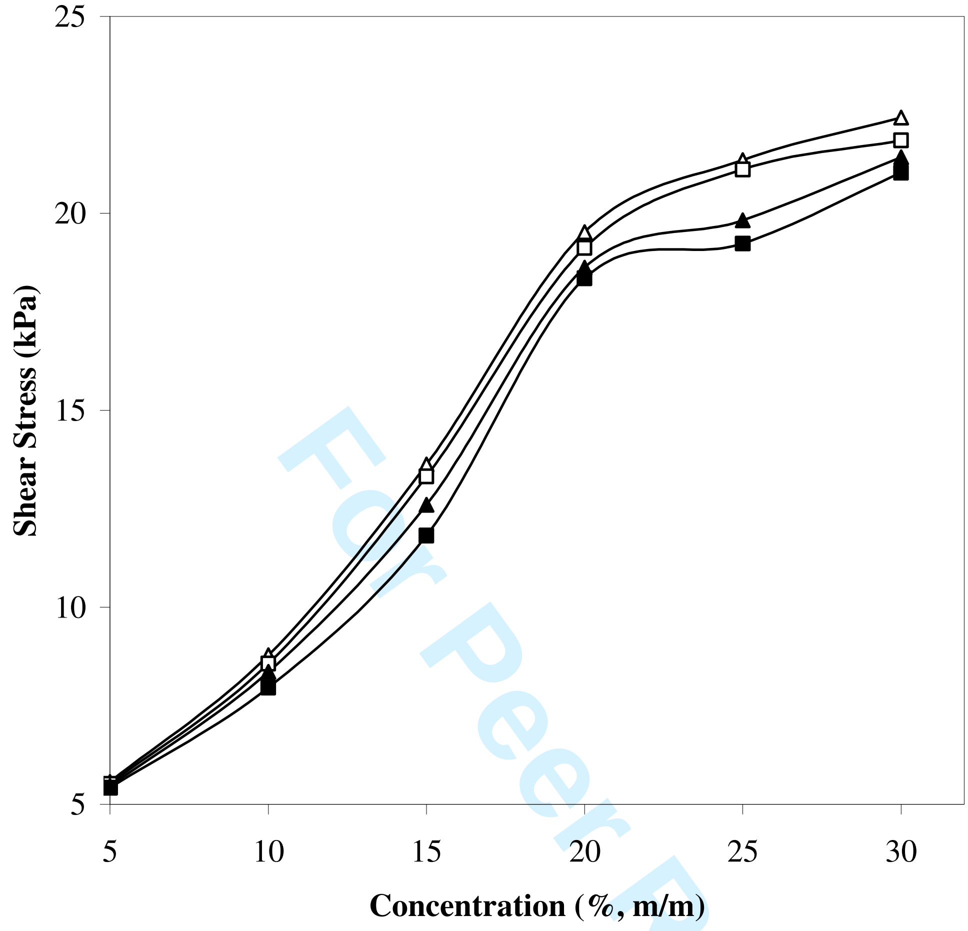

Figure 4

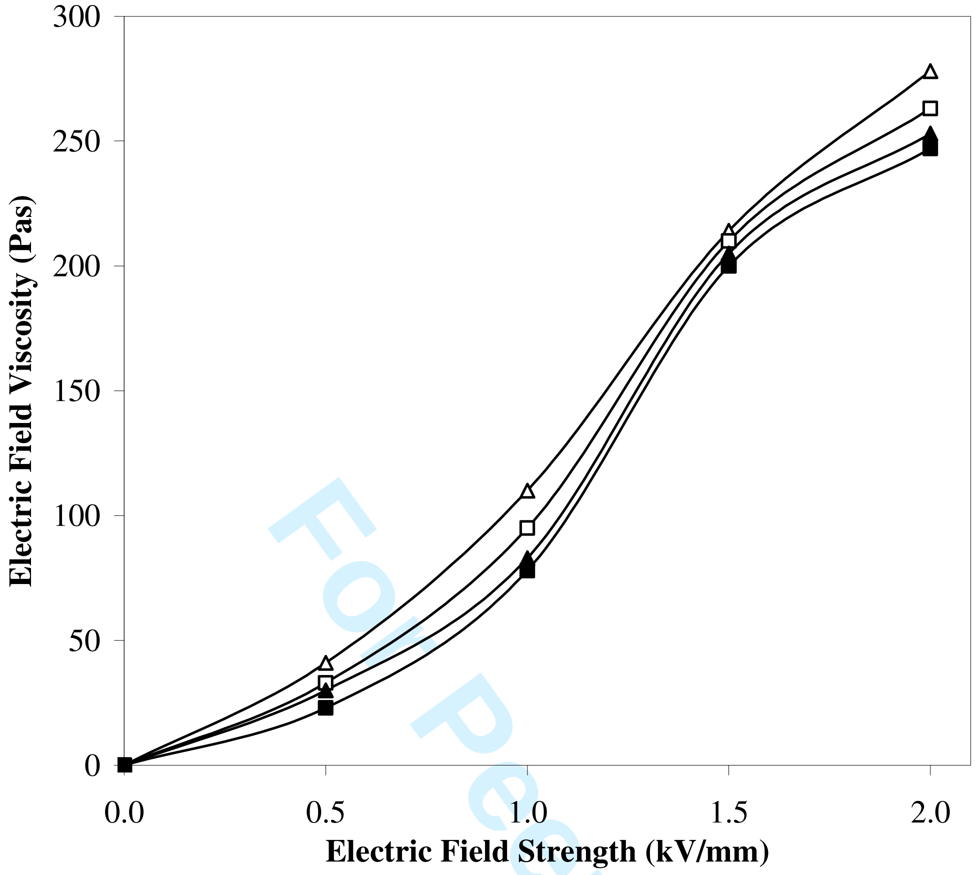
Electric Field Viscosity (Pas)
300
250
200
150
100
50
0
0.0
0.5
1.0
1.5
2.0
Electric Field Strength (kV/mm)

