

## Research Article

# Rheological Properties and Reverse Micelles Conditions of PEO-PPO-PEO Pluronic F68: Effects of Temperature and Solvent Mixtures

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The rheological properties of Pluronic F68 were dissolved in various water/organic liquid mixtures over a wide range of temperatures, all at a concentration of 20 mg/mL. We have considered the following binary mixtures: Pluronic F68/water, F68/p-xylene, and F68/phenol. Various conformational transitions were detected and interpreted. We have also shown that these mixtures retain a Newtonian behavior independently of temperature and conformational changes. For ternary F68/p-xylene/water, F68/phenol/water, and F68/water/phenol mixtures, the behaviour of the solution is intimately related to the temperature and the amount of water and organic solvent added.

## 1. Introduction

The triblocks copolymers based on poly(ethylene oxide)-poly(propylene oxide)-poly(ethylene oxide), usually named Pluronics (manufactured by BASF) or Poloxamers (manufactured by ICI), are able to form direct micelles with PEO coronas and PPO cores or reverse micelles with PEO cores and PPO coronas under some conditions. The investigation of their association properties has had considerable ambiguity due to the fact that some of them are able to form direct micelles, reverses micelles, and various arrangements under several conditions, whereas some of them showed that the dimension of the structures formed was equal to the length of the hydrophobic stretched blocks [1–9]. In this regard, the critical micelle concentration, the critical micelle temperature, the aggregation number, and the polydispersity are usually given with a certain uncertainty [3]. These triblocks copolymers constitute an interesting class of surfactants which have attracted considerable attention due to their possible use in many specialized applications, for example, in the pharmaceutical industries [10] and bioprocessing [11].

In the last few years several experimental and theoretical works have been directed at the investigation of binary Pluronic/organic solvent and ternary Pluronic/water/organic solvents due to the profound changes observed in the solution properties and their wide domain of application [12–15]. Three topics characterizing these systems, namely, (i) the effect of the copolymer architecture on the association behavior [16, 17], (ii) the anomalous micellization and composition [18, 19], and (iii) the reverse micelles caused by the water presence [20], are the subject of numerous studies. For example, Ghaouar et al. [1] used dynamic light scattering and viscosity measurements for the Pluronics L64 and F68 dissolved in aqueous and organic solvent for various concentrations. To investigate their conformational changes they proved, on the one hand, the nonexistence of a critical micelle concentration for various concentrations of both L64 and F68 immersed in pure-p-xylene and, on the other hand, that the concentration regimes do not depend on the nature of the solvents [1].

The aim of this study was an investigation of the rheological properties of binary Pluronic/organic solvent and ternary Pluronic/organic solvent/water system. Hence, we focused on

the various transitions and conformational changes and their relations with the change of solution behavior as a function of temperature.

## 2. Materials and Methods

The PEO-PPO-PEO block copolymer, Pluronic F68, was donated by BASF Corp and was used without further purification. The copolymer has a nominal molecular weight of 8400 and is represented by the formula  $(\text{PEO})_{78}(\text{PPO})_{30}(\text{PEO})_{78}$ . The deionized water used was treated with a Millipore-Q water purification system. The p-xylene and phenol were obtained from Sigma-Aldrich, USA.

Rheological measurements in shear were performed using a Brookfield DV-II + Pro Viscometer cone-plate geometry (USA). This apparatus is equipped with a motor which applies a deformation to the sample and a sensor to measure the stress induced in the sample versus the shear rate. For polymer solutions of low viscosity, the most appropriate spindle is CPE 40 (radius 2.5 cm, angle  $0.8^\circ$ ) that measures the viscosity over the range of 0.15 to 3.065 cP. The temperature was controlled by a thermostatic controlled water bath, type TC-502D-230 (Brookfield Engineering Laboratories Inc., USA), with an accuracy of  $0.1^\circ\text{C}$ . The system was connected with a computer permitting the control of parameters via the software Rheocalc (V2, 4) (Brookfield Engineering Laboratories Inc., USA). The calibration was verified using three standard solutions of known viscosity. We note that for the various concentrations and temperatures used for this work, the data showed that all solutions had a Newtonian behavior.

