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Effect of an amphiphilic polymer on the evaporation behavior of its solutions in toluene and in water

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ABSTRACT

The evaporation behavior of solutions of an amphiphilic polymer, phosphonated polybutadiene in toluene and in water was studied by means of vapor pressure and evaporation rate measurements. The polymer reduces the vapor pressure and evaporation rate of toluene, while the opposite effect was observed in aqueous solutions. The effects were explained on the basis of the Flory–Huggins theory in the toluene solutions and the structure breaking effect in the aqueous solutions.

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1. Introduction

Evaporation and condensation processes play an important role in various fields of science and technology. Although experimental and theoretical studies concerning the evaporation and condensation rates have a long history, it is still difficult to obtain the absolute rate. One of the main difficulties is due to the fact that there is not a clear view of the dynamic processes at liquid surfaces yet [1].

We have previously studied the capacity of an amphiphilic polymer, phosphonated polybutadiene (PPB) as collector of petroleum spills [2], which forms a direct emulsion of toluene in water, where the toluene droplets are interconnected by polymer chains so the emulsion cannot be diluted by water but floats on it. This emulsion was heated to break it and the volatile components were distilled to separate them. Although the boiling points of these components

are 100 (water) and 110 °C (toluene), distillation begins at 180 °C when the liquid starts to boil. This phenomenon led us to investigate the effect of PPB on the liquid–vapor phase transition in both liquids, since the elucidation of this behavior may shed light on the mechanisms that govern the evaporation of solutions.

2. Experimental

Commercial polybutadiene (PASA S.A., Argentina; M_n : 102,500 g mol⁻¹; M_w/M_n : 2.02; 10% of 1,2-addition units) was used as received. The production and characterization of the phosphonated polybutadiene (PPB) was described elsewhere [2]. The equivalent weight of PPB is 899 ± 18, what means that there is one phosphonic group each 15.1 ± 0.3 butadiene monomers. The emulsion was broken by heating and the aqueous and toluene phases were separated by decantation. To determine the PPB content in each phase the polymer was weighed after the evaporation of the solvent of a known amount of solution in a previously weighed vessel.

The vapor pressure at different temperatures was measured by means of an isoteniscope following the procedure described in literature [3]. The evaporation velocity at different temperatures (22.0

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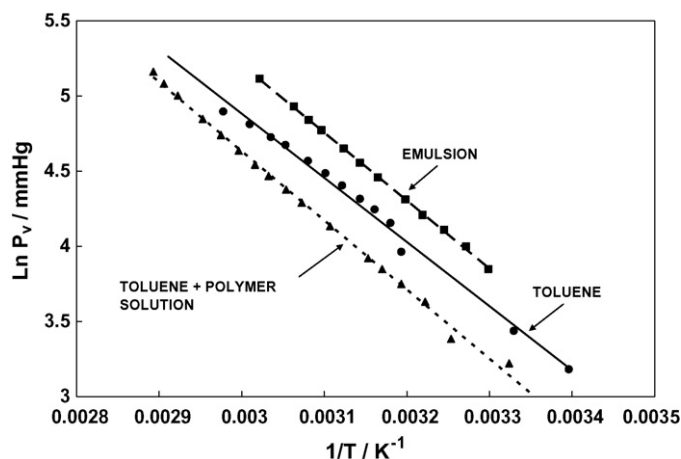


Fig. 1. Neperian logarithm of the vapor pressure (in mmHg) of pure toluene (●), of the toluene PPB solution obtained by rupture of the emulsion (▲) and the entire emulsion (■), as a function of the inverse of the absolute temperature.

and 34.0 ± 0.1 °C) was determined by measuring the loss of weight of a known volume of sample placed in a small pyrex cylinder having 2.9 cm internal diameter in a CAHN 1000 electrobalance, operating in a register range of 100 mg and an output of 10 mV. The pressure was 101.325 kPa. To avoid secondary Archimedes effects, a compensation container was hung in the other arm of the electrobalance. The thermocouple (Fe-constantan) was in contact with the solution through a thin glass sheath.

