Rheological Properties, Shear-Dependent Electrical Resistance, and Settling Phenomena of Polyaniline in ECO Solution

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ABSTRACT: Polyaniline (PAni) was synthesized by the miniemulsion polymerization and dispersed in a poly(epichlorohydrine-*co*-ethylene oxide) (ECO) solution. The rheological properties using an R/S Rheometer and the shear-dependent electrical resistance using a homemade setup were measured. The addition of PAni particles to the ECO solution causes an increase in the shear stress and shear viscosity and the nature of flow changes from a dilatant to a Newtonian fluid. For both the ECO solution and ECO-PAni mixture, the viscosity at any particular shear rate is higher during the increasing cycle of shear rate compared with the decreasing cycle of shear rate called hysteresis. The ECO-PAni mixture executes a step-like viscosity transition over the shear rate region 50–150 s^{-1}, which is known as shear rate-induced phase transition. The resistance of ECO solution decreases with the increase in shear rate and reversibly increases with the decrease in shear rate, whereas the resistance of ECO-PAni mixture increases during both the increasing and decreasing cycles of the shear rate. The difference in resistance at zero shear rate before and after the end of shear cycle is termed as electrical set. The ECO solution and ECO-PAni mixture exhibit 4% and 79% electrical set in terms of decrease and increase of resistance, respectively. The settling process of PAni in the ECO-PAni mixture is slower compared with the pure ECO in solution. The settling time of PAni in the ECO-PAni mixture increases with the decrease in shear rate. © 2009 Wiley Periodicals, Inc. J Appl Polym Sci 114: 238–245, 2009

Key words: conducting; polymers; rheology; viscosity

INTRODUCTION

The widespread venture for finding different practical applications of intrinsically conducting polymers (ICPs) has discovered their use in the form of dispersion or solution as anticorrosive coatings.[1–5] Among the ICPs, extensive study of the polyaniline (PAni) and its composites has been carried out because of their ease of preparation, novel properties, and potential applications.[6–13] For the present investigation, a coating solution was prepared containing the PAni particles as an ICP and the poly (epichlorohydrine-*co*-ethylene oxide) (ECO) as a dispersion stabilizer. The PAni was chosen as an ICP because of its novel properties (especially thermal stability) as mentioned earlier, and the ECO was chosen as a dispersion stabilizer because of its high polarity; hence, it was expected to have better compatibility with PAni and its resistance against oxidation due to the absence of double bond at the backbone. Some interesting studies investigating the rheological and electro-rheological behavior of PAni

in different medium are available in the literature.[14–22] Kim et al.[14] studied the electro-rheological behavior of PAni-Na$^+$-montmorillonite nanocomposite particles in silicon oil. Gangopadhyay[15] used poly(vinyl pyrrolidone) (PVP) as dispersion stabilizer for PAni in aqueous medium and studied the rheological property of this dispersion. Lee et al.[16] and Kim et al.[18] investigated the effect of surfactant on the colloidal stability of PAni in a mineral oil. They found that the colloidal stability and hence electro-rheological property of PAni particles in that mineral oil depends on the threshold surfactant concentration, which again depends on the type of surfactant. The electro-rheological property of PAni in silicon oil was studied by Hiamtup et al.,[17] Kim et al.,[18,19] and Choi et al.[20] Fang et al.[21] prepared PAni/BATiO$_3$ composite in order to obtain superior electro-rheological properties. Cho et al.[22] synthesized spherical, monodispersed polymer microspheres consisting of a poly(methyl methacrylate) core and a PAni shell and used it as the dispersed material for electro-rheological fluids. The knowledge of rheological behavior of Pani-like ICP in the form of solution, emulsion, or suspension in polymeric liquids is of great importance for a number of industrial processes.

The present study deals with the measurement of rheological properties as well as shear-dependent

electrical resistance of ECO solution and ECO-PAni mixture after fabricating and interfacing a suitable array of systems. The PAni particles have a tendency to sediment, as they are sparingly soluble in most of the available solvents. Apart from the chemical nature of both the medium and dispersed particles, the sedimentation process also depends on the density of the medium as well as the size and density of the PAni particles.[19,20] When shear force is applied to a polymer solution/dispersion and then stopped, along with the particles, soluble polymer chains and dipoles also rearrange and then settle after a certain period of time. Therefore, settling kinetics is also an important area of study. However, the literature dealing with the settling phenomena of PAni solution or dispersion is scanty. The present study also deals with the estimation of the rate of settling of PAni from the ECO-PAni mixture after shearing the solution at different shear rates and then stopping the shearing and recording the time required to stabilize the resistance.

