Electrorheological Properties of Poly(lithium-2-acrylamido-2-methyl propane sulfonic acid) Suspensions

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ABSTRACT: In this study, poly(2-acrylamido-2-methyl-1-propane sulfonic acid) (PAMPS) was synthesized using potassium persulfate ($K_2S_2O_8$) as initiator. PAMPS ($\overline{M}_n$ = 36,000 g/mol) was partially converted to a lithium salt (PAMPS–Li), and particle size was determined to be 40 μm. Suspensions of PAMPS–Li at various concentrations were prepared in silicone oil, mineral oil, dioctylphthalate (DOP), and trioctyltrimellitate (TOTM) insulating oils. Colloidal stabilities of these suspensions were determined at 20 and 80°C. The PAMPS–Li suspensions were observed to provide an electrorheological (ER) response upon the application of an external dc electric field. ER properties of these suspensions were investigated at various shear rates ($\dot{\gamma}$) and electric field strengths (E). Further, effects of polar promoters and high temperature on ER activity were determined, and excess shear stresses ($\Delta\tau$) were calculated. A shear-thinning non-Newtonian viscoelastic behavior was observed for the PAMPS–Li suspensions. © 2002 Wiley Periodicals, Inc. J Appl Polym Sci 86: 1106–1112, 2002

Key words: rheology; electrorheological fluid; poly(2-acrylamido-2-methyl-1-propane sulfonic acid); ionomers; dispersions

INTRODUCTION

Electrorheological (ER) fluids are suspensions composed of electrically polarizable particles, dispersed in an insulating fluid, and the disperse phase plays an important role in the ER phenomenon. Cellulose,[1] starch,[2,3] silica,[4] zeolite,[5] titanium oxide,[6] tin (II) oxide, and sulfopyropyl dextran[7] have been widely used as dispersed phases in the formulation of the hydrous (wet) ER fluids, which have several problems relating to durability, limited temperature, and colloidal instability in industrial use. Recently, anhydrous (dry) ER fluids (which do not contain water or any other polar solvent in the disperse phase) have been introduced, which consist of polyaniline,[8] polyurethane,[9] polypyrrole,[10] poly(*tert*-butylacrylate-*stat*-lithium acrylate)-*block*-polyisoprene,[11] and polyisoprene-*block*-poly(carboxylithium styrene)[12] as organic dispersed phases. However, these also have shown some problems in applications such as instability of dispersions to sedimentation, a narrow temperature range of operation, and corrosion. The results obtained in this area are critically evaluated in several reviews by Block and Kelly,[13] Jordan and Shaw,[14] Mezger,[15] and Ünal.[16]

To overcome some of these limitations, ion-containing polymers (ionomers) are preferred as a dispersed phase because they receive ever-increasing attention attributed to the dramatic effect that small amounts of ionic groups exert on ultimate polymer properties. When ionic groups are incorporated into the polymer, this not only causes an increase in tensile strength, melting solution viscosity, and glass-transition temperature, but also changes the rheological properties of the materials and makes them useful for ER studies. Although incorporation of a wide variety of ionic groups is possible, carboxylates have thus far received considerable attention.[17,18]

In this research, PAMPS–Li, as a new organic disperse phase, was synthesized and the rheological and electrical properties pertaining to the ER behavior of PAMPS–Li suspensions were investigated in silicone, dioctylphthalate (DOP), trioctyltrimellitate (TOTM), and mineral insulating oils media.

