Effects of Temperature on the Conductivity of AOT/Isooctane/Water Microemulsions. Influence of Salts

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The effect of temperature on the conductivity of ternary systems sodium bis(2-ethylhexyl)sulfosuccinate + 2,2,4-trimethylpentane + water has been studied. The effect of LiCl, LiBr, $LiClO_4$, KCl, RbCl, CsCl, NH_4Cl, and $CaCl_2$ has also been studied.

Introduction

The microemulsions are ternary systems where water microdroplets are dispersed in a continuous phase of an apolar oil (2,2,4-trimethylpentane). These microheterogeneous systems have important applications in chemistry and in the chemical industry. These systems have great potential as solubilizers (Mittal, 1977; Elworthy, 1968; McBain and Hutchison, 1955) or as chemical nanoreactors (García-Rio et al., 1993; García-Río et al., 1995; García-Río et al., 1996). In particular, the microemulsions of sodium bis(2-ethylhexyl)sulfosuccinate + 2,2,4-trimethylpentane + water (AOT + isooctane + water) present special interest because the addition of a forth component (cosurfactant) is not necessary. On the other hand, these microemulsions have a great area of stability in the phase diagram (Pileni, 1989).

Upon increasing the temperature, the specific conductivity of microemulsions increases gradually up to a certain temperature (threshold of percolation) and then increases sharply. This phenomenon is known as electrical percolation. It is well-known that the electrical percolation and the rate constant corresponding to the mass transfer between droplets in microemulsions are correlated (Jada et al., 1989, 1990). This process of mass transfer between droplets is decisive in determining the reactivity in microemulsions. In this way, moderate quantities of additives can modify the value of the temperature of percolation (Mathew et al., 1988).

The aim of this work is to determine the values of the specific conductivity in these systems with various salts in order to modulate the electrical percolation.

Experimental Section

Aqueous solutions of salts were prepared with distilled−deionized water. All the solutes were supplied by Merck and Sigma at the highest purity available (>99%). All the solutions were prepared by mass with deviations of less than ±0.2% from the desired concentrations. In all of the cases the additive concentration has been referred to the water volume of the microemulsion because all of the additives are soluble only in the water pseudophase of the microemulsion. The solutions (microemulsion + additive) were prepared by direct mixing under vigorous stirring.

Measurements of specific conductivity were accomplished in a Radiometer CDM3 conductivity meter with a conductivity cell constant of 1 cm^{-1}. The conductivity meter was calibrated using a solution of KCl ([KCl] = 0.01 $mol \cdot dm^{-3}$). The inaccuracy of these measurements was ±0.5%. During the measures of conductivity the temperature was regulated with a precision of ±0.1 °C. In general, each conductivity value reported was an average of 5−10 measurements, where the maximum deviations from the average value were always less than 1.5%. The point of percolation has been obtained by studying the influence of temperature on the specific conductivity of the microemulsions.

Results

The effect of the nature and additive concentration on the electrical percolation has been studied. Values of specific conductivity/temperature, κ/T, for LiCl, LiBr, $LiClO_4$, KCl, RbCl, CsCl, NH_4Cl, and $CaCl_2$, varying the additive concentration (0.04 and 0.08 $mol \cdot dm^{-3}$), have been measured. The value of the threshold of percolation has been determined and was compared with the effect observed previously (Alvarez et al., 1998) for sodium salts.

Figure 1 shows the effect of chlorides on the specific conductivity of the AOT + isooctane + water microemulsions. Figure 2 shows the effect of increasing the additive concentration upon the specific conductivity of the ternary system. Figure 3 shows the effect of the charge of the cation on the behavior of the system. We can observe in all of the cases that a moderate additive concentration produces a meaningful variation in the temperature of percolation.

The experimental values of κ/T obtained for the different additives and for different additive concentrations are shown in Table 1. The threshold of percolation t_p has been calculated using the Kim method (Kim and Huang, 1986), which has been described in a previous paper (Alvarez et al., 1998) (Figure 4). In Table 2 are shown the calculated temperatures of percolation for the different additive concentrations.