## 3. Results and Discussion

### 3.1. Binary Mixtures of Pluronic F68/Solvent

**3.1.1. Binary Pluronic F68/Water.** The rheological properties of Pluronic F68 dissolved in water were investigated for various temperatures. Figure 1 illustrates the variation of the dynamic viscosity versus the shear rates for various temperatures; they varied according to the law

$$\tau = \eta\dot{\gamma}, \quad (1)$$

where  $\tau$ ,  $\eta$ , and  $\dot{\gamma}$  represent, respectively, the shear stress, the dynamic viscosity, and the shear rate. We observe that for all tested temperatures, the dynamic viscosity remained constant indicating that the solutions had a Newtonian behavior. Figure 2 shows the variation of the relative viscosity versus temperature for the binary Pluronic F68/water mixtures. We show that the relative viscosity varied following two phases. In the first, where the temperature was varied from 15 to  $30^\circ\text{C}$ , the viscosity decreased slowly with increasing temperatures. We consider that during this phase the PEO-PPO-PEO blocks remained as expanded individual random coils. With increasing temperature, an enhancement of the hydrophobic interactions took place.

The PPO blocks possibly twist and flex to minimize the contact with the solvent ensuring the attractive interactions

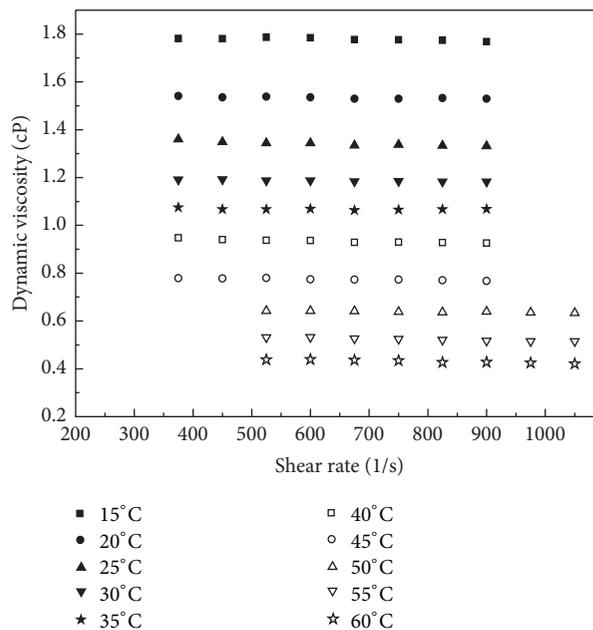


FIGURE 1: Variation of the dynamic viscosity for a solution of concentration of 20 mg/mL of Pluronic F68 in water versus shear rates for different temperatures.

between hydrophobic blocks. We assume that this transition leads to the formation of spherical micelles with hydrophobic PPO core and hydrophilic PEO water-swollen corona.

At the end of this first phase, the hydrodynamic volume of micelles decreased due to the enhancement of the hydrophobic interactions and to the change of PEO chains properties; the micelles compaction was accomplished [21–24]. The second phase, for temperatures between 30 and  $60^\circ\text{C}$  in which the relative viscosity of the solution increases, starts when the micelle formation is accomplished. With increasing temperatures, a modification of the hydrophobic character of the Pluronic F68 which occurs by the disruption of the hydrogen bonds between PEO blocks and the surrounding solvent is developed. This leads to a strengthening of the intermolecular interactions ensuring a variation of the hydrodynamic volume of the micelles due to an overlapping process between the coronae of neighboring micelles. The micelles become structured and organized to form, probably, after successive conformational transitions, lyotropic liquid crystalline phase described as follows: (i) individual micelles to body-centered cubic phase, (ii) body-centered cubic to hexagonal phase, and (iii) hexagonal phase to lamellar phase [25–31]. We consider that the successive transitions are characterized by a higher viscosity with the aspect of gels, even in the dilute regime, where the temperature can play an important role. Consequently, we deduce that the Newtonian behavior observed for binary Pluronic F68/water was not affected by the various transitions and conformational changes.