EXPERIMENTAL

Materials

The monomer (2-acrylamido-2-methyl-1-propane sulfonic acid) was purified by vacuum distillation and the initiator ($K_2S_2O_8$; Aldrich Chemicals, Steinheim, Germany) was used as received. The insulating oils (silicone oil, DOP, TOTM, and mineral oil) were all Aldrich products and used after drying at 130°C for 3 h in a vacuum oven, to remove any moisture present.
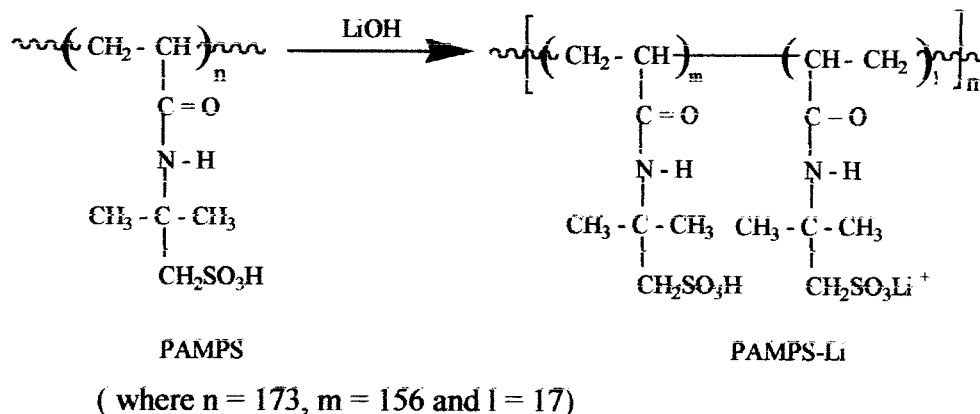
Figure 1 Scheme for salt formation reaction of PAMPS.

Preparation of ionomers of PAMPS

Poly(2-acrylamido-2-methyl-1-propane sulfonic acid) (PAMPS) was synthesized by radical chain mechanism at 80°C, using $K_2S_2O_8$ as the initiator; vacuum dried at 25°C for 48 h; and kept in a desiccator until use. To prepare an ER active ionomer from PAMPS, it had to be partially converted to Li salt (PAMPS–Li) by washing with 10% LiOH(aq). PAMPS–Li was separated from the solvent by freeze-drying. It was then dried in a vacuum oven for 24 h at 50°C and subjected to elemental analysis. The reaction mechanism for the formation of ionomer is described in Figure 1.

Characterization

PAMPS and PAMPS–Li were characterized before ER measurements to be carried out by elemental analysis, FTIR spectroscopy (Mattson Model-1000 FTIR spectrometer), ^{1}H- and ^{13}C-NMR spectroscopy (400 MHz Bruker 400 DPX Avonce spectrometer; Bruker Instruments, Billerica, MA), intrinsic viscosity measurements (in distilled water using a Ubbelohde capillary flow viscometer at 25.0 ± 0.1°C), and end-group analysis (by titrating sulfonic acid units with $0.1M$ NaOH$_{(aq)}$).

The particle size of PAMPS–Li was determined using a Malvern Mastersizer E, version 1.2b particle size analyzer (Malvern Instruments) according to Fraunhofer scattering. Some PAMPS–Li samples were dispersed in ethanol and stirred at a constant temperature of 20°C. The data collected were evaluated by the Malvern software computer according to Fraunhofer diffraction theory.[19]

Preparation of suspensions

Suspensions of PAMPS–Li ionomers were prepared in silicone, DOP, TOTM, and mineral insulating oils at a series of particle concentrations [suspension concentration (c), 5–33 m/m, %]), by dispersing definite amounts of ionomers in calculated amounts of insulating oils according to the formula

$$(m/m, \%) = [m_{polymer}/(m_{polymer} + m_{oil})] \times 100 \quad (1)$$

Determination of sedimentation stability of PAMPS–Li suspensions

Sedimentation stability of PAMPS–Li suspensions prepared in those four insulating oils was determined at 25°C. Glass tubes containing the suspensions, prepared at a series of ionomer concentrations, were immersed into a constant-temperature water bath and formation of first precipitates was recorded to be the indication of colloidal instability.