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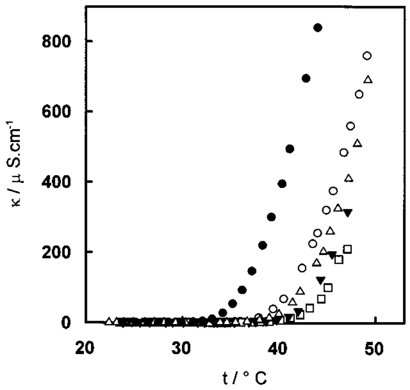

Figure 1. Influence of temperature upon the conductivity of bis-(2-ethylhexyl)sulfosuccinate (AOT) + 2,2,4-trimethylpentane + water microemulsions in the presence of different chlorides: (●) without additive; (○) [LiCl] = 0.04 mol·dm^{-3}; (△) [KCl] = 0.04 mol·dm^{-3}; (▼) [RbCl] = 0.04 mol·dm^{-3}; (□) [CsCl] = 0.04 mol·dm^{-3}.

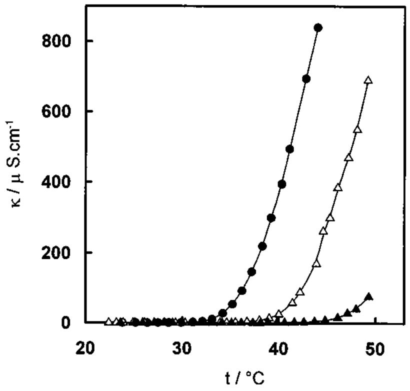

Figure 2. Influence of temperature upon the conductivity of bis-(2-ethylhexyl)sulfosuccinate (AOT) + 2,2,4-trimethylpentane + water microemulsions in the presence of different concentrations of KCl: (●) [KCl] = 0 mol·dm^{-3}; (△) [KCl] = 0.04 mol·dm^{-3}; (▲) [KCl] = 0.08 mol·dm^{-3}.

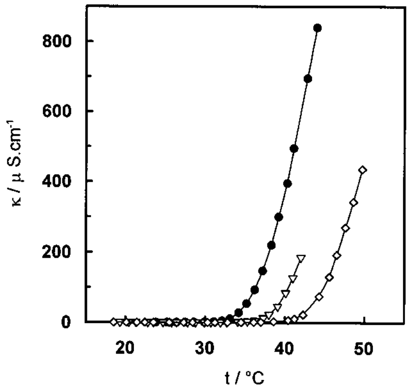

Figure 3. Influence of temperature upon the conductivity of bis-(2-ethylhexyl) sulfosuccinate (AOT) + 2,2,4-trimethylpentane + water microemulsions in the presence of cations with different charge: (●) without additive; (▽) [NH_4Cl] = 0.04 mol·dm^{-3}; (◇) [$CaCl_2$] = 0.04 mol·dm^{-3}.

In all the cases, an increase in t_p has been observed. Also, t_p increases with the additive concentration. This behavior is analogous to what is observed for the sodium salts (Alvarez et al., 1998; García-Río et al., 1994), and it would be justified by their capacity of favoring the opening of channels to facilitate the mass transfer between droplets (García-Río et al., 1994).

The reported values of t_p present a logical trend within the chloride series, which is ranked by increasing effect on t_p in the order HCl < LiCl < NaCl ≤ KCl < RbCl < CsCl (Figure 1), NH_4Cl exerting an effect very similar to that of NaCl.

Discussion and Conclusions

The specific conductivity of these systems was correlated with the temperature by means of an empirical equation (Alvarez et al., 1998) that permits us to predict the experimental values of temperature after and before the threshold of percolation.

$$T = A + B(\kappa)^{1/2} + C/\kappa \qquad (1)$$

The fit of κ/T values was satisfactory for all the studied cases (Figure 5), and the parameters A, B, and C are collected in Table 2. The value obtained for the parameter A corresponds with the temperature of percolation. In all the cases studied, eq 1 reproduces the temperature with a deviation less than 4%.

The effect of added salts upon the behavior of our system can be understood within the more general framework of the effect of salts on the properties of surfactants in solution. It is known that increasing the salt concentration reduces the size of the polar effective area of the surfactants, due to the screening effect of the salt on the electrostatic repulsion between head groups. Such a reduction will cause an increase in the curvature parameter of the surfactant, which is a fundamental parameter in the theory of colloidal aggregate structure. This parameter is defined as v/al, where v is the effective volume of a surfactant molecule, l is the length of its hydrocarbon chain, and a is the effective area of its polar head.