Figure 5

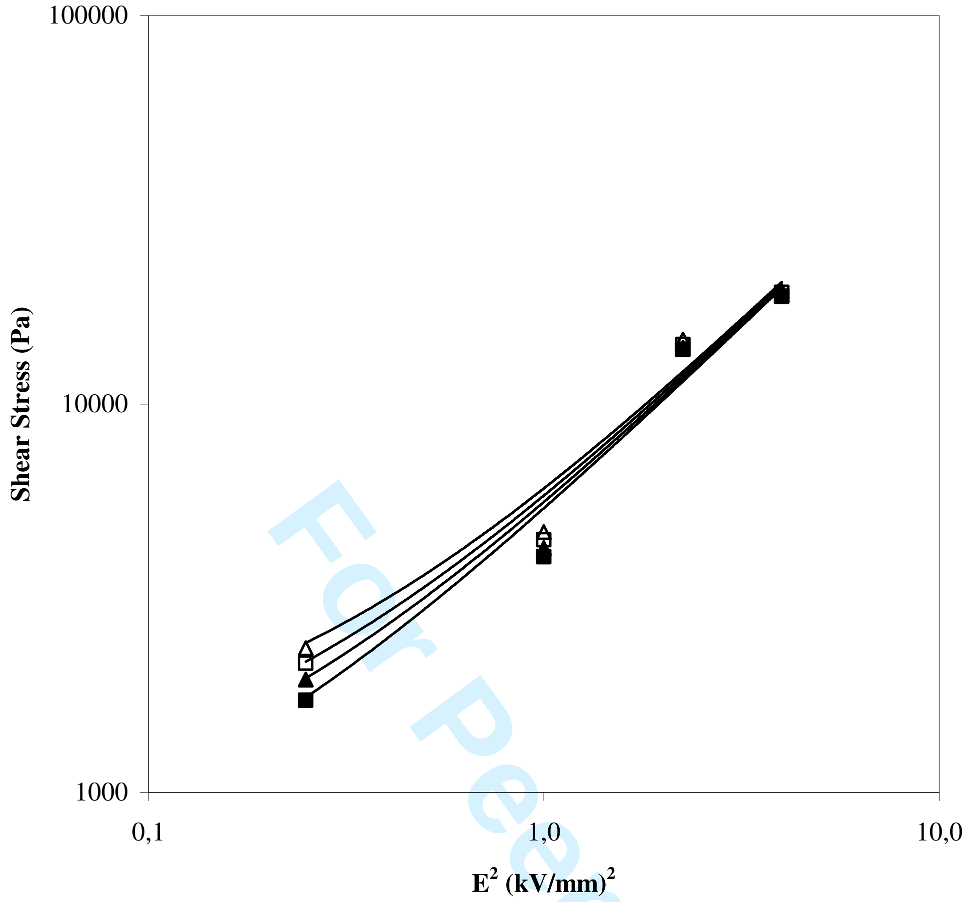

Figure 6

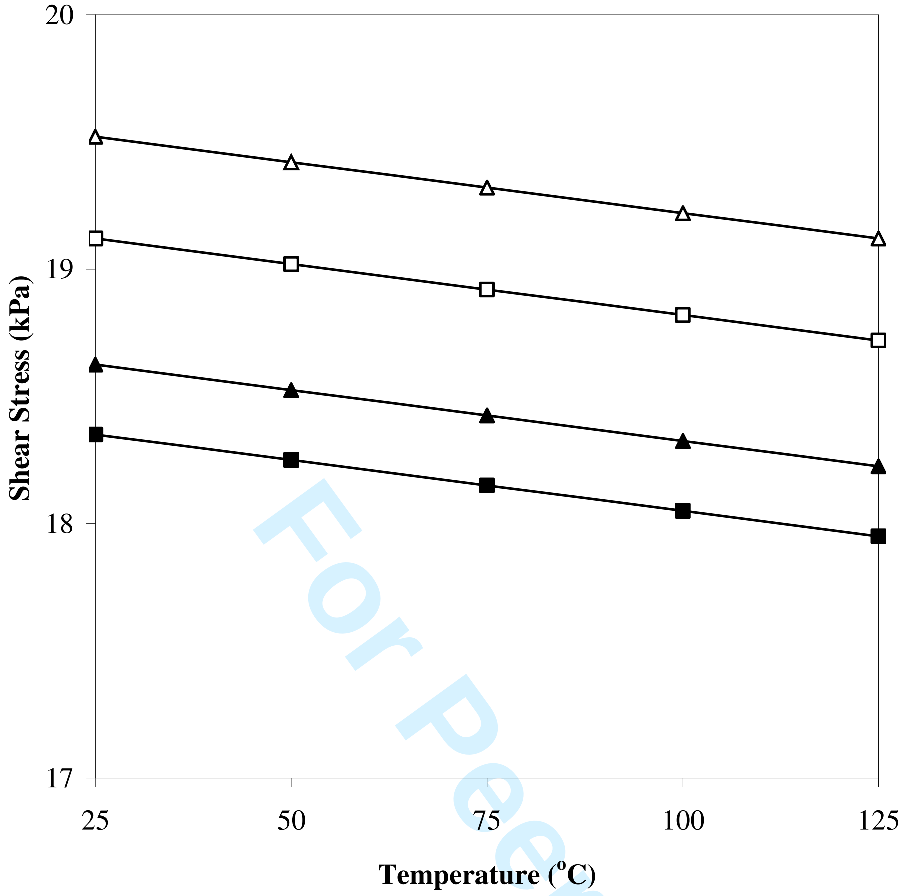

Figure 7