Rheological and electrical tests

Rheological experiments were carried out for the ionomer suspensions prepared in silicone, DOP, TOTM, and mineral insulating oils. Laminar flow behavior and viscoelastic material properties, which influence processing technology and polymers' stability and consistency, were experimentally determined. Flow rate measurements of the suspensions were carried out between two 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 an external high-voltage dc electric source and a voltmeter; the electrodes were dipped into a vessel containing the ionomer suspension. The stepwise electric fields were then applied to the suspension in the direction perpendicular to the parallel plates. After a few seconds the vessel was removed and the flow time for complete drainage measured, using a digital stopwatch under electric field strengths (E) of $E = 0$ and $E \neq 0$ conditions. This procedure was repeated for each ionomer suspension concentration under various field strengths. The viscosity η of fluids

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TABLE I
Chemical Shifts Obtained for PAMPS by ¹H- and ¹³C-NMR Spectroscopy in D_2O

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Assignment	Chemical shifts[a] (^{1}H, δ, ppm)	Assignment	Chemical shifts (^{13}C, δ, ppm)
–C—N$\underline{H}$—C–	4.9, s	–$\underline{C}$=O	178
–C—C$\underline{H}_2$—SO₃H	3.1, s	–$\underline{C}H_2$—CH–	42
–C$\underline{H}_3$—C—C$\underline{H}_3$–	1.2, s	–CH₂—$\underline{C}$H–	46
–CH₂—C$\underline{H}$–	2.3, m	–$\underline{C}H_2$—SO₃H	60
C$\underline{H}_2$—CH–	1.9, t	$\underline{C}H_3$—C—$\underline{C}H_3$	26
		CH₃—$\underline{C}$—CH₃	55

[a] s, singlet; m, multiplet; t, triplet.

flowing between parallel plate electrodes was calculated using the following equation:

$$\eta = \rho g b \ d^3 t / 12V \qquad (2)$$

where ρ is the density of the fluid, g is the gravitational force, b is the width of plates, t is the flow time, and V is the volume of fluid.

The average shear rate $\dot{\gamma}$, measured at the walls of the parallel plates, was calculated from the relation

$$\dot{\gamma} = 3V / b \ d^2 t \qquad (3)$$

To observe the ER strength of ionomeric suspensions, rotational viscometry (Brookfield DV-1+; Brookfield Instruments) was carried out. The shear rates used were relatively low (1–20 s^{-1}) because of instrumental limitations. To measure the viscosity of a liquid, a spindle was simply immersed inside the suspension container, the motor was switched on, and the viscosity was read on the calibrated dial of the instrument. For measuring the viscosity of ionomeric suspensions under an applied field, those parallel plate electrodes were again immersed into the fluid container, keeping the 0.5-cm gap between the electrodes constant; an electric field was then created in the fluid perpendicular to the parallel plates and the spindle was forced to rotate. The voltage used in both flow rate and rotational experiments was supplied by a 0–10 kV (with 0.5-kV increments) dc electric field generator, which enabled resistivity to be created during the experiments. All the measurements were carried out at 20 and 80°C constant temperatures.

RESULTS AND DISCUSSION

Characterization

PAMPS was synthesized by radical chain mechanism and characterized by a series of methods before ER measurements were carried out. End-group analysis was first performed to determine the number-average molecular mass of PAMPS, and from the acid number of polymer, $\overline{M}_n$ was calculated to be 36,000 g/mol.

From capillary viscometry measurements, intrinsic viscosity of PAMPS was determined to be 0.4 dL/g.

Elemental analysis results were used as a check for purity and percentage conversion of sulfonic acid groups into lithium salt by comparison with calculated composition. It was obtained from the elemental analysis results that the amount of Li in PAMPS was 0.14 ± 0.01%, within the experimental error (calculated to be 0.15%). The PAMPS and its Li salt (PAMPS–Li) were assumed to have the chemical structure given in Figure 1.

FTIR spectra of PAMPS and PAMPS–Li showed the expected distinctive absorptions. The absorptions of PAMPS at 1150–1210, 1550, 1650, 2900, 3300–3400, and 3450–3570 cm^{-1} are typical of —S=O, —C—N, —C=O, —C—H, —N—H, and —O—H stretching vibrations, respectively. The O—H band of PAMPS at 3450–3570 cm^{-1} became narrow at the FTIR spectrum of PAMPS–Li.