EXPERIMENTAL

Materials

ECO ($T_g = -30°C$, Mooney viscosity ML_{1+4} at 100°C = 90–102) is from Aldrich (Germany). Aniline, 1-methyl 2-pyrrolidinone (NMP), ethyl methyl ketone (MEK), ammonium peroxydisulfate (APS), stannous chloride are from Merck (Mumbai, India). Hydrochloric acid (HCl) is from Ranbaxy. Sodium dodecyl sulfate (SDS) and cetyl alcohol are from Loba Chemie (Mumbai, India). Methanol is from SISCO Research Laboratories (Bombay, India). All of the chemicals are GR grade. Aniline was distilled before use. All other chemicals were used as received.

Synthesis of polyaniline

Polymerization of aniline was carried out following our previous method.[6] 1 mM SDS, 4 mM cetyl alcohol, and 100 mL deionized (DI) water were taken in a beaker and stirred for 1 h with a high-speed mechanical stirrer to form the miniemulsion. To this miniemulsion, 0.1M aniline and 0.05M HCl were added and stirred, and then the polymerization reaction was carried out through the dropwise addition of APS solution (0.1M APS in 100 mL DI water). The polymerization reaction was carried out for 6 h at 0–5°C with constant stirring. After that, the polymerization reaction was stopped through the addition of 50 mL methanol. Finally, the reaction mixture was treated with 0.01M SnCl$_2$ and 0.05M HCl in order to reaper the defect structure and to improve the conductivity of PAni.[6] The PAni particles thus obtained were filtered, washed several time with DI water,
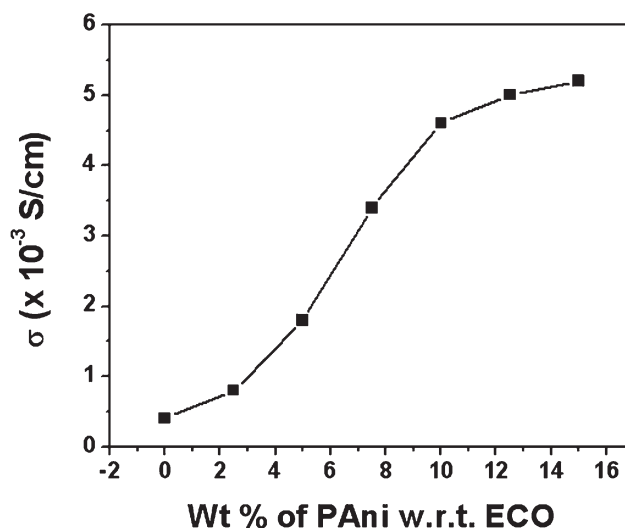

Figure 1 Effect of DC electrical conductivity of solutions on the weight percentage of PAni content.

and finally dried in a vacuum oven at room temperature.

Preparation of ECO solution and ECO-PAni mixture

ECO (5 wt %) solution was prepared by using a mixed solvent of 4 : 1 MEK and NMP. A different weight percentage of PAni with respect to ECO was added to the above prepared solution, and the whole mass was thoroughly mixed continuously for 6 h using an electric stirrer. The solid content of each solution was measured, and extra solvent was added to each solution in order to maintain exact 5 wt % solid content. The DC electrical resistance of all solutions was measured by using a Systronics conductivity meter 304 (Ahmedabad, India). It was found that, with the increase in PAni concentration, the electrical conductivity of the solution initially increased rapidly then slowly beyond 10 wt % of PAni concentration (Fig. 1). It was also observed that, for the mixture containing PAni concentration more than 10 wt %, a few PAni particles started to sediment. Therefore, for the final experiments, a solution was prepared containing 10 wt % of PAni with respect to ECO with the total solid content of 5 wt %. This solution was designated as "ECO-PAni." A neat ECO solution was also prepared for comparison using the same method containing 5 wt % of pure ECO (without PAni) in same solvent mixture and was designated as "ECO."

