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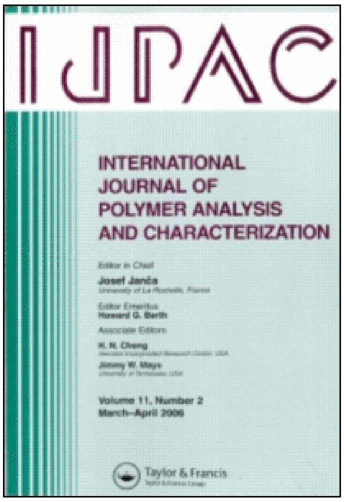

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Mustafa Yavuz[a]; Ayşegül (Uygun) Gok[a]; Songul Sen[b]; Halil Ibrahim Unal[c]
[a] Chemistry Department, Science and Arts Faculty, Suleyman Demirel University, Isparta, Turkey [b] Department of Chemistry, Faculty of Arts and Science, Mehmet Akif Ersoy University, Burdur, Turkey
[c] Chemistry Department, Science Faculty, Gazi University, Ankara, Turkey

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Electrorheological Properties of Polyaniline/Red Mud Composite

Mustafa Yavuz,[1] Ayşegül (Uygun) Gok,[1] Songul Sen,[2] and Halil Ibrahim Unal[3]
[1]Chemistry Department, Science and Arts Faculty, Suleyman Demirel University, Isparta, Turkey
[2]Department of Chemistry, Faculty of Arts and Science, Mehmet Akif Ersoy University, Burdur, Turkey
[3]Chemistry Department, Science Faculty, Gazi University, Ankara, Turkey

Abstract: In this study, electrorheological (ER) properties of polyaniline (PANI) and polyaniline/red mud (RM) composites were investigated. Polyaniline and polyaniline/red mud (PRM) composites were prepared by oxidative polymerization. PANI and PRM particle-based ER suspensions were prepared in silicone oil (SO) and their ER behavior was investigated as a function of concentration, shear rate, electric field strength, frequency, and temperature. Sedimentation stabilities of these suspensions were determined. ER activities of all the suspensions were observed to increase with increasing electric field strength, dispersed particle concentration, and decreasing shear rate. The suspensions show shear thinning non-Newtonian viscoelastic behavior, in which viscosity of suspension decreases with increasing shear rate. Shear stress of composite suspensions increases linearly with increasing applied electric field strength and concentration of the particles. Effect of high temperature on ER activity of ER fluids system was also investigated.

Keywords: Composite; Electrorheological suspensions; Polyaniline; Red mud

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Address correspondence to Mustafa Yavuz, Chemistry Department, Science and Arts Faculty, Suleyman Demirel University, Isparta, Turkey. E-mail: yavuz@fef.sdu.edu.tr, efeym@yahoo.com

INTRODUCTION

Electrorheological (ER) fluid is one kind of smart material and typically consists of electrically polarizable particles dispersed in low-dielectric oils. An ER fluid exhibits reversible changes in its rheological properties as a function of electric field strength. In the presence of strong electric field strength, the particles orient themselves along the electric field line. Once the particles have aligned, there is an increased attractive force between particles due to the proximity of opposite poles. The attractive force between dispersed particles causes chain formation along the electric field lines in a very short period of time (a few milliseconds), and these chains then aggregate to form columns; thus the viscosity can be increased.[1] Furthermore, the ER fluid exhibits an enhancement of shear stress, and in particular it develops a shear stress at zero shear rates. Thus, ER fluids behave like Bingham fluids under applied electric field strength.[2] Because of these characteristics of ER fluids, such as short response time, low power consumption, smoothness of operation, and mechanical simplicity, they have been considered for various applications in mechanical engineering such as engine mounts, shock absorbers, clutches, ER valves, robotic arms, and several control systems.[3,4] Recently, ER fluids have found some newly developed application areas such as human muscle stimulators, spacecraft deployment dampers, seismic controlling frame structures, ER tactile displays, and photonic crystals.[5]

Most applications require fluids that possess a large field-induced yield stress, are stable to settling and irreversible aggregation, are environmentally benign, and draw limited current.[6]