Expected distinctive chemical shifts of particular groups in PAMPS obtained from ¹H- and ¹³C-NMR spectroscopy are given in Table I, in view of the structure given in Figure 1.

Because the origin of ER activity is interparticle interactions,[8] which cause particles to form chainlike structures, particle size is an important parameter. For high ER activity, small particle sizes are sought against colloidal instability and rapid polarization of suspended particles, when an electric field is applied between the electrodes. Although size ranges of 0.04 to 50 μm are specified in the literature,[13] careful studies on the effect of the particle size distribution on the ER response have not been fully carried out. In this study crude PAMPS–Li was ground-milled for a few hours, and then from the particle size measurements the average diameter of particles was determined to be 40 μm.

Sedimentation stability of suspensions

Despite the recent activities surrounding ER fluids and ER effect, few efforts have focused on the colloidal stability of these suspensions. Moreover, few investi-

TABLE II
Sedimentation Stability Results of PAMPS–Li Suspensions (T = 20°C)

Oil	Concentrations (m/m, %)					
	5	10	15	20	25	33
Silicone	60 days	55 days	52 days	41 days	29 days	18 days
Mineral	45 days	30 days	25 days	21 days	17 days	10 days
TOTM	10 days	9 days	7 days	5 days	4 days	2 days
DOP	8 days	6 days	5 days	4 days	2 days	1 day

gations have probed the colloid chemistry of ER fluids.[11,12] Sedimentation stabilities of PAMPS–Li suspensions, prepared in those four insulating media in a series of concentrations, were determined at 20°C and the results obtained are tabulated in Table II.

As reflected from the table, sedimentation stability of suspensions was increased with decreasing particle contents of the suspensions. Maximum colloidal stability was observed with silicone oil suspensions for 5% concentration as 60 days. This is between the colloidal stability value of polyisoprene-*block*-poly(carboxylithium styrene) copolymer[12] (52 days) and polypyrrole[10] (63 days), both prepared in silicone oil at 5% particle concentration. Because the maximum colloidal stability was achieved in silicone oil, effects of temperature and promoters onto ER activity were also carried out only for silicone oil suspensions.

Electrorheology

Because the ER phenomena are widely attributed to the chaining of micron-sized polarizable particles, when subjected to an external electric field, flow and rotational viscometry studies are conducted to observe the viscosity change and ER response of PAMPS–Li suspensions.

Flow measurements

To observe the effect of dc electric field on the ER activity, flow-rate measurements were carried out on the PAMPS–Li suspensions. For this purpose, PAMPS–Li suspensions were prepared at a series of concentrations (5–33 m/m, %) in four insulating oils, and flow times were measured under $E = 0$ and $E \neq 0$ conditions. Results obtained just from silicone oil suspensions are depicted in Figure 2. As reflected from the graph, flow times of suspensions showed only slight increases with increasing electric field strength but sharp increases were observed for all the concentrations studied after threshold energies were supplied. From Figure 2 it was determined that the amount of threshold energy required was observed to increase with decreasing suspension concentration (500 V/mm for $c = 33\%$, and 1000 V/mm for $c = 5\%$). Minimum and maximum flow times were obtained

for the PAMPS–Li/silicone oil system as 25 and 88 s for $c = 5\%$ and $c = 33\%$, respectively.

Similar trends were observed from the same kind of graph drawn for the suspensions prepared in the other three insulating oils, and the maximum flow times determined (for $c = 33\%$) were in the order of silicone oil (88 s) > mineral oil (85 s) > TOTM (44 s) > DOP (33 s).