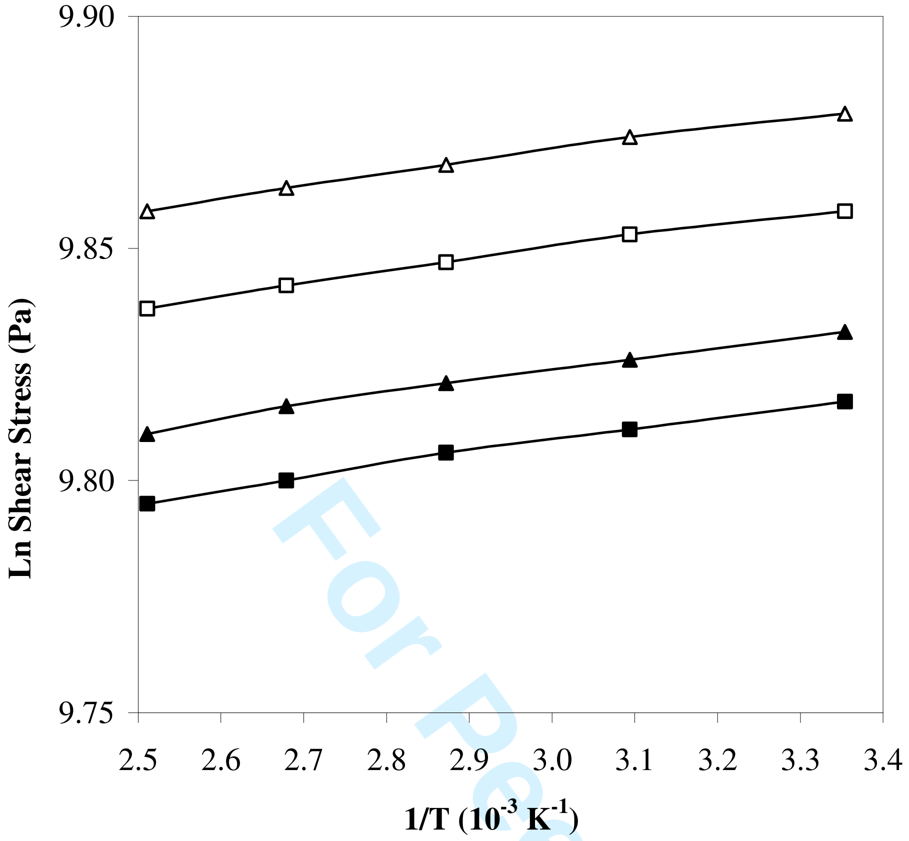
9.90
9.85
9.80
9.75
Ln Shear Stress (Pa)
2.5
2.6
2.7
2.8
2.9
3.0
3.1
3.2
3.3
3.4
1/T (10^{-3} K^{-1})