3. Results and discussion

3.1. The PPB in toluene solution

3.1.1. Vapor pressure

Fig. 1 shows the results of vapor pressures, measured with the isoteniscope, of pure toluene, the unbroken emulsion (E) and the PPB solution in toluene (T + P, $C = 1.2679$ g%) obtained by emulsion breakage. The emulsion was broken at 110 °C (to ensure the complete process, it was heated under reflux at 120 °C for 15 min), but the P + T solution ebullition began at 170 °C.

By analysis of the data the enthalpy values of pure toluene ($\Delta H_{\text{vap,T}} = 35.5 \pm 0.8$ kJ mol⁻¹), the T + P solution ($\Delta H_{\text{vap,T+P}} = 38.2 \pm 0.9$ kJ mol⁻¹) and the complete emulsion ($\Delta H_{\text{vap,E}} = 37.6 \pm 0.4$ kJ mol⁻¹) were obtained. The enthalpy of evaporation of the T + P solution was then 2.76 kJ mol⁻¹ higher than that of the pure toluene used in the experiment.

On the other hand, the vapor pressure in the T + P solution is lower than that of pure toluene. Fig. 2 shows the relative change in vapor pressure $\Delta P_v/P_v^\circ$, where $\Delta P_v = P_v - P_{v,s}$, and P_v° is the vapor pressure of pure toluene and $P_{v,s}$ is that of the sample (P + T for the solution, E for the complete emulsion). For both samples $\Delta P_v/P_v^\circ$ is a linear function of temperature, showing high correlation coefficients.

The unbroken emulsion (E) shows a vapor pressure higher than toluene and T + P.

By using the Clausius–Clapeyron equation together with the values obtained for the P + T solution, the normal boiling point of the solution was computed, resulting $T_b = 116.8 \pm 2.8$ °C, which is considerably lower than the experimentally observed when the emulsion was broken in order to eliminate the toluene by distillation (~170 °C) to determine the polymer content in the hydrocarbon phase. As a consequence, the increase in the boiling point (from 110.6 in pure toluene to ~170 °C in the P + T solution) is in part caused by a combination of the vaporization enthalpy

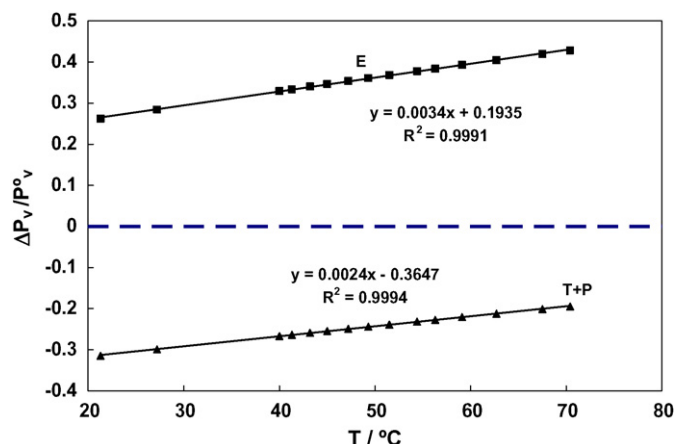


Fig. 2. $\Delta P_v/P_v^\circ$ as a function of absolute temperature for the complete emulsion (E, ■) and the P + T solution obtained by rupture of the emulsion (T + P, ▲).

increase and vapor pressure decrease, but another mechanism must also be involved.

The ebullioscopic constant of toluene is $\Delta T/c_2 = k_{\text{eb}} = 3.33 \times 10^3$ °C mol⁻¹, where c_2 is the solute concentration in g L⁻¹ [4] we computed the molar mass of the polymer was a very small value, confirming that colligative properties cannot be used in this kind of solutions.

The molar weight of the polybutadiene used in the synthesis was 102,500 Da, which, together with the molar weight of the monomer ($M_{\text{butadiene}} = 54.1$ g mol⁻¹) gives an average of 1985 monomers per molecule. The proportion of phosphonic groups in PPB is 15.1 [2] thus living 897.8 phosphonic groups per PPB molecule, and then, the molecular weight of PPB results $M_{\text{PPB}} = 112,663$ Da. The polymer content in the solution was 1.2679 g%, that is to say 10.991 g L⁻¹ = 9.756×10^{-5} mol dm⁻³. With these data we computed the value of the ebullioscopic increase that would be expected, $\Delta T = 3.25 \times 10^{-4}$ °C. As a consequence, the observed phenomenon cannot be caused by common colligative properties of solutions.