**3.1.2. Binary Pluronic F68/p-Xylene.** Figure 3 shows the variation of the dynamic viscosity versus shear rate (with no time dependency) for a concentration of 20 mg/mL of

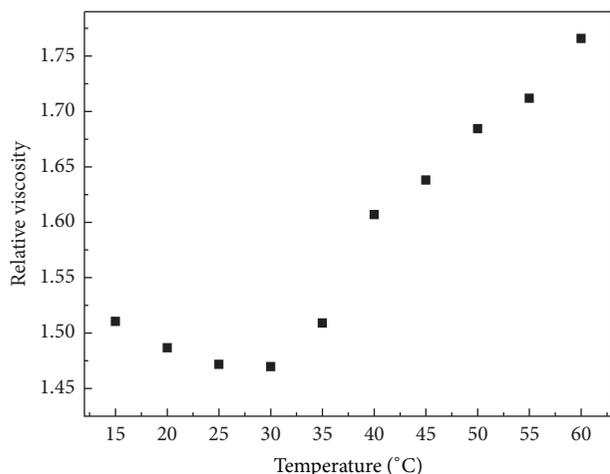


FIGURE 2: Variation of the relative viscosity for a solution of concentration of 20 mg/mL of Pluronic F68 in water for various temperatures.

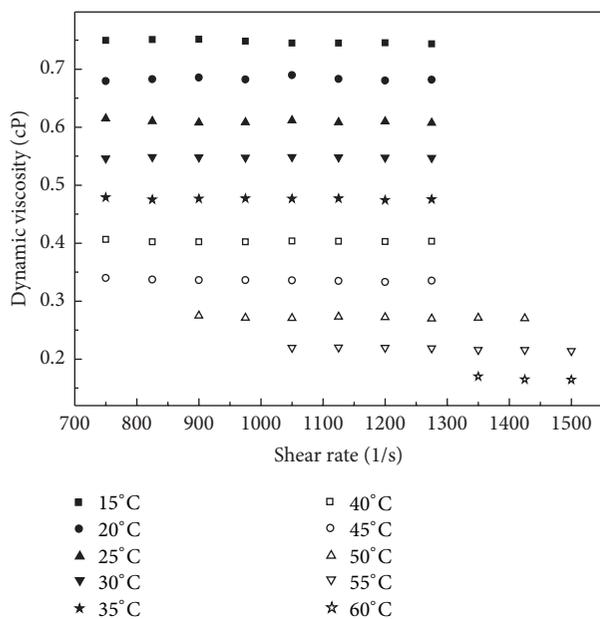


FIGURE 3: Variation of the dynamic viscosity versus the shear rate with no time dependency for a concentration of 20 mg/mL of Pluronic F68 in pure p-xylene.

Pluronic F68 in pure p-xylene. The constancy of the dynamic viscosity with shear rate indicates that the solution exhibits a Newtonian behavior for all temperatures. Figure 4 depicts the variation of the relative viscosity versus temperature for a concentration of 20 mg/mL of Pluronic F68 in pure p-xylene. The behavior was similar, but the viscosity values were lower than those observed for the binary F68/water mixture. We suggest this is due to the fact that the Pluronic F68 in pure p-xylene does not form polymolecular structures as direct and reverse micelles for the range of temperatures considered. In fact, between 15 and 30°C the lower decrease of the relative viscosity indicates that the PEO-PPO-PEO blocks are swollen and extended around 15°C, but they progressively

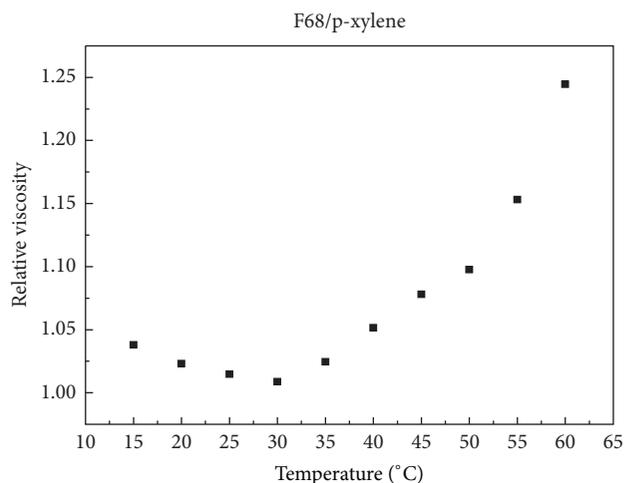


FIGURE 4: Variation of the relative viscosity versus temperature for a concentration of 20 mg/mL of Pluronic F68 in pure p-xylene.