Measurement of electrical conductivity of polyaniline powder

The PAni powder was compacted into a disk pellet with a diameter of 12.6 mm and thickness of about

1.0 mm under a pressure of 10 tons. The DC electrical conductivity of this solid pellet sample was measured by a two-point probe method using a DC milli-Ohm meter (GOM 802; GW Instek, Taipei, Taiwan).

Determination of particle size of polyaniline

The particle size and morphological analysis of polyaniline powder were carried out using a scanning electron microscope (SEM) (JSM 5800; JEOL, Tokyo, Japan). All samples were coated with gold prior to test.

Measurement of rheological properties and shear-dependent electrical resistance

The rheological properties of the ECO solution and ECO-PAni mixture were measured by using an R/S Rheometer (R/S CC, version 2.7; Brookfield Viscometer, Essex, England) with a coaxial spindle (CC25). A coaxial Teflon cylinder was fabricated with two parallel copper electrodes (2 cm × 3 cm) fitted on the wall of the cylinder. These two electrodes were connected to a DC milli-Ohm meter (GOM 802; GW Instek). The milli-Ohm meter was again interfaced with another computer in order to measure the electrical resistance at different shear rate. The shear rate of the rheometer was programmed to increase from 0 to 1000 s^{-1} and decrease from 1000 to 0 s^{-1} at a rate of 50 s^{-1} per 60 s, and the shear stress and shear viscosity corresponding to each shear rate were obtained from the experiment. The rheological properties and electrical resistance data were collected simultaneously in two different computers.

In order to study the settling phenomena, the ECO solution and ECO-PAni mixture were subjected to shearing for 300 s at different shear rates followed by sudden stoppage of shearing, and polymers were allowed to settle for the next 1000 s. The whole process was monitored through the continuous measurement of resistance of the solutions until the attainment of equilibrium, which generally occurred within 900 s. Hence, the experiment was carried out for 1000 s in each case.

RESULTS AND DISCUSSION

Electrical conductivity and particle size of polyaniline

The electrical conductivity of the resultant PAni is 1.3×10^{-2} S/cm. Depending on the oxidation state and protonation level, the PAni can either be highly conducting or insulating in nature. The doped half-oxidized and half-reduced emeraldine base (EB) form is significantly conducting, whereas the fully
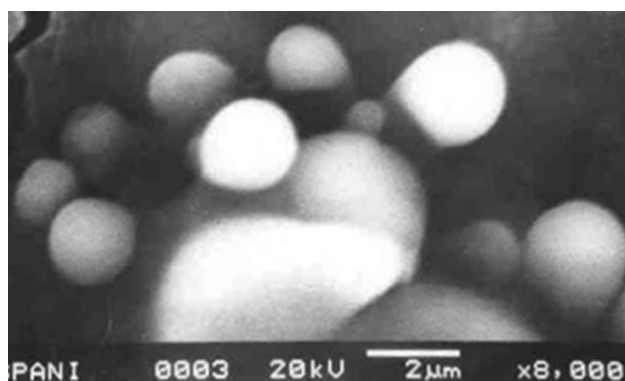

Figure 2 SEM image of polyaniline.

reduced leucoemeraldine base (LEB) and the fully oxidized pernigraniline base (PNB) are mainly insulating in nature.[6] The synthesized PAni is conducting in nature, which indicates that this PAni contains a high concentration of EB form.

The dispersion stability of PAni in a medium also depends on its shape and size. The shape and size of PAni particles were observed in a SEM image. Figure 2 presents the SEM image of PAni. The PAni particles are spherical in nature with an average diameter of 1–5 μm.

Rheological properties and shear-dependant electrical resistance of ECO solution and ECO-PAni mixture

For the ECO solution, the shear stress and shear viscosity values at very low shear rate were too small to be detected by the instrument. However, with the increase in shear rate, both the shear stress and shear viscosity increased and were detectable by the instrument when the shear rate was ≥650 s^{-1}. Therefore, rheological properties of ECO solution were measured over the shear rate region 650–1000 s^{-1}. However, with the addition of PAni to ECO solution, there was appreciable increase in both the shear stress and shear viscosity, and the changes in both properties over the whole shear rate region could be detected by the instrument.