Flow times given for the suspensions are the maximum flow times, which could be observed under the applied field. When E was further increased, bridge formation occurred for all the suspensions and no flow was observed. Similar behavior was reported for alumina suspensions in silicone oil.[20]

Rotational viscometry

Effect of concentration on electric field viscosity. The change in electric field viscosity ($E \neq 0/E = 0$) with suspension concentration of PAMPS–Li particles dispersed in silicone oil at various shear rates ($\dot{\gamma} = 1$–20 s^{-1}) and constant electric field strength ($E = 700$ V/mm) and temperature (20°C) is shown in Figure 3.

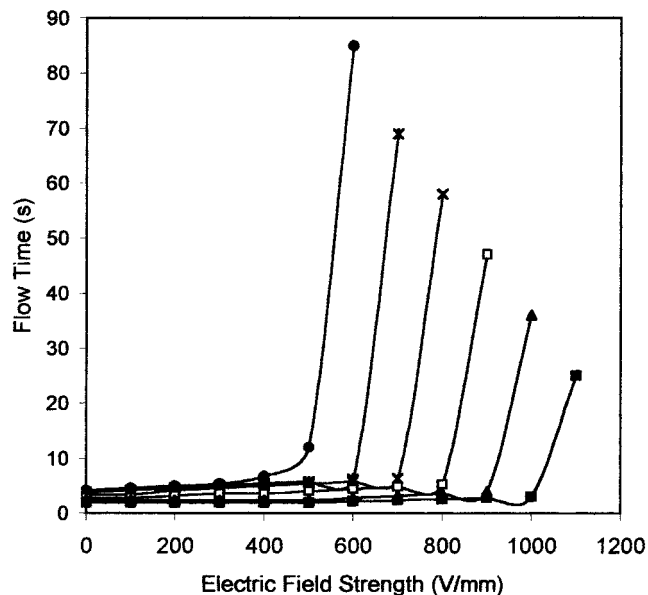**Figure 2** Effect of electric field strength on flow time. c (m/m, %): (■) 5, (▲) 10, (□) 15, (×) 20, (*) 25, (●) 33.
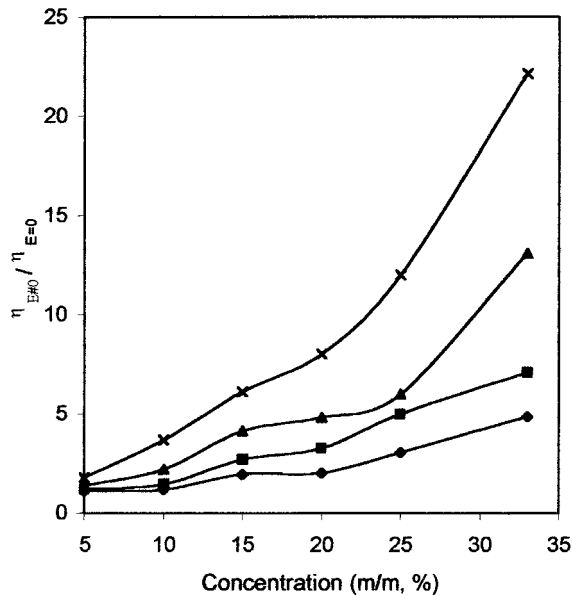
Figure 3 Change of viscosity with concentration. $T = 20°C$ and $E = 0$–700 V/mm. γ (s^{-1}): (◆) 1; (■) 4; (▲) 10; (✗) 20.

As reflected from the graph, electric field viscosity increases with increasing suspension concentration and decreasing shear rate, showing a typical shear-thinning non-Newtonian behavior. A similar trend was also observed from the suspension concentration versus electric field viscosity graphs drawn for suspensions prepared in the other three insulating oils. The maximum electric field viscosity changes observed were in the order of silicone oil (30 Pa·s) > mineral oil (23 Pa·s) > DOP (22 Pa·s) > TOTM (20 Pa·s) at 1 s^{-1} constant shear rate.

As a result of polarization forces acting between the PAMPS–Li ionic particles, electric field viscosity (or ER activity) increases with increasing PAMPS–Li concentration in the suspensions. The relationship between applied electric field (E) and polarization forces (F)[21] is expressed by the following:

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

where ϵ_2 is the dielectric constant of the particle, ρ is the distance between particles, and r is the particle radius.