recover their unperturbed conformations with increasing temperatures because both PPO and PEO chains have the same properties in p-xylene in this range of temperature [30]. Above 30°C, an increase of the viscosity was observed. This fact is probably due to the thermal expansion of PPO and PEO chains, since the p-xylene becomes a good solvent for both PEO and PPO blocks with increasing temperatures. The PEO-PPO-PEO blocks become less soft that make the formation of the direct or the reverse micelles in pure p-xylene impossible. These results are in good agreement with the results obtained by Ghaouar et al. [1] using DLS measurements for various concentrations of F68 and L64 dissolved in pure p-xylene at room temperature, where they have shown the nonexistence of a CMC for both Pluronics studied. An additional factor that makes the direct or the reverse micelles formation impossible in pure p-xylene is that the PPO block was much shorter than the PEO blocks for Pluronic F68 [3, 12].

Hence, we believe that the Newtonian behavior of the binary Pluronic F68/p-xylene is due to the nonformation of complex conformations as direct or reverse micelles.

**3.1.3. Binary Pluronic F68/Phenol.** Figure 5 shows the variation of the dynamic viscosity versus the shear rate with no time dependency for a concentration of 20 mg/mL of Pluronic F68 in pure phenol. The constancy of the dynamic viscosity with the shear rate indicates that the solution exhibits a Newtonian behavior for all temperatures. Figure 6 illustrates the corresponding relative viscosity versus temperature of Pluronic F86 dissolved in pure phenol. For the range of temperatures lying between 15 and 30°C, the relative viscosity remained practically constant, with a higher value than those obtained for F68/water and F68 p-xylene solutions in the same range of temperatures. During this phase, we suggest that the PEO-PPO-PEO blocks were swollen due to the accumulation of phenol inside the blocks for this range of temperatures [31], where we think that during this range of temperatures, the formation of micelles becomes impossible.

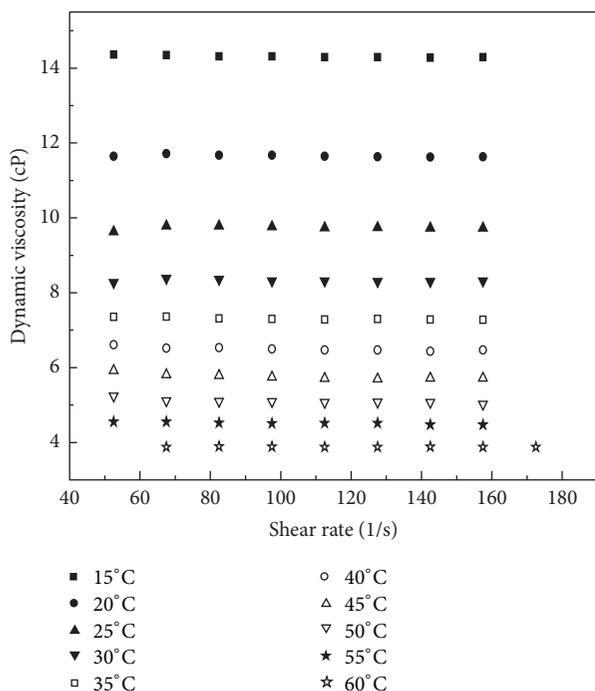


FIGURE 5: Variation of the dynamic viscosity versus the shear rate with no time dependency for a concentration of 20 mg/mL of Pluronic F68 in pure phenol.