Specifically, the presence of a salt will reduce a, thereby increasing the "spontaneous" negative curvature of the surfactant and its tendency to form microemulsions. Addition of salts implies a decrease in the area per head group, but the volume of the dispersed phase will be the same. In principle, a decrease of the total area at the total dispersed volume corresponds to an increase in the microdroplet radius. However, this increase implies a reduction in the surfactant curvature, which is opposite to the natural tendency of AOT upon salt addition. This variation in the surfactant curvature likewise explains the increase in t_p caused by salts, if the specific conductivity of microemulsions is assumed to be due to the passage of cations through transient channels formed between droplets that have collided. These channels constitute a positive surfactant curvature, and their formation will be more difficult the lower the natural tendency of the surfactant to adopt positive curvature. Indeed, the relative efficiency of cations in increasing t_p ($H^+ < Li^+ < Na^+ \leq K^+ < Rb^+ < Cs^+$, see Figure 1) exhibits exactly the order predicted by the radii of the hydrated cations. The smaller the cation, the greater its association with the surfactant heads and the greater the consequent reduction in the area of the latter. Thus, cation−AOT association in w/o microemulsions appears to involve the hydrated cation—at least at $W = [H_2O]/[AOT] = 22.2$. This retention of the cation's hydration layer appears to be typical of association due to weak electrostatic fields. This interpretation of the effect of electrolytes on the properties of w/o microemulsions is in keeping with the relative insensitivity of t_p to the anion accompanying the cation in the salt. If repulsion by the AOT head group keeps the anion in distant aqueous regions in the microdroplet, it can have little effect on the area of these heads.

The strong effects of Na_2SO_4 and Ca_2Cl (Table 2) may be attributed to the former releasing two sodium ions upon dissociation (rather than the single cation released by all

Table 1. Specific Conductivity Values at Different Temperatures, without Additive and in the Presence of Different Concentrations of Salts in Sodium Bis(2-ethylhexyl)sulfosuccinate (AOT) + 2,2,4-Trimethylpentane + Water Microemulsions