According to this equation, the distance between the particles decreases with increasing suspension concentration because of increased polarization force and electric field viscosity.[12] Similar behavior was observed in the studies of polyaniline suspensions.[8]

Effects of electric field strength on viscosity. Figure 4 shows the change in viscosity with electric field strength for the PAMPS–Li/silicone oil system at various shear rates ($\dot{\gamma} = 1$–20 s^{-1}) and 33% constant suspension concentration. As reflected from the graph increase in the ER activity of suspensions is directly

proportional to the change in applied electric field strength, and inversely proportional to the change in shear rate.

Similar behavior was also observed for the other suspensions prepared in those three insulating oils and maximum electric field viscosities obtained were changed in the order of silicone oil (120 Pa·s) > DOP (110 Pa·s) > mineral oil (78 Pa·s) > TOTM (75 Pa·s).

When PAMPS–Li suspensions were subjected to an external applied electric field, due to the polarization forces acting between the particles, chain formation took place between the electrodes. The fibrillar structure formed across the direction of shearing force leads to increased viscosity of suspensions. PAMPS–Li particles are affected by the hydrodynamic interactions as well by the viscous forces (F), which have the magnitude of[22]

$$F = 6\pi\eta_s r^6 \dot{\gamma} \tag{5}$$

where η_s is the viscosity of suspension, r is the radius of particle, and $\dot{\gamma}$ is the average shear rate. On the other hand, when the shear rates were increased, the viscosity of suspensions almost became independent of the applied field strength and showed very little response to the increase in electric field strength. It may be concluded that, at high shear rates, the viscous forces are dominant, and the fibrillar structure of the suspension does not vary appreciably with applied electric field strength. Similar behavior was reported by Jordan and Schwendt in the ER study of zeolite/silicone oil suspensions.[23]

Change in excess shear stress with electric field strength. The effect of electric field strength on excess shear stress was studied at different electric field strengths
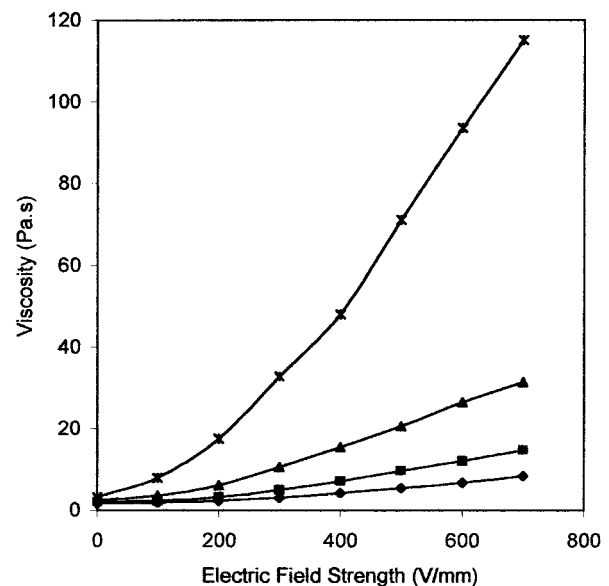