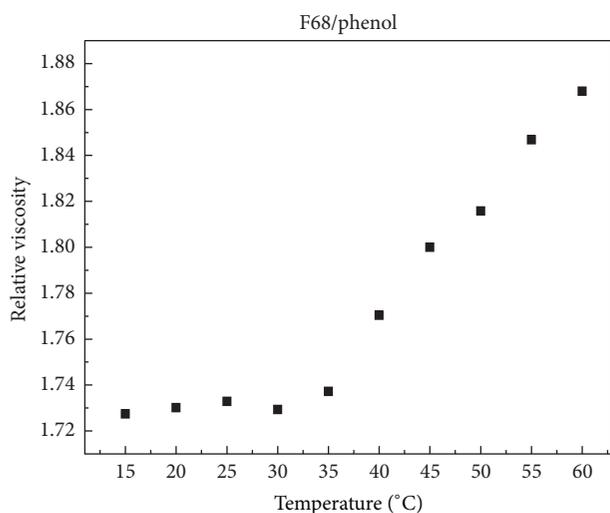


FIGURE 6: Variation of the relative viscosity versus temperature for a concentration of 20 mg/mL of Pluronic F86 dissolved in pure phenol.

With increasing temperature the relative viscosity increased along with a novel transition start. The phenol becomes hydrophobic in this range of temperatures which covers the core formed by PPO chains inducing a micelle growth [32]. The increase of the relative viscosity is then because of the anisotropic growth of the micelles size resulting from the successive shape transformation from the micelles to the ellipsoidal or to the worm-like structures [32–36]. Hence, neither the swollen PEO-PPO-PEO blocks nor the great size

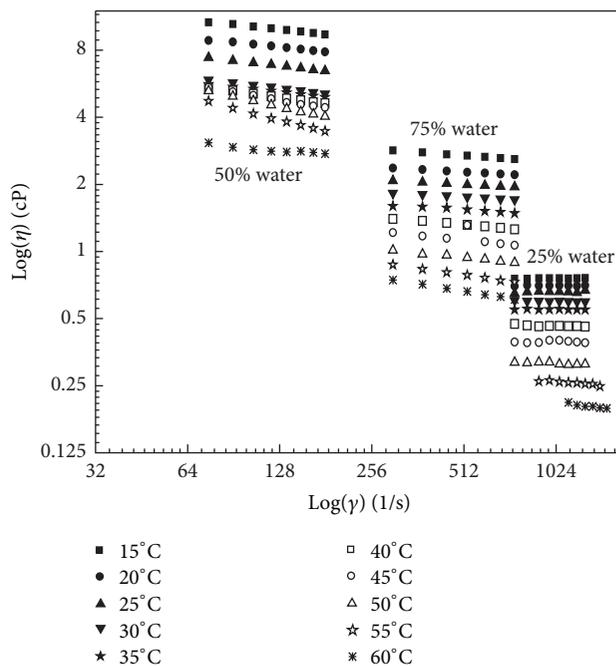


FIGURE 7: Variation of the dynamic viscosity versus the shear rates for various temperatures of Pluronic F68 in p-xylene with different water content.

of the micelles and conformations affected the Newtonian behavior of solution.

### 3.2. Ternary Pluronic F68/Organic Solvent/Water Mixtures

**3.2.1. Pluronic F68/p-Xylene/Water.** Figure 7 shows the logarithmic variation of the dynamic viscosity versus the shear rates for various temperatures of Pluronic F68 in p-xylene with different water content. We show that for 25% water, the dynamic viscosity remained constant with shear rate for all temperatures, indicating that the solution had a Newtonian behavior. For water concentrations of 50 and 75%, the variation of the dynamic viscosity became nonlinear and the solution had a non-Newtonian behavior. The behaviour corresponds to a liquid-plastic phase.