Figure 3(a,b) represents the shear stress (τ) as a function of shear rate (γ) for the ECO solution and ECO-PAni mixture, respectively. The shear stress increases exponentially for the ECO solution, and it increases almost like a straight line for the ECO-PAni mixture. The shear stress plots show that the ECO solution behaves like a dilatant and the ECO-PAni mixture as a Newtonian fluid.[23] The value of shear stress at different shear rate is much higher for the ECO-PAni mixture compared with that of the ECO solution. During the increasing and decreasing cycle of shear rate, the changes in shear stress for both of the systems are almost identical, which
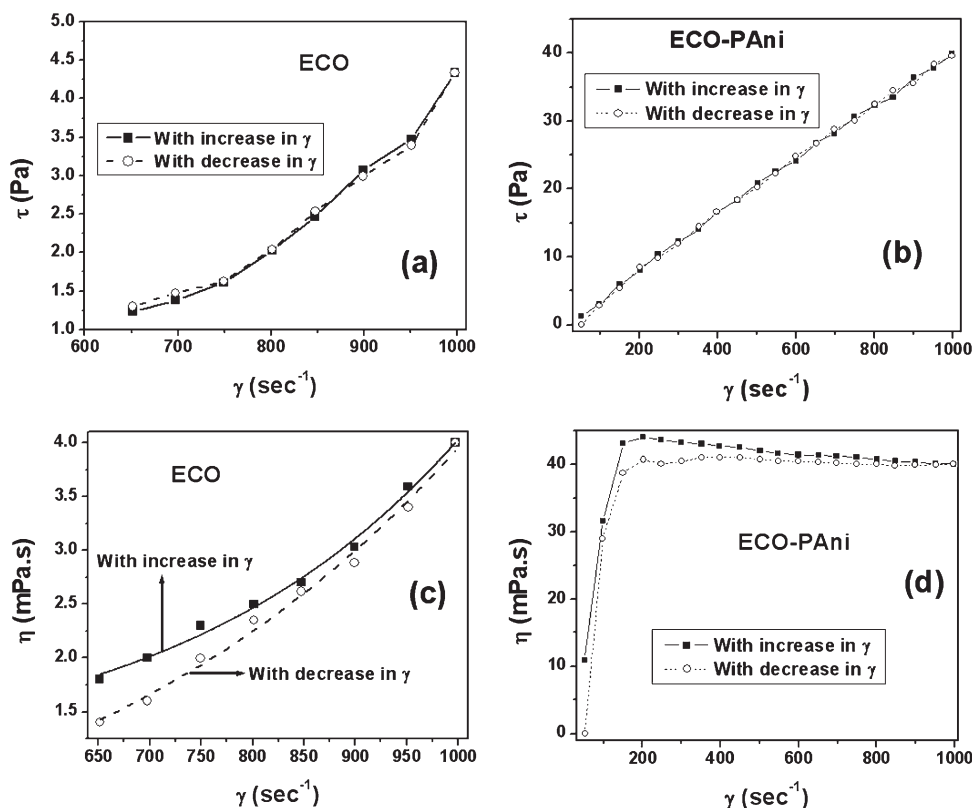
Figure 3 Shear stress as a function of shear rate for (a) ECO solution and (b) ECO-PAni mixture and shear viscosity as a function of shear rate for (c) ECO solution and (d) ECO-PAni mixture.

means the ascending and descending plots are almost coinciding with each other.

Figure 3(c,d) represents the shear viscosity (η) as a function of shear rate (γ) for the ECO solution and ECO-PAni mixture, respectively. The viscosity of the ECO solution increases exponentially with the shear rate, which means the nature of this solution is like a shear-thickening or dilatant.[23] Although most of the polymer solutions are shear-thinning or pseudo plastic in nature,[15,20,24] some polymer dispersion exhibits shear-thickening or dilatant behavior.[25] The viscosity of ECO-PAni mixture increases steadily over the shear rate 50–150 s^{-1} and then it becomes almost plateau at a higher shear rate region. The viscosity of ECO-PAni mixture suddenly jumps up during the increase in shear rate and jumps down during the decrease in shear rate over the shear rate region 50–150 s^{-1}. This reversible step like viscosity transition at a critical shear rate region is known as shear rate-induced phase transition.[25] The flow behavior of ECO-PAni is initially complex and then (after 200 s^{-1}) like a Newtonian fluid.[23] When the shear rate of the system is increased to the maximum and then decreased to the minimum, the change of viscosity follows the similar trend with decreased magnitude during decreasing shear rate. There is a difference in the magnitude of viscosity at