Figure 8

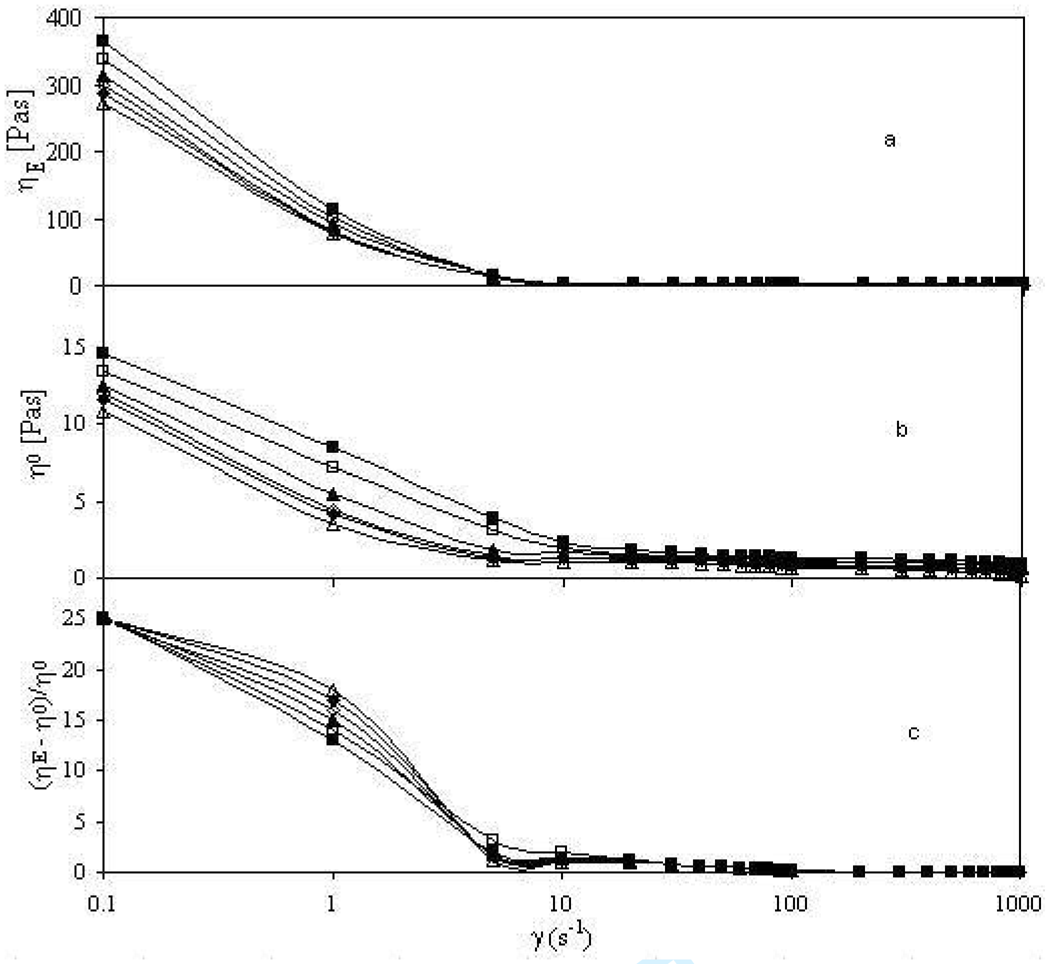
400
300
200
100
0
η_E [Pas]
a
15
10
5
0
η^0 [Pas]
b
25
20
15
10
5
0
$(\eta E - \eta^0)/\eta^0$
c
0.1
1
10
100
1000