To overcome some of the limitations (i.e., thermal stability, aggregation, and corrosion) of water-based systems, dry-based systems have been investigated with anhydrous particles. Anhydrous ER suspensions using polymer particles, i.e., poly(acene quinone) radicals,[7] polyaniline,[8,9] polypyrrole,[10] copolyaniline,[11] poly(methyl methacrylate)-*block*-polystyrene,[12] polystyrene-*block*-polyisoprene,[13] and inorganic-organic composite particles[14] were reported in the literature. Among various ER materials, polymeric particles have attracted great interest because of their thermal stability at high temperature, lower density, and ease of preparation.[15] Conducting polymer-inorganic composites are often prepared to improve the physical and chemical properties of conducting polymers by coating a conducting polymer on inert core inorganic particles or forming polymeric composites by carrying out the polymerization in the presence of inorganic particles.[16] Recently, conducting polymer-coated inorganic-organic composite particles (polyaniline-coated inorganic particles[17] and conducting polymer-inorganic composites[18] were used for ER suspensions, and they showed promising ER responses.

It is also important to investigate fluid sedimentation and sludge deposit formation.[19] Most of the studies in the literature are focused on the ER effect of suspensions, but very few have investigated the colloidal stability of suspensions. Another target for ER fluids is long service stability, particularly at high temperatures.[20]

In this study, polyaniline (PANI) and polyaniline/red mud composite (PRM), a novel organic dispersed phase, were synthesized, characterized by several techniques. Their suspensions were prepared in silicone oil (SO) to investigate the ER properties. Red mud was a waste product of Etibank Seydisehir Aluminum Production Plant (Konya, Turkey) and costs nothing. Results of the synthesis and characterization part of the PRM composites were recently published.[21]

EXPERIMENTAL SECTION

Materials

Aniline was distilled under vacuum prior to use (Fluka, Germany).

Silicone oil was used after drying at 130°C for 3 h in a vacuum oven to remove any moisture present ($\rho = 0.97\,\text{g/cm}^3$, $\eta = 1000\,\text{mPas}$, $\epsilon = 2.61$).

Red mud was a waste product of the factory provided by Etibank Seydişehir Aluminum Plant (Konya, Turkey), and its analytical data are shown in Table I. All the other chemicals were analytical grade (Aldrich, USA) and used as received.

Synthesis of PANI and PRM Composites

A weighed amount of red mud powder was suspended in 100 mL of 1 M HCl, then 0.8 mL of aniline was injected into this suspension, which was stirred magnetically for 2 h. Thereafter, $(NH_4)_2S_2O_8$ (3.99 g) was added to the mixture, which was stirred for 24 h. The precipitated composite was filtered and rinsed with 1 M HCl and distilled water. The target mass loading of monomer in the composites was from 13 to 31% m/m. PANI

Table I. Chemical composition of RM

	Chemical composition						
	Al_2O_3	Fe_2O_3	TiO_2	Na_2O	CaO	SiO_2	Ig loss
Composition ratio (%)	18.71 ± 0.59	39.70 ± 0.67	4.90 ± 0.54	8.82 ± 0.96	4.47 ± 0.56	14.52 ± 0.37	8.35 ± 0.40

homopolymer was synthesized under the same conditions for comparison with the composites.

PANI and PRM composite were characterized by Fourier transform-infrared (FT-IR) and ultraviolet (UV) spectroscopy, atomic force microscopy (AFM), thermogravimetric analysis (TGA), scanning electron microscopy (SEM), and X-ray diffraction (XRD) analysis. The details are given in our previous article.[21]

Preparation of Suspensions

Suspensions of PANI and PRM composite were prepared in SO at a series of concentrations ($c = 1\text{–}7.5\%, \mathrm{m/m}$) by mixing definite amount of dispersed phase with calculated amount of continuous phase according to the formula:

$$(\%, \mathrm{m/m}) = [\mathrm{m}_{\text{dispersed phase}}/(\mathrm{m}_{\text{dispersed phase}} + \mathrm{m}_{\text{oil}})] \tag{1}$$

Determination of Particle Size and Conductivity

Particle size of the samples was determined using a Malvern Mastersizer E, version 1.2b, particle size analyzer (UK). During the particle size measurements, some samples were dispersed in ethanol and stirred at a constant temperature of 20°C. The data collected were evaluated according to Fraunhofer diffraction theory by Malvern Software. Conductivity of PANI and PRM composite was determined by the four-probe technique.