t/°C	κ/μS cm^{-1}	t/°C	κ/μS cm^{-1}	t/°C	κ/μS cm^{-1}	t/°C	κ/μS cm^{-1}	t/°C	κ/μS cm^{-1}	t/°C	κ/μS cm^{-1}	t/°C	κ/μS cm^{-1}	t/°C	κ/μS cm^{-1}
Without Additive, [AOT] = 0.50 mol·dm^{-3}, [H$_2$O]/[AOT] = 22.2															
23.8	0.37	29.9	1.29	35.2	54.00	40.3	395.00	27.5	0.62	33.1	10.90	38.3	220.00	44.0	840.00
25.2	0.43	31.1	2.60	36.2	93.00	41.1	495.00	28.6	0.82	34.2	28.00	39.2	300.00	45.1	1000.00
26.4	0.51	32.1	5.00	37.2	147.00	42.8	695.00								
[LiCl] = 0.04 mol·dm^{-3}, [AOT] = 0.50 mol·dm^{-3}, [H$_2$O]/[AOT] = 22.2															
23.4	0.32	30.0	0.62	37.9	14.70	45.6	375.00	27.3	0.44	34.4	2.10	43.5	225.00	49.1	760.00
24.1	0.34	30.8	0.72	39.4	39.50	46.7	485.00	28.5	0.49	35.4	3.25	44.0	255.00		
25.1	0.36	32.0	0.94	40.5	68.00	47.4	560.00	29.2	0.55	36.0	4.45	44.9	320.00		
26.0	0.39	33.1	1.27	42.4	156.00	48.3	650.00								
[LiCl] = 0.08 mol·dm^{-3}, [AOT] = 0.50 mol·dm^{-3}, [H$_2$O]/[AOT] = 22.2															
24.6	0.39	29.1	0.63	34.3	1.70	41.2	32.00	27.2	0.52	32.2	1.00	40.1	20.50		
25.6	0.45	29.8	0.68	35.5	2.65	41.9	44.00	28.4	0.58	33.1	1.15	40.9	27.50		
26.4	0.48	30.6	0.83	38.7	9.80	42.2	52.00								
[KCl] = 0.04 mol·dm^{-3}, [AOT] = 0.50 mol·dm^{-3}, [H$_2$O]/[AOT] = 22.2															
22.4	0.32	29.3	0.49	36.7	3.83	44.6	262.50	26.1	0.38	33.9	1.28	41.4	58.00	48.1	550.00
23.2	0.33	30.3	0.56	38.0	8.10	45.3	300.00	27.1	0.40	34.6	1.65	42.2	88.50	49.2	690.00
23.9	0.34	31.1	0.63	39.0	13.70	46.1	385.00	28.1	0.43	35.5	2.15	43.9	170.00		
24.9	0.35	32.0	0.77	40.0	25.20	47.2	470.00								
[KCl] = 0.08 mol·dm^{-3}, [AOT] = 0.50 mol·dm^{-3}, [H$_2$O]/[AOT] = 22.2															
23.6	0.33	31.0	0.47	37.3	0.95	43.7	4.85	28.2	0.41	34.1	0.64	40.6	1.75	48.0	41.00
25.1	0.36	32.1	0.50	38.1	1.02	44.8	7.90	29.0	0.42	35.1	0.69	41.6	2.30	49.3	76.00
25.9	0.37	32.9	0.53	38.9	1.15	46.1	15.30	30.0	0.44	36.1	0.76	42.6	3.15		
27.1	0.39	33.2	0.54	39.8	1.38	47.1	28.50								
[RbCl] = 0.04 mol·dm^{-3}, [AOT] = 0.50 mol·dm^{-3}, [H$_2$O]/[AOT] = 22.2															
29.0	0.43	29.0	0.43	34.4	1.04	41.0	17.00	31.6	0.58	32.3	0.63	37.6	2.80	45.5	195.00
24.1	0.32	30.1	0.48	35.4	1.40	42.0	39.00	32.3	0.63	33.3	0.75	38.7	4.65	47.1	315.00
30.1	0.48	31.6	0.58	36.9	2.20	44.3	124.00	33.3	0.75	33.6	0.82	39.8	8.40		
[RbCl] = 0.08 mol·dm^{-3}, [AOT] = 0.50 mol·dm^{-3}, [H$_2$O]/[AOT] = 22.2															
24.1	0.35	30.7	0.44	37.8	0.79	45.4	20.00	28.0	0.39	34.6	0.56	42.5	2.52	49.5	195.00
25.0	0.36	31.7	0.46	38.4	0.95	46.2	31.50	28.8	0.40	35.5	0.60	43.9	6.20		
25.7	0.37	32.7	0.49	39.9	1.36	47.1	50.00	29.7	0.42	36.7	0.67	44.9	12.80		
26.6	0.38	33.5	0.52	41.0	1.70	48.4	138.00								
[CsCl] = 0.04 mol·dm^{-3}, [AOT] = 0.50 mol·dm^{-3}, [H$_2$O]/[AOT] = 22.2															
23.5	0.28	30.1	0.42	36.1	1.35	43.2	42.00	26.8	0.32	33.8	0.76	40.1	7.10	47.1	210.00
24.0	0.28	30.9	0.47	37.4	2.05	44.4	68.00	28.1	0.35	34.6	0.81	41.2	14.10		
24.8	0.29	31.5	0.51	38.1	2.75	45.1	100.00	29.5	0.40	35.3	1.10	42.2	23.50		
26.2	0.31	32.8	0.61	39.0	4.35	46.2	180.00								
[CsCl] = 0.08 mol·dm^{-3}, [AOT] = 0.50 mol·dm^{-3}, [H$_2$O]/[AOT] = 22.2															
24.2	0.35	33.6	0.55	40.9	2.35	47.9	47.00	29.6	0.44	36.6	0.93	44.4	9.30	50.0	110.00
25.2	0.37	34.5	0.59	41.7	3.15	48.3	55.00	30.6	0.46	37.6	1.11	45.2	13.80		
26.1	0.38	35.4	0.63	42.3	3.85	48.6	62.00	31.9	0.49	39.1	1.46	46.0	20.00		
28.5	0.41	36.0	0.73	43.1	5.10	49.0	73.00	32.9	0.52	39.8	1.75	46.9	29.50		
[CaCl$_2$] = 0.04 mol·dm^{-3}, [AOT] = 0.50 mol·dm^{-3}, [H$_2$O]/[AOT] = 22.2															
18.5	0.23	28.4	0.35	38.6	2.75	47.6	270.00	23.5	0.28	34.5	0.72	44.3	74.00		
20.1	0.25	30.3	0.41	40.4	6.85	48.6	342.50	25.5	0.30	36.1	1.16	45.6	130.00		
21.4	0.26	31.2	0.45	41.2	10.80	49.7	435.00	27.3	0.33	37.0	1.52	46.5	192.50		
22.4	0.27	32.8	0.56	42.3	22.50	50.2	485.00								
[NH$_4$Cl] = 0.04 mol·dm^{-3}, [AOT] = 0.50 mol·dm^{-3}, [H$_2$O]/[AOT] = 22.2															
19.3	0.24	25.8	0.34	31.9	0.72	38.0	22.50	22.8	0.27	28.8	0.46	35.3	3.65	41.0	127.00
20.4	0.25	26.4	0.35	32.7	1.04	39.1	46.00	23.5	0.28	29.9	0.55	36.2	6.00	42.0	185.00
21.3	0.26	27.8	0.41	34.1	1.95	40.1	84.00	24.7	0.31	30.7	0.63	37.3	12.40		
[LiClO$_4$] = 0.04 mol·dm^{-3}, [AOT] = 0.50 mol·dm^{-3}, [H$_2$O]/[AOT] = 22.2															
19.3	0.22	27.3	0.38	36.6	17.40	43.2	330.00	23.8	0.27	32.5	1.70	40.3	130.00	47.1	740.00
20.8	0.23	28.6	0.48	37.5	30.50	44.1	415.00	25.3	0.30	34.6	4.40	41.2	170.00		
21.9	0.24	30.0	0.64	38.3	49.50	45.1	525.00	26.5	0.34	35.9	9.80	42.2	245.00		
23.0	0.25	30.7	0.79	39.2	75.00	46.2	620.00								
[LiBr] = 0.04 mol·dm^{-3}, [AOT] = 0.50 mol·dm^{-3}, [H$_2$O]/[AOT] = 22.2															
25.4	0.22	31.4	0.79	38.6	100.00	43.4	470.00	29.3	0.39	35.3	5.70	41.1	195.00	46.5	830.00
26.2	0.24	32.5	1.17	39.1	112.00	44.8	530.00	30.5	0.54	37.6	36.00	42.3	275.00	47.6	940.00
28.4	0.32	34.4	3.15	40.2	145.00	45.5	660.00								
[KI] = 0.04 mol·dm^{-3}, [AOT] = 0.50 mol·dm^{-3}, [H$_2$O]/[AOT] = 22.2															
19.3	0.24	30.0	0.39	38.5	2.20	46.1	145.00	26.7	0.31	34.1	0.65	43.1	36.00		
20.9	0.25	31.1	0.43	40.1	5.80	47.2	215.00	27.4	0.32	35.2	0.78	44.1	66.00		
23.4	0.27	32.1	0.48	41.1	10.90	48.2	295.00	29.0	0.35	36.2	0.98	45.0	104.00		
24.7	0.28	33.6	0.60	42.2	19.50	49.1	385.00								