Figure 4 Effect of electric field strength on viscosity. $T = 20°C$ and $c = 20\%$. γ (s^{-1}): (◆) 1; (■) 4; (▲) 10; (✗) 20.
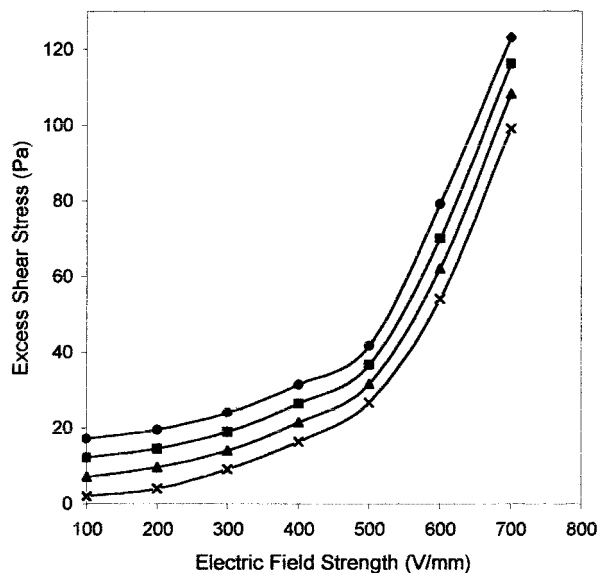
Figure 5 Change of excess shear stress with electric field strength. $\dot{\gamma} = 20\ \text{s}^{-1}$, $T = 20°C$, and $c = 20\%$: (◆) silicone oil; (■) mineral oil; (▲) TOTM; (✕) DOP.

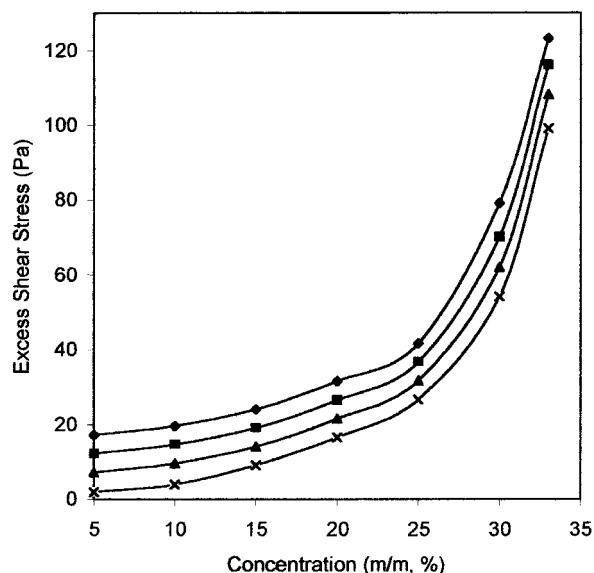**Figure 6** Change of excess shear stress with concentration. $\dot{\gamma} = 20\ \text{s}^{-1}$, $T = 20°C$, and $E = 700\ \text{V/mm}$: (◆) silicone oil; (■) mineral oil; (▲) TOTM; (✕) DOP.

and the results obtained are shown in Figure 5. It is seen that keeping the shear rate ($\dot{\gamma} = 20\ \text{s}^{-1}$), suspension concentration ($c = 33\ \text{m/m, \%}$) and temperature constant ($T = 20°C$), increasing electric field strength up to 700 V/mm accompanied by a significant enhancement in the excess shear stress. The increase in the excess shear stresses were in the order of silicone oil (130 Pa) > mineral oil (125 Pa) > TOTM (115 Pa) > DOP (102 Pa). The results obtained are consistent with the previous studies carried out by Yu and Shaw on the polypyridinium particles in a mineral oil.[24]

Change in excess shear stress with concentration. The effect of the variation of suspension concentrations on the excess shear stress was evaluated by changing the concentrations of suspensions in the 5–33% range, by keeping the shear rate ($\dot{\gamma} = 20\ \text{s}^{-1}$), electric field strength ($E = 700\ \text{V/mm}$), and temperature ($T = 20°C$) constant; the results obtained are given in Figure 6. It is clear that the excess shear stress increase is directly related to the suspension concentrations. Changes of excess shear stress were in the order of silicone oil (130 Pa) > mineral oil (125 Pa) > TOTM (115 Pa) > DOP (102 Pa). As seen from the results, maximum excess shear stress was obtained again in the PAMPS–Li/ silicone oil system. The increase in the excess shear stress with increasing concentration may be attributed to the increased interparticle interactions with increasing particle concentration in the suspension, which results with an enhanced ER activity. Similar behavior was reported in the literature by Gow and Zukoski for polyaniline/silicone oil suspensions.[8]