Sedimentation Stability Measurements

The colloidal stability of suspensions against sedimentation was determined at constant temperature (25.0° ± 0.1°C). Glass tubes containing the suspensions were immersed in a constant temperature water bath, and formation of first precipitates was taken to be the indication of colloidal instability.

Rheological Measurements

Suspensions were mechanically stirred before each measurement against sedimentation. Rheological properties of the suspensions were determined with a Termo-Haake RS600 parallel plate Electro-rheometer (Germany). The gap between the parallel plates was 1.0 mm, and the diameters of the upper and lower plates were 35 mm. All the experiments were carried out at various temperatures (25°–125°C, with 25°C increments). The voltage used in these experiments was supplied by a 0–12.5 kV (with 0.5 kV increments) dc electric field generator (Fug Electronics, HCL 14, Germany), which enabled resistivity to be measured during the experiments.

RESULTS AND DISCUSSION

The results obtained from particle size measurements of PANI and PRM composites are given in Table II. Particle sizes of the samples were in the range of 7.78–14.42 μm. The composites exhibited conductivity values in the range of 4.2×10^{-3}–5.2×10^{-2} Sm^{-1} with changing RM percentage,[21] which are slightly above the values expected for ER active materials.[4] That is why difficulties occurred during ER measurements of suspensions having dispersed particle concentrations higher than 7.5%, which probably indicates lower concentrations for these composites. Low dispersed particle concentration is suitable in terms of applications and industrial uses.

Sedimentation Stability of Suspensions

Despite recent studies on ER fluids and the ER effect, little effort has focused on the colloidal stability of these suspensions. A few investigations have studied the colloidal chemistry of ER fluids.[15] When the density of the particles is not the same as that of the dispersing medium, the particles of micron size settle down according to Stokes' law.[22] In order to solve the traditional problem of particle sedimentation, several workers have developed different approaches.[23] Density difference between dispersed phase and continuous phase plays an important role in the sedimentation ratio of ER fluids.[24] Figure 1 shows the sedimentation ratio of the ER suspensions as a function of time. The sedimentation stability ratios of PANI and PRM composites are close to each other. As the RM content of the composites increased, colloidal stability increased, and

Table II. Composition and particle size values of PAn and PRM composites

	Sample				
	PAn	PRM1	PRM2	PRM3	RM
RM content	—	13	24	31	100
Particle size (μm)	10.36	8.24	8.18	14.42	7.78

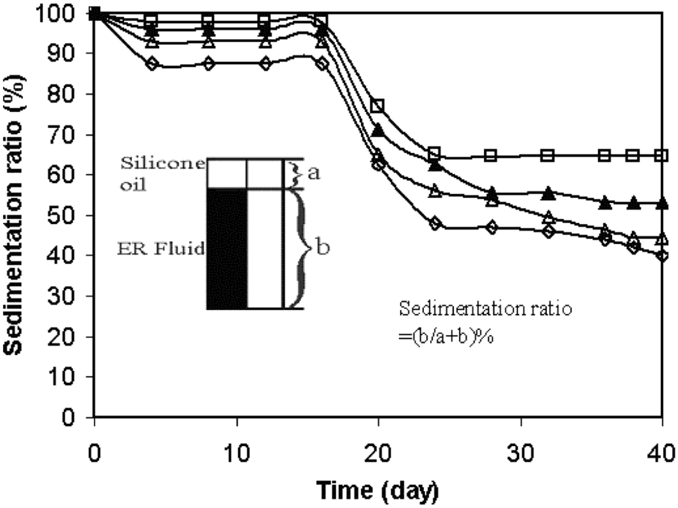

Figure 1. Sedimentation stabilities of suspensions: (◇) PANI, (Δ) PRM1, (▲) PRM2, (□) PRM3, c = 4% m/m, T = 20°C.

maximum colloidal stability was observed from PRM3 (containing 31% RM) suspensions. The highest colloidal stability was observed for PRM3/SO suspension system (c = 30% m/m) with 69% unsettled dispersed particles after 40 days, which is satisfactory for commercial applications.