the minimum shear rate before and after the beginning of the shear cycle [Fig. 3(c,d)]. This may be due to the easier motion of the polymer chains or relaxation after initial shearing. This phenomenon may be considered as some kind of shear-dependent hysteresis of viscosity.[25,26] Both the shear stress and shear viscosity plots indicate that the addition of PAni in the ECO solution causes appreciable (almost 10-fold) increase in both the shear stress and shear viscosity, and the flow behavior is also changed from the dilatant to a Newtonian fluid.

Arrangement of the PAni particles in ECO solution is expected to affect the electrical conductivity of the total mixture. When such mixture is subjected to a motion at different shear rate, the resistivity of the mixture is expected to change against shear rate depending on the arrangement of PAni particles under motion. Figure 4(a) represents the shear-dependent electrical resistance (R) as a function of shear rate (γ) for the ECO solution. For the ECO solution, when the shear rate is increased, resistance decreases exponentially; and again, when the share rate is decreased, resistance increases exponentially and follows the same route, but the gain in resistance against decrease in shear rate is rather slow and initial resistance at the zero shear rate is not attained. That means there is a difference in the
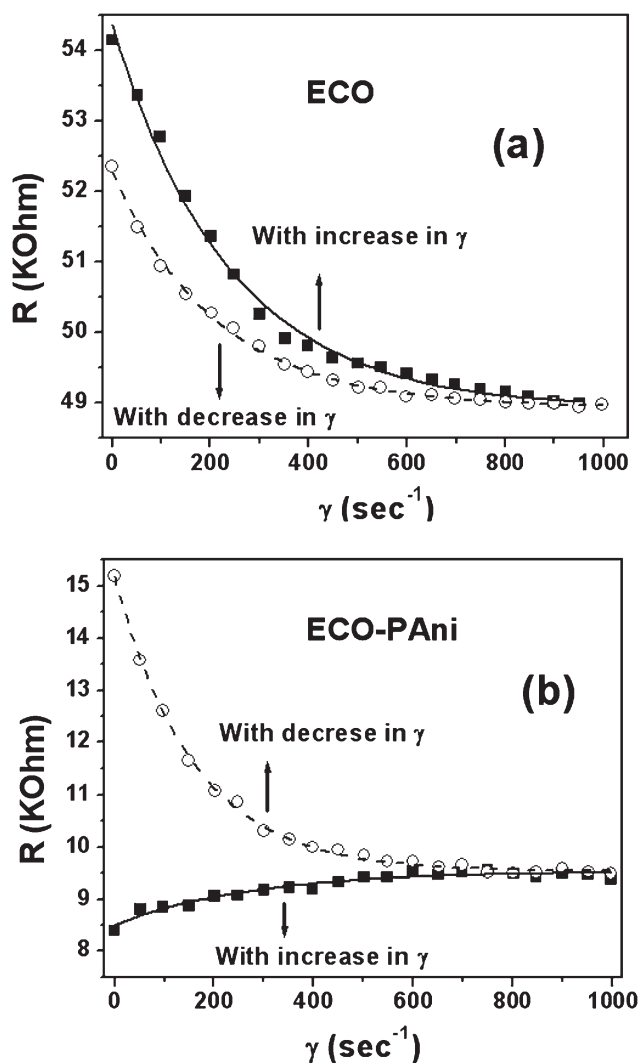
Figure 4 Electrical resistance as a function of shear rate for (a) ECO solution and (b) ECO-PAni mixture.

trical set. During the decrease of shear rate, the process of disorientation of polymer chains (i.e. linear conformation to nonlinear conformation) is less effective mainly due to inertia of motion. Hence, at any particular shear rate, the chains are more oriented during the decrease of shear rate compared with that of during the increase in shear rate. Consequently, a lower resistance is encountered during the decrease of shear rate. At the stationary condition, i.e. at zero shear rate, the chain conformations before the start and after the end of shear cycle are different. After the end of shear cycle, the chains are relatively more oriented, and consequently electrical resistance is expected to be lower.