Figure 8 illustrates the variation of the relative viscosity for various temperatures for Pluronic F68 in p-xylene with different water content. We show that for 25% water, the viscosity increased while the solution retained its Newtonian behavior for all temperatures. We consider that for a water amount of 25%, there was no remarkable change compared to a binary F68/p-xylene mixture. For an amount of water of 50%, the relative viscosity increased for temperatures lower than 35°C. The growth of the viscosity was suggested to be due to the folding of PPO blocks permitting the penetration of the end PEO blocks into the micelle core. The longer lengths of the PEO blocks make the formation of the reverse micelles easy [37]. Around 35°C, a sudden decrease of the relative viscosity was observed. We suggest this is due to the disintegration of the reverse micelles, where the PEO-PPO-PEO blocks begin to behave as free chains in solution. For this range of temperature, the water becomes a poorer

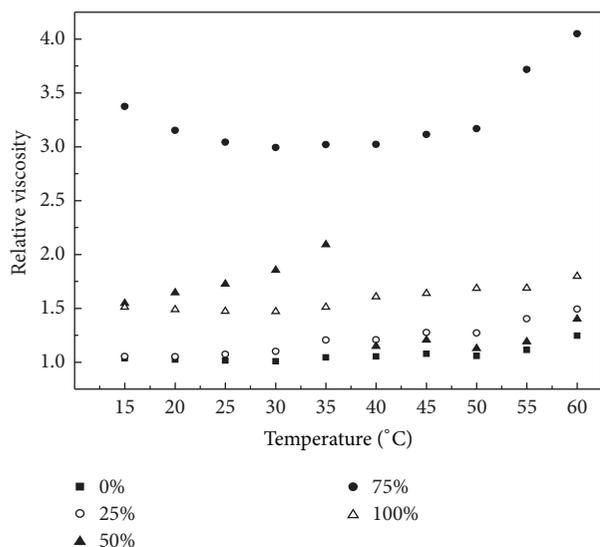


FIGURE 8: Variation of the relative viscosity for various temperatures for a solution of 20 mg/mL of Pluronic F68 in p-xylene with different water content.

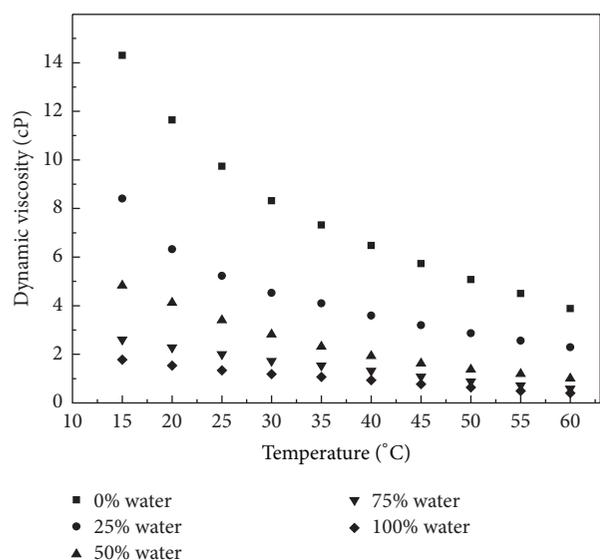


FIGURE 9: Variation of the dynamic viscosity of Pluronic F68 in phenol for various amounts of water added to a solution of 20 mg/mL versus temperature.

solvent, whereas the p-xylene is considered a better solvent for the PEO-PPO-PEO blocks, and the solution is regarded as two phases. The first reaches the water and PEO chains, whereas the second consists of p-xylene and PPO chains ensuring the separation between the two phases. Hence, we related the non-Newtonian behavior to the reverse micelles conformation for temperatures lower than 35°C and to the phase separation for temperatures greater than 35°C. For a water amount of 75%, the relative viscosity increased considerably. We consider the water excess leads to a change of the solution properties by influencing the micelles formation, where we assume that various polymolecular reverse micelles in different shapes and architectures are formed [38, 39].