Electrorheological Properties

Effect of Suspension Concentration on Viscosity and Shear Stress

The change in electric field viscosity with suspension concentration at a constant shear rate ($\dot{\gamma} = 1.0\,\mathrm{s}^{-1}$), electric field strength ($E = 2\,\mathrm{kVmm}^{-1}$), and temperature ($T = 20^{\circ}C$) is depicted in Figure 2. Suspension concentration exerts the principal effect on ER activity. Electric field viscosity of PANI and PRM composite suspension are very close because their conductivities are close. When particle concentration increases, electric field–induced viscosity of the suspension increases. This may be attributed to the polarization forces acting between particles. In dilute suspensions (large distance between dispersed particles), the magnitude of this polarization force in the direction of the applied electric field (E) is[25]

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

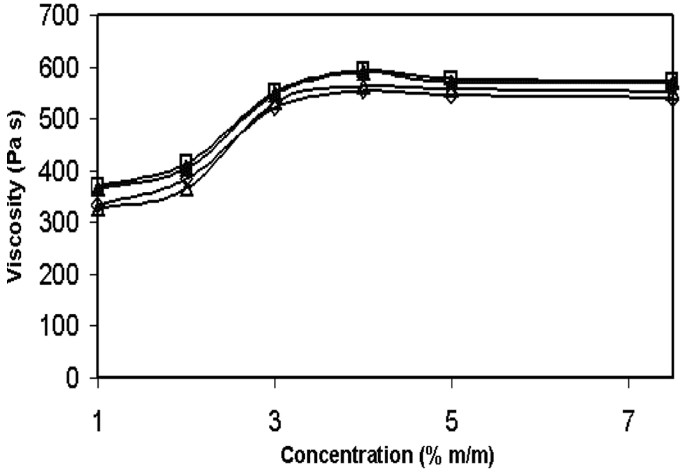

Figure 2. Viscosity changes with concentration: (Δ) PANI, (◇) PRM1, (▲) PRM2, (□) PRM3, $\gamma = 1\,s^{-1}$, E = 2 kV/mm, T = 20°C.

where ε_2 is the dielectric constant of the particle, ρ is the distance between particles, and r is the radius of the particle. An increased suspension concentration will decrease the distance between the particles, which will result in an increased polarization force. We assigned 4% particle concentration as optimum and reached 572 Pas electric field–induced viscosity. In our previous study, we observed 20% optimum concentration for polyisoprene-*block*-poly(styryllithium),[13] Wu and Shen[25] reported an 11% optimum concentration in the ER studies of chitin and chitosan/SO suspensions, and Langelova et al.[8] reported 10% optimum concentration in the ER studies of PANI/SO suspensions.

Dependence of shear stress on particle concentration is given in Figure 3. Shear stress of the suspensions increases linearly with increasing suspension concentration, RM content, and dispersed particle size. This is due to the increased intermolecular attractions of the suspended particles, which cause the association of particles. The highest increment was observed in the PRM3/SO suspension system as $\Delta\tau = 1.64\,kPa$ at E = 2 kV/mm for c = 4% m/m. A similar result was reported by Yoon and Kim for polypyrole-silica-methylcellulose suspensions[16] and by Tian et al.[26] for zeolite/SO suspension.

Effect of Shear Rate on Shear Stress and Viscosity

Change of shear stress of PRM2/SO with shear rate at various electric field strengths (E = 0 kV/mm and E = 0.5–2.5 kV/mm) is shown in Figure 4. In the absence of electric field (E = 0 kV/mm) the PRM2/SO