Effect of shear rate and temperature on viscosity. Change of viscosity of suspensions with shear rate (at $c = 33\%$, $E = 0\ \text{V/mm}$, and $E = 700\ \text{V/mm}$, $T = 20$ and $80°C$

conditions) were investigated in those four insulating oils and just the results obtained in silicone oil are shown in Figure 7. As seen from the graph, viscosity of suspensions sharply decreases with increasing shear rate, giving a typical curve of shear-thinning non-Newtonian viscoelastic behavior.[25] It was also observed that high temperatures caused only a slight decrease on the viscosity of suspensions at low shear rates within experimental error. Similar trends were also observed for the suspensions prepared in the other three insulating media. These observations are
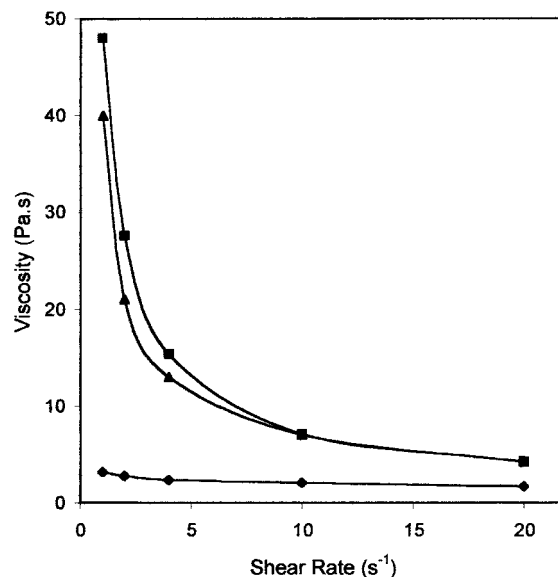

Figure 7 Variation of viscosity with shear rate. $T = 20°C$ and $c = 20\%$: (◆) 0 V/mm, 20°C; (▲) 700 V/mm, 80°C; (■) 700 V/mm, 20°C.

consistent with the results reported by Kimura and coworkers for liquid crystalline polymer/dimethylsiloxane blends.[26]

Effect of promoter on ER activity. The influence of moisture on the ER activity was also investigated by adding polar solvents, such as glycerol and ethanol, at 2400 ppm concentration into PAMPS–Li/silicone oil suspensions at 33% particle concentration. It was observed that ER activity of suspensions was sensitive to the presence of glycerol and ethanol and addition of promoters caused an increase in the electric field viscosity (ER activity) of the suspensions. Electric field viscosities of glycerol- and ethanol-promoted suspensions were observed to increase from 2.4 to 3.2 Pa·s and from 2.4 to 3.9 Pa·s, respectively. Most ER formulations require the presence of a polar promoter and, for such systems, complete drying destroys the ER effect.[1,13] Alcohol, ethylene glycol, dimethylamine, and formamide[13] have been reported in the literature as polar promoters. 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.[27] Enhancement of the ER activity of the PAMPS–Li/silicone oil system by the addition of polar promoters, investigated in this work, could prove particularly important for industrial applications.

CONCLUSIONS

1. Colloidal stability of polymeric salt in silicone oil was found to be 60 days ($c = 5\%$).
2. Flow times of suspensions were observed to increase with increasing electric field strength and suspension concentration in all the insulating media studied. The highest flow time was obtained in silicone oil as 88 s ($c = 33\%$, $E = 700$ V/mm).
3. ER activity of all the suspensions was observed to increase with increasing electric field strength, concentration, and decreasing shear rate.
4. Excess shear stress was found to sharply increase with increasing field strength and suspension concentration.
5. The viscosity of all the suspensions was decreased sharply with increasing shear rate, show-

ing a typical shear-thinning non-Newtonian viscoelastic behavior.
6. The addition of polar promoters enhanced the ER activity of the PAMPS–Li/silicone oil system within the limits studied.
7. The performance of insulating oils studied was in the order of silicone > mineral > DOP > TOTM.

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