$\gamma (s^{-1})$

Figure 9

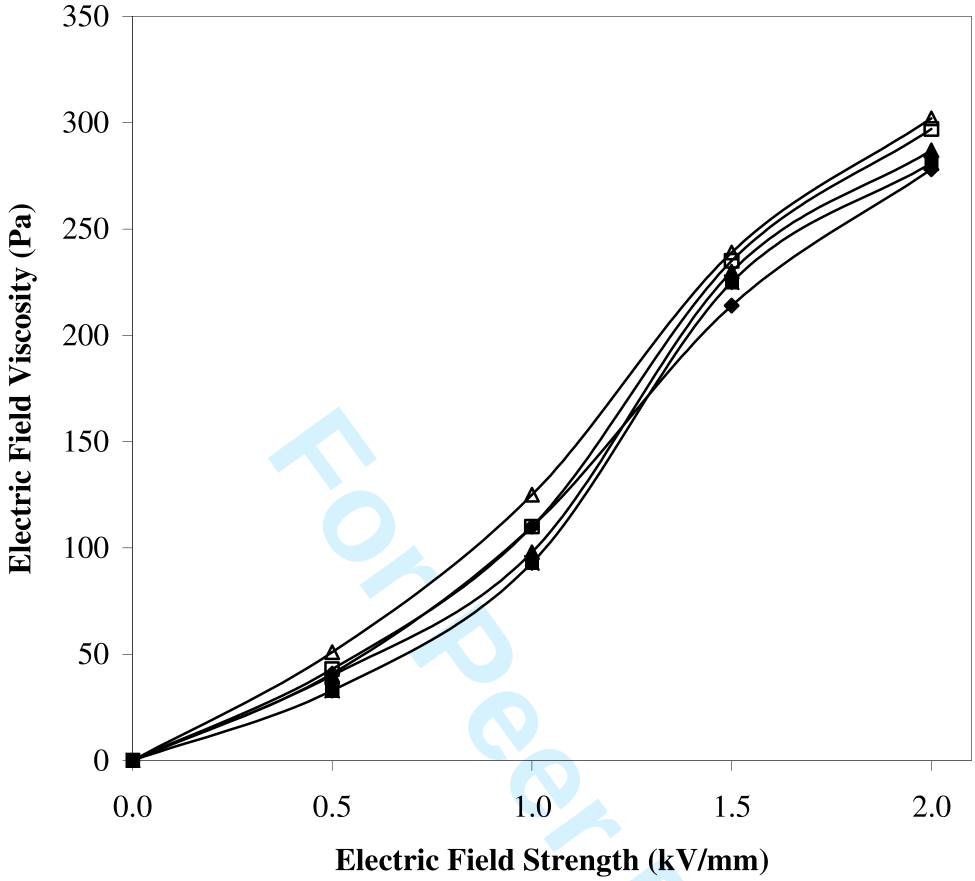
350
300
250
200
150
100
50
0
0.0
0.5
1.0
1.5
2.0
Electric Field Viscosity (Pa)
Electric Field Strength (kV/mm)