For the ECO-PAni mixture, with the increase in shear rate, the electrical resistance is increased slowly; and again with the decrease in shear rate, the electrical resistance is increased rapidly as shown in Figure 4(b). For the ECO-PAni mixture, the resistance/conductance mainly depends on the arrangement of suspended PAni particles and soluble PAni chains. For the ECO-PAni mixture, the conductance is due to the mobility of charge carrier from one PAni particle or PAni chain to other. If the average gap between the PAni particles or PAni chains is less (<10 nm), the charge carrier can easily hop the gap and exhibit high electrical conductivity. However, due to any kind of turbulence, if these gaps increase, then the discontinuity in the conductive network is formed leading to the increase in resistance. During the increase in shear rate, there is initially a small increase in resistance due to the disruption of conductive network formed by PAni particles, as shown in Figure 5(b). However, the effect becomes marginal with further increase in the shear rate, and the change in resistance with increase in
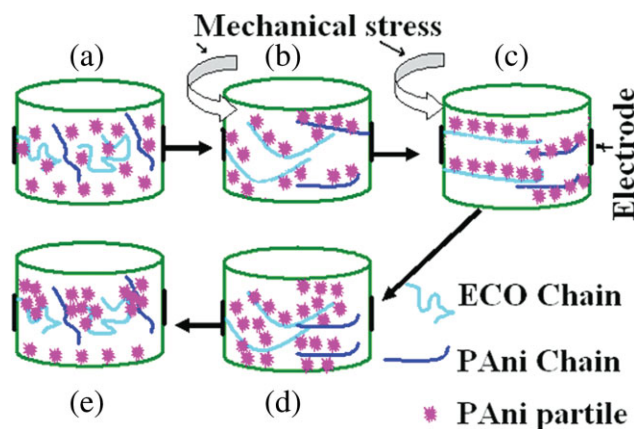
resistance at the beginning and after the end of the shear cycle. This phenomenon may be termed as hysteresis and electrical set. The reason for this behavior may be explained as follows: at rest, the ECO chains in solvent are more coiled. With the increase in shear rate, polymer chains assume more and more oriented along the direction of the motion and attain a relatively linear conformation, whereas dipoles present in the same polymer backbone and can orient themselves along the electric field that is applied perpendicular to the motion. Thus, there is some dipole–dipole bridging leading to the increase in conductance (i.e. decrease in resistance). However, when the shear rate is decreased, polymer chains are reluctant to disorient completely and attain the same conformation that was attained at the same shear rate during the increase of shear rate. As a result, there is always a resistance gap at the minimum shear rate, leading to hysteresis and elec-

Figure 5 Different conformation of ECO chain, PAni chain, and PAni particle occurs during the increase and decrease of shear rate. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

Process	Minimum electrical resistance (R_0) kΩ	Amplitude (A)	Decay constant (k)	R at zero shear rate (kΩ)	Change of R (%) at zero shear rate after shear cycle (electrical set)
Increase in shear rate of ECO solution	48.9 ± 0.1	5.4 ± 0.1	237 ± 12	54.3 ± 0.2	
Decrease in shear rate of ECO solution	48.9 ± 0.1	3.3 ± 0.04	211 ± 6	52.2 ± 0.14	4% decrease of R
Increase in shear rate of ECO-PAni mixture	9.52 ± 0.02	−1.05 ± 0.05	260 ± 37	8.47 ± 0.07	
Decrease in shear rate of ECO-PAni mixture	9.52 ± 0.02	5.64 ± 0.05	160 ± 3	15.16 ± 0.07	79% increase of R

shear rate is also marginal. This may be due to the formation of stable streamline conducting network during shearing at higher shear rate as shown by Figure 5(c). However, from the highest experimental shear rate when the shear rate is progressively decreased, there is an increase in disturbance in the stream line motion of PAni particles. This leads to the disruption of continuous conductive network [Fig. 5(d)] and aggregation and sedimentation of some PAni particles at zero shear rate [Fig. 5(e)]. The average gap between the adjacent conducting particles continuously increases with decrease in the shear rate, leading to the increase in resistance of the ECO-PAni mixture. The phenomena of hysteresis and electrical set is much more pronounced for the ECO-PAni mixture compared with ECO solution because of presence of polar and conducting PAni particles. The electrical resistance value of ECO-PAni mixture is much less than that of ECO solution at any shear rate for the same reason.