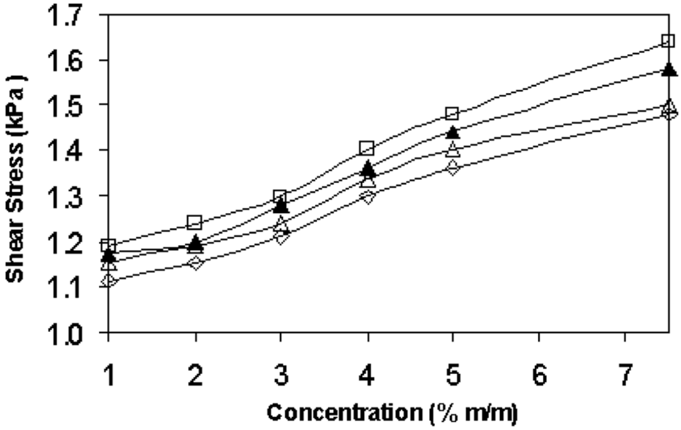

Figure 3. Change of shear stress with concentration: (Δ) PANI, (◇) PRM1, (▲) PRM2, (□) PRM3, $\gamma = 1\,s^{-1}$, E = 2 kV/mm, %, T = 20°C.

suspension shows Newtonian flow behavior, but when various electric field strengths are applied (E = 0.5–2.5 kV/mm), the suspension shows again a shear stress increase with Bingham behavior, which is caused by the induced polarization forces.[27] We also observed Bingham flow behavior for all samples under E = 2.0 kV/mm electric field strength (Figure 5); yield stress values were PRM3 ($\tau_0 = 82$ Pa) > PRM2 ($\tau_0 = 80$ Pa) > PRM1 ($\tau_0 = 76$ Pa) > PANI ($\tau_0 = 72$ Pa). Similar behavior is reported by Fang et al.[28] for PANIi/$BaTiO_3$ suspensions and by Cheng et al.[29] for polypyrrole-mesoporous silica suspensions.

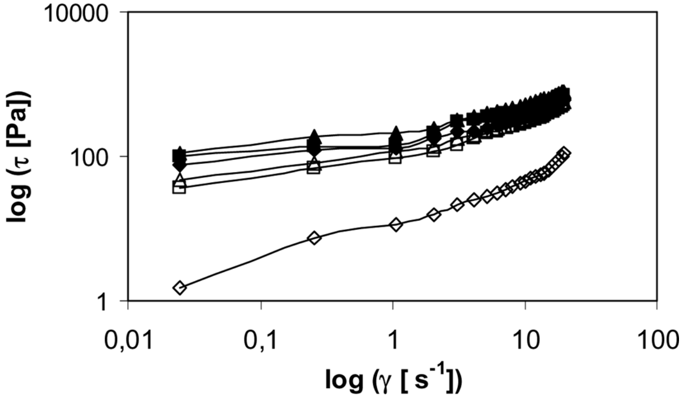

Figure 4. Shear stress vs. shear rate, sample PRM2: (◇) E = 0 kV/mm, (□) E = 0.5 kV/mm, (Δ) E = 1 kV/mm, (◆) E = 1.5 kV/mm, (■) E = 2 kV/mm, (▲) E = 2.5 kV/mm, c = 4% m/m, T = 20°C.

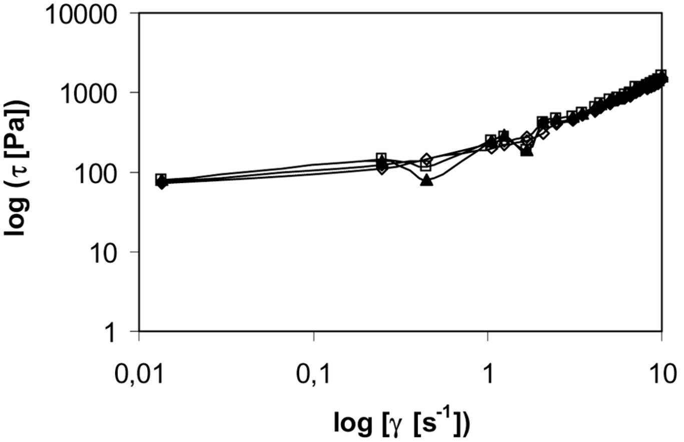

Figure 5. Change of shear stress with shear rate: (Δ) PANI, (◇) PRM1, (▲) PRM2, (□) PRM3, c = 4% m/m, E = 2 kV/mm, %, T = 20°C.