The system was then analyzed by means of the Flory–Huggins theory [5], which deals with a mixture of a solvent 1 and a polymeric solute 2, which behaves as a flexible chain formed by r flexible segments, each of them has the same size as the solvent molecules. In a system formed by N_1 solvent molecules and N_2 molecules of polymer, the total number of lattice sites is $(N_1 + rN_2)$. The fractions of sites occupied by solvent and polymer (Φ_1^* and Φ_2^*) are given by:

$$\Phi_1^* = \frac{N_1}{N_1 + rN_2} \text{ and } \Phi_2^* = \frac{rN_2}{N_1 + rN_2} \quad (1)$$

For real solutions of polymers, which are not athermal (i.e. those whose mixing enthalpy is zero, which approximately occurs when polymer and solvent have the same basic structure), the activity of solvent in the mixture is given by:

$$\ln a_1 = \ln(1 - \Phi_2^*) + \left(1 - \frac{1}{r}\right) \Phi_2^* + \chi \Phi_2^{*2} \quad (2)$$

and the related activity coefficient (on a mole fraction basis) is:

$$\ln \gamma_1 = \ln \left[1 - \left(1 - \frac{1}{r}\right) \Phi_2^*\right] + \left(1 - \frac{1}{r}\right) \Phi_2^* + \chi \Phi_2^{*2} \quad (3)$$

The value of γ_1 is strongly dependent on the value of r for low values of this parameter, however, for large ones ($r > 100$) γ_1 becomes independent on r . The semi-empirical Flory–Huggins interaction parameter χ represents the residual contribution caused by the mixing enthalpy, and is determined by intermolecular forces. It is supposed that χ is not dependent on the composition. However it was experimentally observed that this parameter is dependent on

the polymer concentration and this dependence is demonstrated in more sophisticated theories. The Flory-Huggins parameter is determined by the energies that characterize the interactions between pairs of segments of the polymer, between pairs of solvent molecules and between a polymer segment and a solvent molecule, which is considered as the “exchange energy” w :

$$\chi = \frac{w}{k_B T} \quad (4)$$

where k_B is the Boltzmann constant. In terms of the solubility parameters it may be demonstrated that:

$$\chi = v_1 \frac{(\delta_1 - \delta_2)^2}{RT} \quad (5)$$

where v_1 is the solvent molar volume and δ_1 and δ_2 are the solubility parameters of solvent and polymer, respectively.

When $\chi > 0$ there is a superior limiting temperature in which partial miscibility exists, whereas if $\chi \leq 0$ complete miscibility may be obtained at any temperature. If $\chi = 0$ the solution is athermal. In many polar systems negative values of χ have been observed [5].

There is a critical composition $\Phi_2^{*c} = 1/(1 + \sqrt{r})$ in which an incipient instability of solution appears.

The above equations are commonly used to represent properties of solutions whose components strongly differ in molecular size. In general, these solutions show negative deviations of Raoult's law, which increase when the size difference rises.

For polybutadiene $\delta_2 = 17.4 \text{ (J cm}^{-3}\text{)}^{1/2}$ [6] and for toluene $\delta_1 = 8.9 \text{ (J cm}^{-3}\text{)}^{1/2}$ [7]. The molar volume of toluene can be computed with its density (0.8669 g cm^{-3}) [8] and its molar weight giving $106.3 \text{ cm}^3 \text{ mol}^{-1}$. Taking the polybutadiene solubility parameter as if it were that of the PPB, the value of $\chi = 3.10$ is obtained for the studied system. This value predicts that the solution must show a positive deviation to the Raoult's law with repulsion between components as indicated by Eq. (6). However, this is not the observed behavior.

The molar volume of toluene is $106.3 \text{ cm}^3 \text{ mol}^{-1}$ and that of butadiene $87.09 \text{ cm}^3 \text{ mol}^{-1}$ so, the segment of polymer with the same volume as one solvent molecule may be supposed formed by 1.22 butadiene monomers. This gives $r = 1553$. Then the values $\Phi_1^* = 0.9978$ and $\Phi_2^* = 1.269 \times 10^{-3}$ were computed. Since $\Phi_2^{*c} = 0.0247$, the solution is stable. Then, with Eq. (2) the values of χ as a function of the temperature were obtained and represented, giving a the linear relationship $\chi = (0.270 \pm 0.001)T \text{ (K)} - 108 \pm 01$ ($R^2 = 0.9974$).