Figure 10

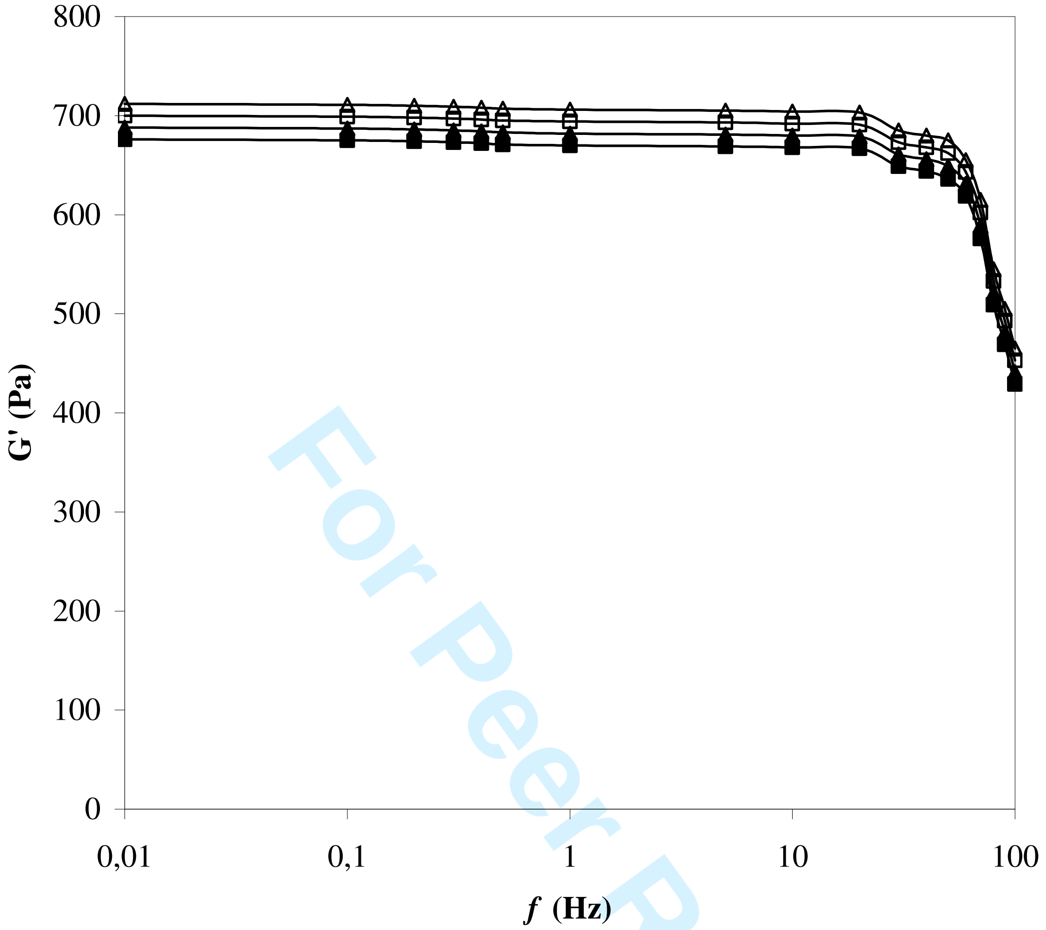

Figure 11

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