the other salts considered) and to Ca^{2+} being divalent (whereas all the other cations are monovalent).

The concentrations of salt required to produce the above effects on t_p are very much smaller than the Na$^+$ concen-

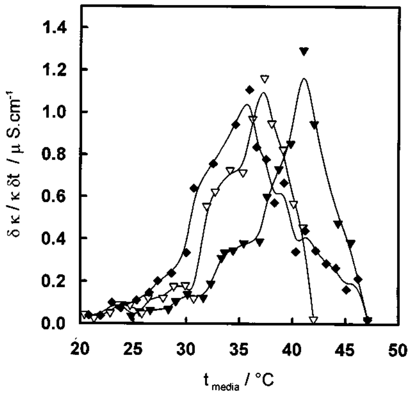

Figure 4. Determination of percolation temperature obtained by the Kim method (Kim and Huang, 1986), for bis(2-ethylhexyl)-sulfosuccinate (AOT) + 2,2,4-trimethylpentane + water microemulsions in the presence of lithium perchlorate, [AOT] = 0.50 mol·dm^{-3}, $W = [H_2O]/[AOT] = 22.2$: (▼) [CsCl] = 0.04 mol·dm^{-3}; (▽) [NH_4Cl] = 0.04 mol·dm^{-3}; (◇) [$LiClO_4$] = 0.04 mol·dm^{-3}.