Accordingly Eq. (4), the negative value indicates an attractive interaction between polymer and toluene, which is coherent with the negative deviation to the Raoult's law shown by P+T solutions. The difference between results from Eqs. (2) and (5) indicates that Eq. (5) is not applicable in all situations. A difference between the values of solubility parameters of polybutadiene and PPB cannot explain the discrepancy.

The high χ negative values may be associated to the polar nature of PPB. It is known that π -electrons of aromatic rings may form hydrogen bonds with water or other proton-donors [9–12]. This also contributes to the attractive interaction between PPB and toluene.

The activity coefficient of toluene as a function of temperature was obtained by Eq. (3) and is shown in Fig. 3. It may be seen that in the temperature range explored in this work the relationship is linear and the slope is positive. By extrapolation, $\gamma_1 = 1$ at $130 \pm 3 \text{ }^\circ\text{C}$.

The values of χ indicate that the solution is not athermal and then there is an enthalpic contribution to the solubilization process of PPB in toluene. Consequently, the non-ideality of the system is caused by a combination of a mixture of components having very different molecular weights and thermal effects. Because of the approximations here made, and the relatively simple Flory-

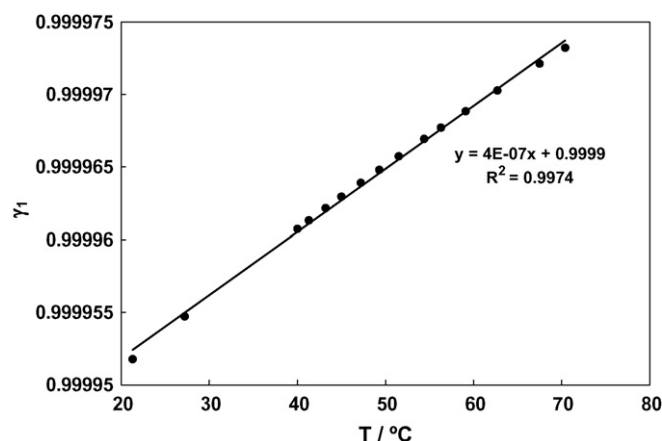


Fig. 3. Activity coefficient of toluene as a function of the temperature in P+T solutions, computed with Eq. (5).

Huggins theory it is not possible to attain more quantitative information, but a general idea about the causes of the non-ideality may be obtained.

The theory interprets the term

$$\Delta H^m = \chi \Phi_1^* \Phi_2^* (N_1 + rN_2) \quad (6)$$

as an estimation of the enthalpy of mixing. However, direct measurements of ΔH^m in other systems studied in literature gave discrepancies, which were attributed to that the value of χ obtained from experimental activities has an entropic as well as enthalpic part [5]. The values of ΔH^m obtained with Eq. (8) were represented as a function of temperature, giving the linear relationship $\Delta H^m \text{ (J mol}^{-1}\text{)} = (6.4110 \pm 0.0006)T \text{ (K)} - (2760 \pm 1)$ ($R^2 = 1$). The negative values are in agreement with the possible formation of hydrogen bonds between toluene and phosphonic acid groups.

3.1.2. Evaporation velocity

The evaporation velocity follows a time-dependent law of the form [13]:

$$v = v_0 - Kt^{1/2} \quad (7)$$

where v_0 is the initial velocity ($\text{mg s}^{-1} \text{ m}^{-2}$), t the time and K is related to the rate of diffusion of molecules with sufficient energy to evaporate, from the bulk to the surface, where the initial evaporation created a deficit of energetic molecules.

The only hindrance for the initial rate of evaporation v_0 is the reluctance of the molecules of the evaporating substance to leave the surface in the initial state of equilibrium, which is related to the evaporation enthalpy [13].

Fig. 4 shows the evaporation rate of pure toluene and the T+P solution.