The change of electric field–induced viscosity of all the suspensions with shear rate under similar conditions (c = 4% m/m, E = 2 kV/mm, T = 20°C) is shown in Figure 6. Viscosity of all suspensions sharply decreases with increasing shear rate from 14 to 1.0 Pas, until $\dot{\gamma} = 2\,s^{-1}$, and then becomes shear independent. This is typical shear-thinning non-Newtonian viscoelastic behavior of PANI and PRM/SO suspensions. PANI and PRM particles are affected by the hydrodynamic interactions and the viscous forces (F), which have the following magnitude[14]:

$$F = 6\pi\eta_s r^2\dot{\gamma} \tag{3}$$

where η_s is the viscosity of the suspension and $\dot{\gamma}$ is average shear rate.

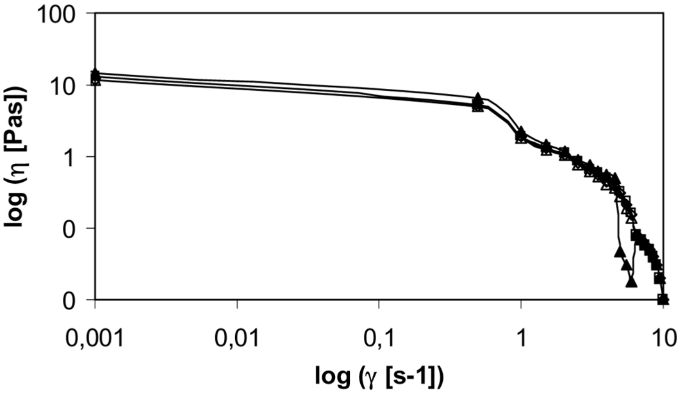

Figure 6. Change of viscosity with shear rate: E = 2 kV/mm, T = 20°C, c = 4% m/m, (Δ) PANI, (◇) PRM1, (▲) PRM2, (□) PRM3.

Although the viscous forces are proportional to the shear rate, at higher shear rates, the electric field–induced structure of suspension is broken and the viscosity almost becomes independent of the shear rate. Similar results were reported by Lengalova et al.[8] in polyaniline/SO suspensions, by Wu et al.[30] for polyaniline derivatives/SO suspensions, and by Yavuz and Unal[31] for polyisoprene-*b*-poly(tert-butylmethacrylate)/SO suspensions.

Effect of Electric Field Strength on Shear Stress and Polarization Forces

Shear stress is one of the critical parameters in the ER phenomenon that has attracted considerable attention both theoretically and experimentally.[32] Figure 7 shows the dependence of shear stress on the square of electric field strength for the four samples studied. Shear stress increases proportionally to the square of electric field strength as expected:

$$\tau_E \alpha \varphi K_f E^2 \beta^2 \tag{4}$$

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 and Chen[33] and Davis.[34] Electric field–induced shear stress values were observed to increase with increasing RM content of the composites and changed in the order: PRM3 ($\tau = 1926\,\text{Pa}$) > PRM2 ($\tau = 1816\,\text{Pa}$) > PRM1 ($\tau = 1592\,\text{Pa}$) > PANI ($\tau = 890\,\text{Pa}$). Lu and Zhao[35] reported that the

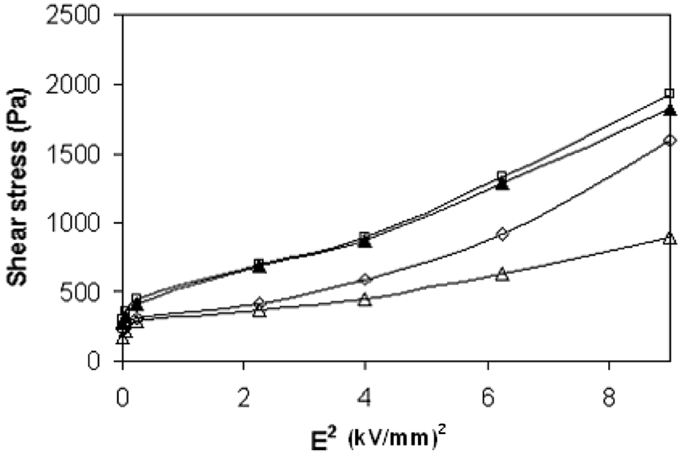

Figure 7. Effect of square electric field strength on shear stress vs. E: (Δ) PANI, (◇) PRM1, (▲) PRM2, (□) PRM3, $\gamma = 1\,\text{s}^{-1}$, c = 4% m/m, T = 20°C.