The exponential loss and gain of resistance during the increase and decrease of shear rate for the ECO solution and ECO-PAni mixture is mathematically expressed by the equation ExpDec1:

$$R = R_0 + Ae^{-\gamma/k} \tag{1}$$

Here, R is the electrical resistance in kΩ at any shear rate, R_0 is the minimum resistance, A is the amplitude, γ is the shear rate in s^{-1}, and k is the decay constant.

The amplitude and the decay constant are presented in Table I. When the shear rate is decreased, the k shows lower value compared with the values obtained with increase in shear rate. This fact indicates that the gaining of the resistance during the decrease of shear rate is slower compared with losing the same during the increase of shear rate. The R at zero shear rate is the sum of R_0 and A. From the R at zero shear rate, the change or electrical set in terms of resistance was calculated and presented in Table I. The ECO solution exhibits −4%, whereas the ECO-PAni mixture exhibits +79% electrical set in terms of decrease or increase of resistance (R).

Determination of settling kinetics of polymers after shearing at different shear rate

After shearing a polymer solution once it is stopped, the particles, soluble polymer chains, and dipoles are rearranged and then settle after a certain period of time. The internal reorientation will be reflected in their conductance. The conductance may increase or decrease, but once the settling of the polymer is complete, the conductance will remain almost constant. Therefore, the change of conductance with time gives an idea about the settling phenomena of polymers in solvent. Figure 6(a,b) represents the conductance as a function of time for the ECO solution and ECO-PAni mixture, respectively. Both the ECO solution and ECO-PAni mixtures were sheared at different shear rate for 300 s and then stopped, but the electrical resistance was measured up to 1000 s (one reading each second) and conductance was calculated. For the ECO solution during the shearing with a particular shear rate, the conductance changes in a zigzag pattern. Under the shearing condition, there may be continuous change in dipoles, and hence there may be continuous formation and destruction of conducting network. When the shearing is stopped, the conductance suddenly drops straightway and then remains constant with time. This sudden drop of conductance may be due to the complete destruction of the conducting network; because when the shearing is stopped, there is a chaotic motion experienced by the solvent and polymer molecules and they are totally disoriented and settle quickly from their ordered streamline motion. At the end, the constant conductance with time indicates that the molecules are now free from any motion and totally settled with the present orientation. For the ECO-PAni mixture, the nature of change of conductance is different. The initial zigzag nature of conductance during shearing is increased with the increase of shear rate. When the shearing is stopped, the conductance decays exponentially instead of straightway fall, and it becomes constant after a longer time compared with the ECO solution. In the ECO-PAni mixture, some PAni chains are

dissolved in solvent, some PAni particles are entrapped in ECO, and some PAni particles remain suspended or dispersed. All three types of PAni take different time to settle, and hence conductance is expected to decay slowly. The width of the initial zigzag pattern of conductance is increased with the increase of shear rate. This is obvious because PAni is conducting, and hence formation and destruction of conducting network with increase of shear rate is more pronounced and reflected in their conductance value. At a higher shear rate, the polymer chains undergo more stretching leading to better bridging of chains and/or dipoles and/or particles. This is the reason for the higher conductance at higher shear rate for both systems. In order to understand the nature of settling of PAni in the ECO-PAni mixture, the decay constant of the conductance was

TABLE II
Rate Constant (k) of Settling Phenomena of PAni in the ECO-PAni Mixtures, After Shearing at Different Shear Rate, Obtained from Curve Fitting with Exponential ExpDec1 Equation Using Origin 6.1 and Regression Analysis

γ (s^{-1})	k of ECO-PAni mixture
10	15 ± 1.5
50	28 ± 2.0
100	40 ± 1.5
300	44 ± 1.0
500	48 ± 0.7
700	54 ± 1.4
900	60 ± 1.3

determined after fitting all the curves with exponential ExpDec1 equation using Origin 6.1:

$$C = C_0 + Ae^{-t/k} \qquad (2)$$

Here, C is the conductance in Siemens at any time, C_0 is the minimum conductance, A is the amplitude, t is the time in second, and k is decay constant.