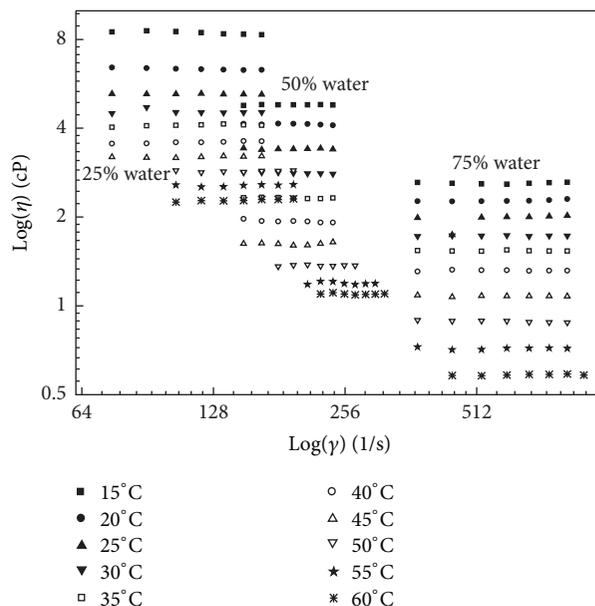


FIGURE 10: Variation of the dynamic viscosity versus the shear rate for a range of temperatures lying between 15 and 60°C.

Indeed, we observe probably the following transitions: (i) reverse micelles to reverse body-centred cubic phase, (ii) reverse body-centred cubic phase to reverse hexagonal phase, and (iii) reverse hexagonal phase to reverse lamellar phase [40, 41]. We recall that the solution has a non-Newtonian behavior during all of the conformational transitions. For a water concentration of 100%, the variation was similar to that in Figure 3. The rheological properties of the ternary F68/p-xylene/water mixture depended strongly on the water concentration. It started with a Newtonian behavior for small amounts of water (0 and 25%), where we have considered that the PEO-PPO-PEO blocks were free and extended. The solution changed to a non-Newtonian liquid plastic behavior with increasing water concentrations, where the reverse micelles are formed. With higher water concentrations, the solution returned back to its Newtonian behavior, where the direct micelles are formed.

**3.2.2. Ternary F68/Phenol/Water Mixture.** Figure 9 represents the variation of the dynamic viscosity of Pluronic F68 in phenol with various amounts of water added to a solution of 20 mg/mL versus temperature. The dynamic viscosity decreased with increasing temperatures and water concentration. We suggest that the water modifies the environment and degrades the accumulation of the phenol near the micelles. The PEO chains interact favorably with the surrounding solvent to form hydrogen links with water, whereas the phenol covers the core formed by PPO chains. With increasing water rates, the micelle growing is stopped by the rejection of the phenol covering the PPO core. The solution is regarded as two phases. The first consists of phenol and the second consists of micelles surrounded by water.

Figure 10 shows the logarithmic variation of the dynamic viscosity versus the logarithm of shear rate for a range of

temperatures between 15 and 60°C. For water contents of 25 and 50%, the dynamic viscosity had a nonlinear behavior for temperatures lower than 20°C, with the solution behaving as a non-Newtonian fluid, whereas for temperatures superior to 20°C the variation became linear and the solution was considered Newtonian. For a water content of 75%, the solution remained Newtonian for all temperatures. Consequently, with a progressive water addition the behavior of the solution passes from a non-Newtonian to a Newtonian.

#### 4. Conclusions

We recall that during this work, we are interested in the rheological properties of Pluronic F68 dissolved in various mixtures and a wide range of temperatures for a concentration belonging to the dilute regime. We have shown that for binary F68/water, the solution has a Newtonian behavior during various transitions that start from elongated PEO-PPO-PEO blocks to elongated structures. For binary F68/p-xylene there are no complex structures detected and the solution keeps its Newtonian behavior for all temperatures. The various transitions also observed for binary F68/phenol, where the PEO-PPO-PEO blocks pass from the swollen state to micelles with greater size, show that the solution has a Newtonian behavior.

We have also shown that for the ternary F68/p-xylene/water mixture, the rheological properties and the conformational change of Pluronic F68 depend strongly on water concentration. The solution begins with a Newtonian behavior for little amounts of water and loses this behavior to non-Newtonian liquid plastic behavior with increasing water concentrations, where we have assumed the non-Newtonian behavior to the existence of the reverse micelles. For ternary F68/water/phenol and F68/phenol/water mixtures, the behavior of solution depends strongly on temperature and the amount of phenol or water added.

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