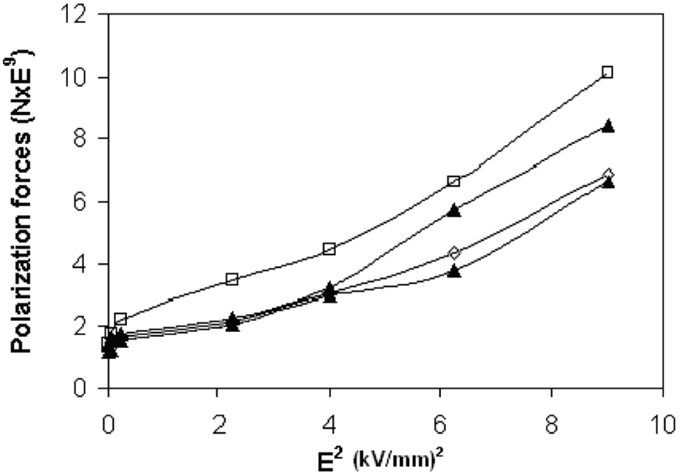

Figure 8. Effect of square electric field strength on polarization forces: (Δ) PANI, (◇) PRM1, (▲) PRM2, (□) PRM3, $\gamma = 1\,s^{-1}$, $c = 4\%$ m/m, $T = 20°C$.

ER effect of PANI increased with increasing montmorillonite content in PANI/montmorillonite composites. In addition, leaking current density in these experiments was limited to $J < 40\,\mu A/cm^2$.

Figure 8 shows the changes in polarization forces (redistribution of the charges of molecules) with the square of externally applied electric field strength at constant conditions ($c = 4\%$ m/m, $\dot{\gamma} = 1.0\,s^{-1}$, $T = 25°C$). It is known that in two-component ER systems (containing dispersed and continues phases) the interfacial polarization is responsible for the ER effect.[4] Electric field–induced polarization forces were observed to increase with increasing electric field strength for all the PRM composite suspensions examined (Equation (2)). This trend is due to the increased magnitude of polarization forces acting between the charged particles. When an electric field was applied to the PANI and PRM composite/SO suspensions, polarization forces caused the aggregation of particles, and a chain formation between the upper and lower plates occurred during the measurements in the rheometer. Similar behavior was reported by Gow and Zukoski for polyaniline/SO suspensions.[36]

Effect of Temperature on Shear Stress

Figure 9 shows the changes in the shear stress of PANI and composite suspensions under various temperatures at constant concentration ($c = 4\%$ m/m) and electric field strength ($E = 2\,kV/mm$). The shear

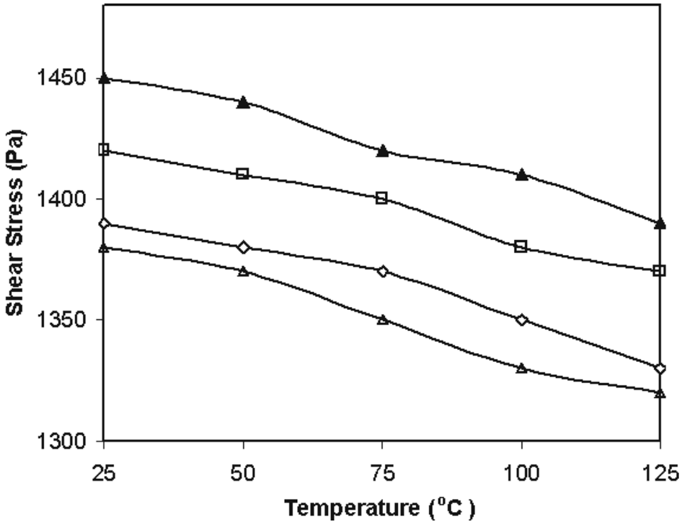

Figure 9. Effect of temperature on shear stress: (Δ) PANI, (◇), PRM1, (▲) PRM2, (□) PRM3, E = 0 kV/mm, c = 4% m/m.