At $22 \text{ }^\circ\text{C}$ the initial rate of evaporation in the T+P sample ($v_0 = 5.68 \times 10^{-7} \text{ kg m}^{-2} \text{ s}^{-1}$) is lower than that of pure toluene (T, $v_0 = 1.46 \times 10^{-6} \text{ kg m}^{-2} \text{ s}^{-1}$). The constant K is also lower for T+P ($K = 2.95 \times 10^{-7} \text{ kg m}^{-2} \text{ s}^{-3/2}$) than that for T ($K = 2.65 \times 10^{-6} \text{ kg m}^{-2} \text{ s}^{-3/2}$). The initial velocity reduction may be due to a reduction of the toluene activity caused by the polymer–toluene interaction (as indicated by the negative values of the Flory-Huggins interaction parameter χ). The reduction of K indicates that the reposition of molecules of toluene having enough energy to evaporate at the surface is slower in the T+P solution than in pure toluene. One interpretation of K relates it with the molecular diffusion coefficient of the evaporable species by the equation [14]:

$$K \text{ (kg m}^{-2} \text{ s}^{-3/2}\text{)} = 2M(D/\pi)^{1/2}(p^*/RT - c_i) \quad (8)$$

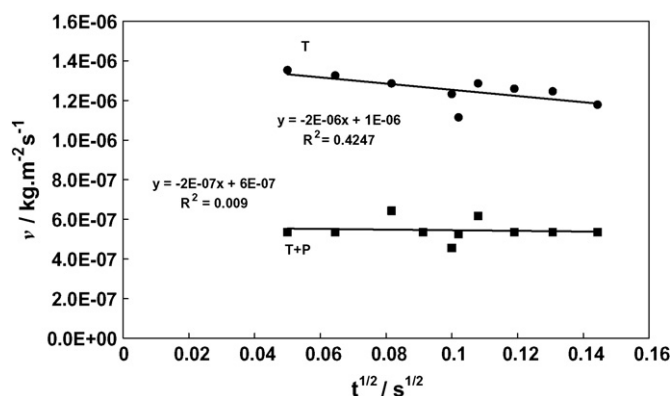


Fig. 4. Evaporation rate of pure toluene (T, ●) and toluene with polymer (T+P, ■) at 22 °C, as a function of time.

where M is the molecular weight of the volatile component, D its diffusion coefficient, p^* its vapor pressure at the experiment temperature and c_i the concentration of the volatile component in the gaseous phase, which depends on the experimental conditions and can be obtained from the experiment with pure toluene. The pure toluene self-diffusion coefficient at 22 °C is $D_T = 1.17 \times 10^{-6} \text{ m}^2 \text{ s}^{-1}$ [15]. With the vapor pressure of pure toluene at 22 °C the value $c_i = 1.56 \text{ mol m}^{-3}$ was obtained in the experiment conditions. With these data and the vapor pressure of the T+P system the toluene self-diffusion coefficient in that solution resulted $D_{T+P} = 3.6 \times 10^{-9} \text{ m}^2 \text{ s}^{-1}$. As a consequence, the self-diffusion coefficient of toluene was strongly reduced in the system P+T, which may explain the reduction in the evaporation rate.

Taking into account the concentration of polymer in toluene and the value of r , it may be computed that there are about 61 molecules of toluene by each segment of the polymer. Due to the negative value of χ , many of these toluene molecules have an attractive interaction with the polymer. The solvated polymer may also hinder the movement of the toluene molecules. Then, the toluene molecules which may migrate freely are much less than the total solvent content of the solution. This steric effect is then added to the thermal effects detected in the vapor pressure experiment.

3.2. The aqueous phase

3.2.1. Vapor pressure

The vapor pressure measurement in the aqueous solution of the polymer (W+P) showed a linear dependence with the expression:

$$\ln(P_v/\text{mmHg}) = \frac{-(2355 \pm 166)}{T} + 12.03 \pm 0.55 \quad (R^2 = 0.9927)$$

giving an evaporation enthalpy of water in the W+P system $\Delta H_{\text{vap,W+P}} = 19.6 \pm 1.4 \text{ kJ mol}^{-1}$. That of pure water in the experiment was $\Delta H_{\text{vap,W}} = 43.31 \pm 0.12 \text{ kJ mol}^{-1}$.