The decay constant (k) and the settling time of PAni in the ECO-PAni mixtures after shearing at different shear rate obtained from curve fitting and regression analysis are shown in Table II. With the increase in shear rate, the continuous increase of decay constant (k) value indicates the faster decay of conductance that means decrease of settling time of PAni with increased shear rate. Figure 6(b) shows that, during shearing condition, the conductance value at higher shear rate is higher. That means, during the shearing at higher shear rate, PAni undergoes more streamline, regular orientation and forms a better conducting path compared with shearing at lower shear rate. Hence, when the shearing is stopped, it takes less time to settle.
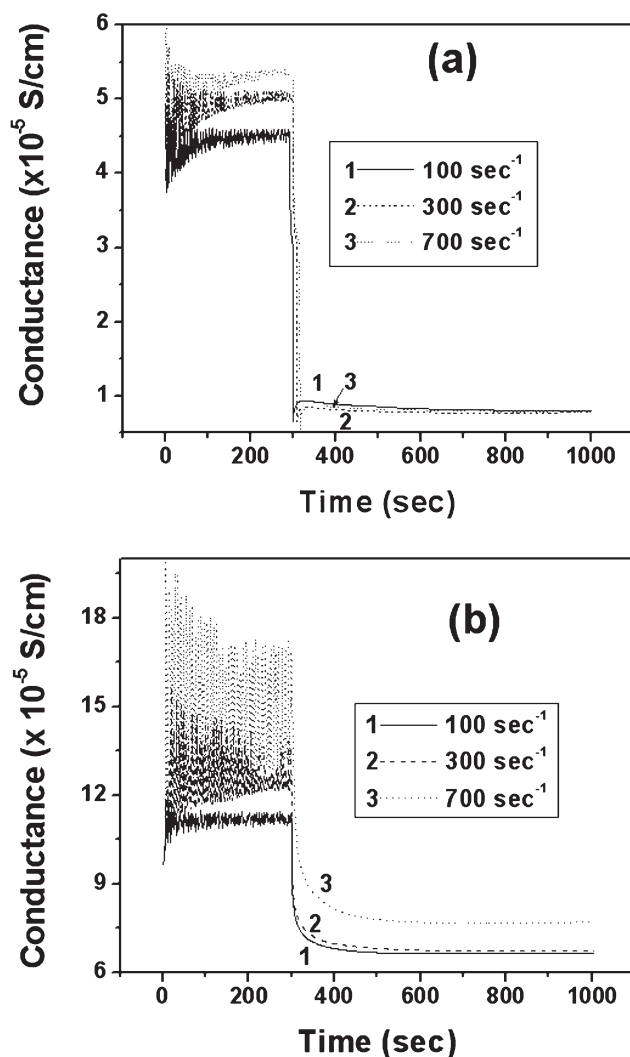

Figure 6 Conductance as a function of time for (a) ECO solution and (b) ECO-PAni mixture. The solutions were sheared at different shear rate for 300 s and then stopped, but electrical resistance was measured up to 1000 s and conductance was calculated.

CONCLUSIONS

The addition of PAni particles to the ECO solution causes an increase in the shear stress and shear viscosity of the solution and the nature of flow changes from a dilatant to a Newtonian fluid. The shear stress and shear viscosity of ECO solution and ECO-PAni mixture follow the similar trend during both the increasing and decreasing cycle of shear. However, the viscosity at any particular shear rate is higher during the increasing cycle of shear rate compared with the decreasing cycle of shear rate due to the easier motion of the polymer chains or relaxation after initial shearing, called hysteresis. The viscosity of ECO-PAni mixture suddenly jumps up during the increase in shear rate and jumps down during the decrease in shear rate over the shear rate region 50–150 s^{-1}. This reversible step, like viscosity transition

at a critical shear rate region, is called shear rate-induced phase transition. The resistance of ECO solution decreases with the increase in shear rate and reversibly increases with the decrease in shear rate, whereas the resistance of ECO-PAni mixture increases during both the increasing and decreasing cycle of the shear rate. This phenomenon is termed as hysteresis and electrical set in terms of electrical resistance. The ECO solution exhibits 4% decrease in electrical resistance, whereas the ECO-PAni mixture exhibits 79% increase in electrical resistance after the completion of the shear cycle. The ECO polymer chains settle very quickly in solution. However, the settling of PAni in the ECO-PAni mixture is rather slow. The settling time of PAni in the ECO-PAni mixture increases with the decrease in shear rate.

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