stress of the suspensions was observed to slightly decrease with increasing temperature. There are two reasons the temperature might change the ER effect. The first one is that the temperature can change the polarizability of the ER suspensions. The second one is that the temperature can directly influence particle thermal motion. If the Brownian motion could be intensified at high temperature and could become strong enough to compete with particle fibrillation, the ER effect would become weaker.[2] The shear stress losses ($\Delta\tau = \tau_{125} - \tau_{25}$) between 25° and 125°C are also calculated and found to be $\Delta\tau = 60\,\mathrm{Pa}$ for PRM1, PRM2, and PANI, and 50 Pa for PRM3. Similar shear stress losses were reported by Block and Kelly[2] in silica/SO suspensions and by Yanyu et al.[37] in inorganic/polymer blend/SO suspensions.

The effect of polar promoters on ER strength of suspensions was also studied by adding various promoters (i.e., water, ethanol, glycerol); however, no ER enhancement was observed and, thus, they were classified as dry-based ER fluids.

Effect of Frequency on Shear Stress

The external stress frequency is an important factor for characterizing the dynamic viscoelastic properties of ER fluids. Figure 10 demonstrates the

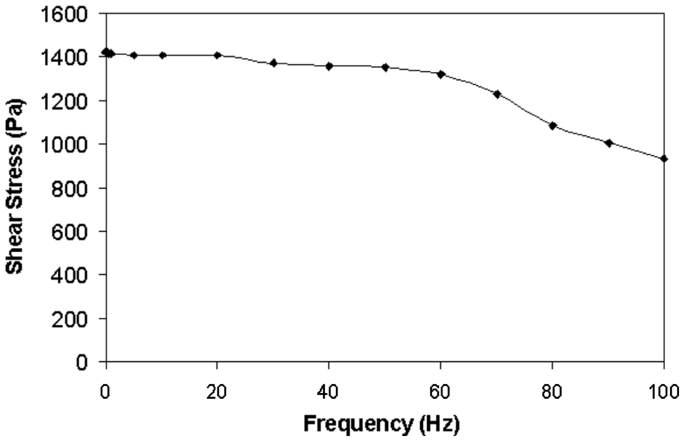

Figure 10. Dependence of shear stress on frequency for PRM2, E = 2 kV/mm, c = 4% m/m, T = 20°C.

behavior of the PRM2/SO sample under an externally applied frequency. The shear stress remains unchanged at linear the viscoelastic region, then it slightly decreases with the further increase in frequency. When the frequency increases from 60 to 100 Hz, shear stress decreases from $\tau = 1320\,\mathrm{Pa}$ to $\tau = 930\,\mathrm{Pa}$. The situation can be compared with dipole relaxation in an electromagnetic field: when the field frequency is too high, the dipoles are no longer able to follow the direction of the electric field. As a consequence, the shear stress losses occur. The same features were reported by many other investigators in the studies of poly(Li-2-hydroxyethyl methacrylate)/SO suspensions by Unal et al.[38] polyaniline/SO suspensions by Hiamtup et al.,[39] and amorphous TiO_2/SO suspensions by Sung et al.[40]

CONCLUSION

Sedimentation stability of suspensions was determined to be 40 days for PRM3, which contains 31% RM. ER activity of all the suspensions was observed to increase with increasing electric field strength, dispersed particle concentration, RM content, increasing particle size and decreasing shear rate, and frequency. The suspensions exhibited Newtonian flow behavior in the absence of an electric field and Bingham flow behavior in the presence of externally applied electric field strength. The electric field–induced viscosities of PANI and PRM composite/SO suspensions decreased sharply with increasing shear rate, thus showing a shear-thinning non-Newtonian flow behavior. The ER strength of all the suspensions was observed to be slightly sensitive to high temperature, and they were classified as dry-based ER fluids.

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