3.2.2. Evaporation rate

The evaporation rate in the aqueous polymer solution (W+P) gave $K = (2.12 \pm 0.21) \times 10^{-5} \text{ mg m}^{-2} \text{ s}^{-3/2}$, $v_0 = (8.71 \pm 0.25) \times 10^{-5} \text{ mg m}^{-2} \text{ s}^{-1}$, $R^2 = 0.953$ and for pure water (W), $K = (3.17 \pm 0.67) \times 10^{-6} \text{ mg m}^{-2} \text{ s}^{-3/2}$, $v_0 = (2.07 \pm 0.18) \times 10^{-5} \text{ mg m}^{-2} \text{ s}^{-1}$ with $R^2 = 0.755$. In this case the inclusion of the polymer produces a behavior which is the inverse of that produced in toluene: both the initial evaporation velocity and the rate of reposition to the surface of molecules with enough energy to evaporate are higher in the W+P phase than in pure water.

To apply Eq. (8) we used $D_s = 3 \times 10^{-5} \text{ m}^2 \text{ s}^{-1}$ [16] and $p^* = 2642.73 \text{ Pa}$ [8] for pure water and $p^* = 7731 \text{ Pa}$ for the system W+P at 22 °C, determined in this work. This gave $D_s = 2.49 \times 10^{-5} \text{ m}^2 \text{ s}^{-1}$ in the W+P solution.

It may be expected that the inclusion of bulky, scarcely ionized polar groups ($-\text{PO}_3\text{H}_2$) and consequently scarcely charged (having a low ionization constant $\text{p}K_{a1} \approx 3$ [17] and whose partial molar volume is $43.22 \pm 0.96 \text{ cm}^3 \text{ mol}^{-1}$ [18]) causes a structure breaking effect on water, thus increasing the proportion of water molecules weakly bound to the hydrogen bonded network. Consequently these molecules are free to migrate to the interface and evaporate. The perturbation of the hydrogen bonds by the solute has been proposed to explain an increase of the evaporation rate of water in solutions of cationic surfactants [19].

Details on the structure breaking effect of ions on water may be found in a recent review [20]. The validity of this effect in dilute solutions was stated by several authors [21,22]. With a concentration of PPB of $3.15 \times 10^{-5} \text{ mol dm}^{-3}$ the concentration of phosphonic groups is $0.0283 \text{ mol dm}^{-3}$. Assuming the value of $\text{p}K_2 = 7.985 \pm 0.003$ which was determined for n-decane phosphonic acid [17], the ionization degree of the phosphonate groups in pure water results $\alpha \approx 0.059$, which is the average charge of these groups. One way of quantifying the structure breaking or structure making capacity of ions is by the ΔG_{HB} value, the change in the average total geometrical factors over all the configuration of the N water molecules of either kind caused by the introduction of a particle of solute [20]. Positive values of ΔG_{HB} are shown by structure maker and negative values by structure breaker solutes. Plotting ΔG_{HB} for PO_4^{3-} (0.9–1.1), HPO_4^{2-} (0.4–0.7), H_2PO_4^- (–0.1 to 0.1) and H_3PO_4 (–0.6 to –0.37) (obtained from reference [20]) against the value of the charge, the ΔG_{HB} value for the phosphonate group in water may be estimated between –0.34 and –0.57, which corresponds to a strong structure breaking capacity.

Consequently, the evaporation of water in the W+P solution requires less energy than in pure water because the structure breaking effect reduces the proportion of hydrogen bonds which must be broken to produce the solution-to-vapor transition of molecules. An alternative but equivalent interpretation is that the hydrogen bonds are weaker than in pure water and consequently require less energy to break. This may also be the explanation of the high vapor pressure of the complete emulsion, since the continuous phase has a vapor pressure higher than pure water.

4. Concluding remarks

The phosphonated polybutadiene solution in toluene shows a reduction of the vapor pressure, the evaporation enthalpy and the evaporation rate which may be explained by a strong attractive interaction between the polymer and the solvent, as indicated by the Flory-Huggins theory. This interaction may in part be due to hydrogen bonds between the phosphonic acid groups and the π -electrons of toluene. On the contrary, the aqueous solution show an increase in the vapor pressure and reduction in the evaporation enthalpy in comparison with pure water, which is attributed to a structure breaking effect of the phosphonate groups. This effect on water partially compensates the effect of the polymer on toluene and explains the evaporation behavior of the complete emulsion.

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