Table 2. Fitting Parameters (Eq 1) and Percolation Temperature t_p Obtained by the Kim Method, for Sodium Bis(2-ethylhexyl)sulfosuccinate (AOT) + 2,2,4-Trimethylpentane + Water Microemulsions

additive	$[H_2O]/[AOT]$	C/mol·dm^{-3}	A	B	C	t_p
none	22.20		32.60	0.39	−3.30	33.0
LiCl	22.20	0.040	36.15	0.48	−4.12	37.0
LiCl	22.20	0.080	36.73	0.77	−4.97	37.1
NaCl[a]	22.20	0.040	37.35	0.47	−4.99	38.0
NaCl[a]	22.20	0.080	42.46	0.55	−6.56	45.0
KCl	22.20	0.040	37.47	0.45	−4.68	38.5
KCl	22.20	0.080	44.11	0.60	−6.73	46.6
RbCl	22.20	0.040	38.94	0.47	−4.70	40.1
RbCl	22.20	0.080	44.21	0.37	−6.78	47.8
CsCl	22.20	0.040	39.09	0.57	−4.17	40.5
CsCl	22.20	0.080	43.03	0.70	−6.17	43.5
$CaCl_2$	22.20	0.040	40.48	0.44	−4.84	41.8
NH_4Cl	22.20	0.040	36.15	0.43	−3.76	37.6
LiBr	22.20	0.040	36.45	0.44	−2.08	38.1
NaBr[a]	22.20	0.040	38.10	0.48	−4.20	39.0
NaI[a]	22.20	0.040	40.73	0.44	−4.53	39.0
KI	22.20	0.040	40.73	0.43	−4.65	38.5
$LiClO_4$	22.20	0.040	34.84	0.46	−3.16	35.7
$NaClO_4$[a]	22.20	0.040	35.97	0.49	−3.87	38.0
$NaClO_4$[a]	22.20	0.080	40.68	0.59	−5.41	41.0
$NaNO_3$[a]	22.20	0.040	35.60	0.49	−3.69	37.0
NaSCN[a]	22.20	0.040	36.64	0.45	−3.66	37.0
Na_2SO_4[a]	22.20	0.040	38.55	0.51	−4.20	40.0

[a] In: Alvarez et al. *J. Chem. Eng. Data* **1998**, *43*, 519−522.

trations from the AOT. For example, a [CsCl]/[AOT] ratio of only 1/62.5 (CsCl and AOT concentrations of 8 × 10^{-3} and 0.5 mol·dm^{-3}, respectively, both referred to the whole microemulsion) is sufficient to raise t_p by 7 °C (Table 2). This shows not only that the high ion concentration in the droplets is not sufficient to "saturate" the interface but also that alteration of the environment of a small fraction of head groups gives rise to significant changes in the case with which interdroplet channels can be opened for ion transport.

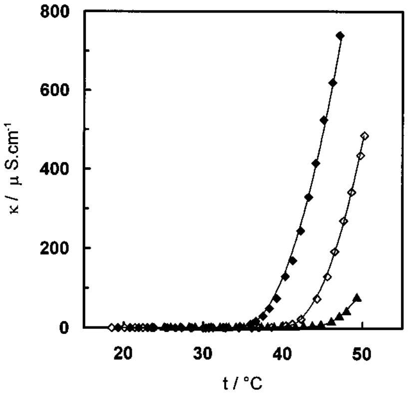

Figure 5. Fit of temperature−conductivity of AOT (sodium bis-(2-ethylhexyl)sulfosuccinate) + 2,2,4-trimethylpentane + water microemulsions to eq 1 in the presence of different additive concentrations ([AOT] = 0.50 mol·dm^{-3}, $[H_2O]/[AOT]=22.2$): (—) calculated from eq 1; (◇) [$CaCl_2$] = 0.04 mol·dm^{-3}; (◆) [$LiClO_4$] = 0.04 mol·dm^{-3}; (▲) [KCl] = 0.08 mol·dm^{-3}.

The possibility that the effects of salts on t_p are due to their altering the properties of the water (viscosity and fluidity) in the droplets, rather than to their association with AOT head groups, is ruled out by our data. Our studies include "structure-breaking ions" (Rb^+, Cs^+, I^-, and ClO_4^-), which increase the disorder of water. On the other hand, our list also includes "structure-making ions" (Li^+ and SO_4^{2-}), which enhance the icelike structure of water, inducing a greater energetic cost in the disruption of these structures. If the effect of added salts upon the percolation threshold is related to their effects on the viscosity or fluidity of the microemulsion water, we should have detected different patterns of behavior for salts of net structure-breaking effect than for the structure-making ions. The fact that this distinction is of no consequence in the observed experimental behavior confirms that the effect of electrolytes is related to their interfacial association.

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