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Influence of Linear Aliphatic Alcohols upon the Electric Percolation of AOT-Based Microemulsions

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1. Introduction

Microemulsions are dynamic structures described as spherical droplets of a dispersed phase (water) into a continuous phase (isooctane) and stabilized by a surfactant (AOT) (see Scheme 1). The components organise themselves in time and space by means of different interactions or collisions, giving rise to coalescence and redispersion processes.
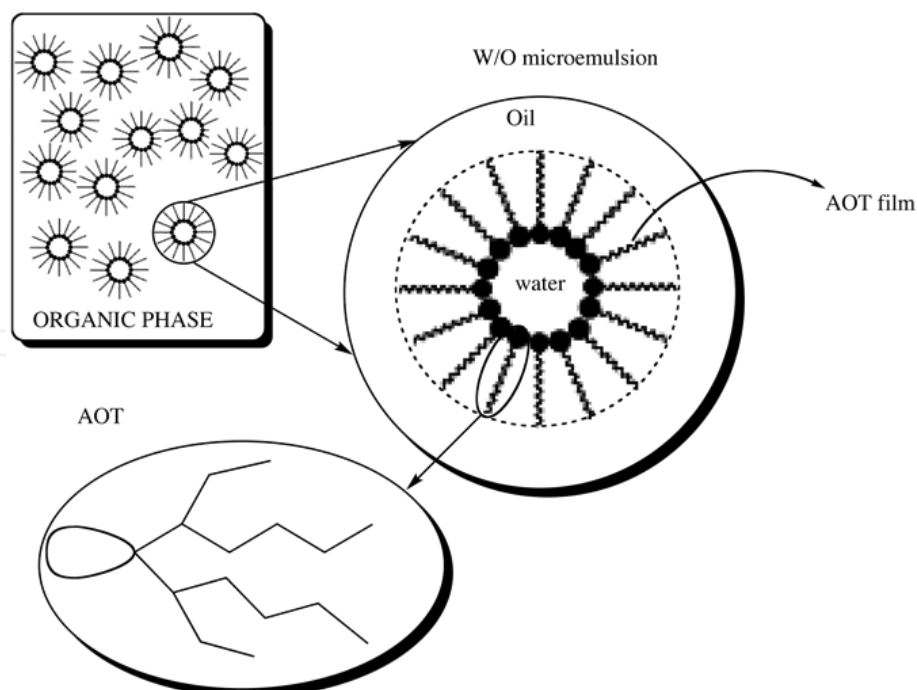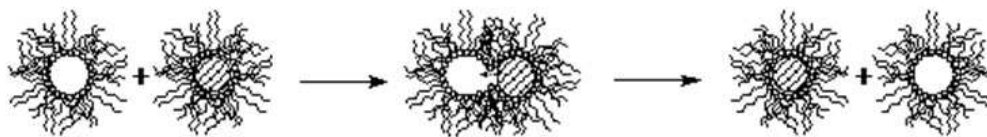

Scheme 1.

In normal conditions, they exhibit a low conductivity (0.001-0.1 mScm^{-1}) which is bigger than the corresponding one to the pure isooctane ($\sim 10^{-8}$ mScm^{-1}) (Lang et al., 1988; Eastoe et

al., 1991). This differential behaviour is justified by the fact that microemulsions are capable of carrying electric charge, and in this sense, the transport capacity is associated with a phenomenon known as electrical percolation, which is an increase in conductivity at a given value of the volume fraction of the dispersed phase, or at a certain temperature, when holding the composition of the microemulsion constant (Mathew et al., 1988; Maitra et al., 1990). This increase is associated with an increase in the electric charge flow between the discrete droplets forming the dispersed phase. Matter exchange rate between droplets increases during percolation phenomena, both ionic and neutral microemulsions; however, in all cases this rate is always lower than the diffusion control limit (Atik & Thomas, 1981; Jada et al., 1989; Jada et al., 1990; Lang et al., 1990).

The mechanism proposed to explain the electric percolation phenomenon is based on the formation of channels exchanging matter between the disperse water droplets in the continuous phase. It is then necessary to have an effective collision between at least two water droplets of the microemulsion, causing the droplets to fuse. Subsequently, an exchange of matter between the water droplets would take place (allowing the charge conduction), which would produce their separation by means of a process of fission (see scheme 2).

Scheme 2.

Moderate or low concentrations of additives have a significant effect on the percolation threshold (García-Río, L. 1994). In the literature there are a large amount of studies about the influence of different additives in general (García-Río et al., 1997; Moulik et al., 1999; Hait et al., 2001; Hait et al., 2002a; Dasilva-Carvalhal et al., 2003; Dasilva-Carvalhal et al., 2005; Dasilva-Carvalhal et al., 2006; Arias-Barros et al., 2010) and, in particular, amphiphilic additives upon the percolation phenomena and the internal dynamics of microemulsions (Nazario et al., 1996; Nazario et al., 2000; García-Río et al., 2000; García-Río et al., 2005; Cid-Samamed et al., 2008). The additives that increase the "rigidity" of the membranes increase the value of the percolation threshold of a microemulsion, (Ray et al., 1993), while those that make them more flexible favour the process (Mathew et al., 1988; Ray et al., 1993). In this sense, the addition of cationic surfactants to AOT-based microemulsions causes an increase in the percolation temperature while the addition of nonconjugated bile hydroxy salts has the opposite effect (Ray et al., 1993).

Regarding the influence of amphiphilic additives upon the electrical percolation of AOT-based microemulsions, previous studies in our laboratory have examined the influence of n-alkyl amines (García-Río et al., 2000) which exhibit a linear dependence of the percolation temperature with the amine chain length, and therefore with the hydrophobicity of the amines. However, the influence of sodium alkyl sulfonates on the electrical percolation of AOT-based microemulsions exhibit a bimodal behaviour according to which a group of alkyl sulfonates increase the percolation temperature (C_3-C_5), only to subsequently decrease it (C_6-C_{18}) (García-Río et al., 2005)

In the present chapter, the analysis of the influence of 1-n-alcohols and 2-n-alcohols upon the electric percolation of AOT/isooctane/water microemulsions is reported.

2. Experimental procedure

2.1 Materials and methods

AOT was supplied by Aldrich (purity 98%), prior to use it was kept in a vacuum desiccator for two days to minimize the water content due to its hygroscopic nature. It was used without any further manipulation. Fluka and Sigma-Aldrich supplied additives, with highest purity commercially available (between 97-99%) were employed.

Microemulsions were made "*in situ*": once microemulsion was prepared by mixing of its three components (water, isooctane, AOT), an adequate amount of organic additive was added (determined by weighing), to obtain the desired concentration of additive. The solution was stirred and heated to achieve a homogenous and isotropic sample. Then the conductivity of samples were measured as a function of temperature. In this study, composition of microemulsions was kept constant, being the values of [AOT] equal to 0.5 M (referred to total microemulsion volume), and $W=[AOT]/[H_2O]=22.2$. Water used for microemulsions was distilled-deionized, with a conductivity value around 0.10-0.15 $\mu S \cdot cm^{-1}$.

Microemulsions were introduced in 50 mL containers and were properly sealed up for thermostatization. A Teflon bar with magnetic core was used for stirring of the system to maintain a homogenous medium; the system was properly sealed with a lid with two openings, one of them to introduce an electrode and the other one to insert a thermometer to determine the electric conductivity and the temperature of the sample in each moment. The cell with the sample was immersed in a mixed ethanol-water bath, and temperature was measured simultaneously with electrical conductivity. A scheme of the experimental system is shown in the Figure 1.
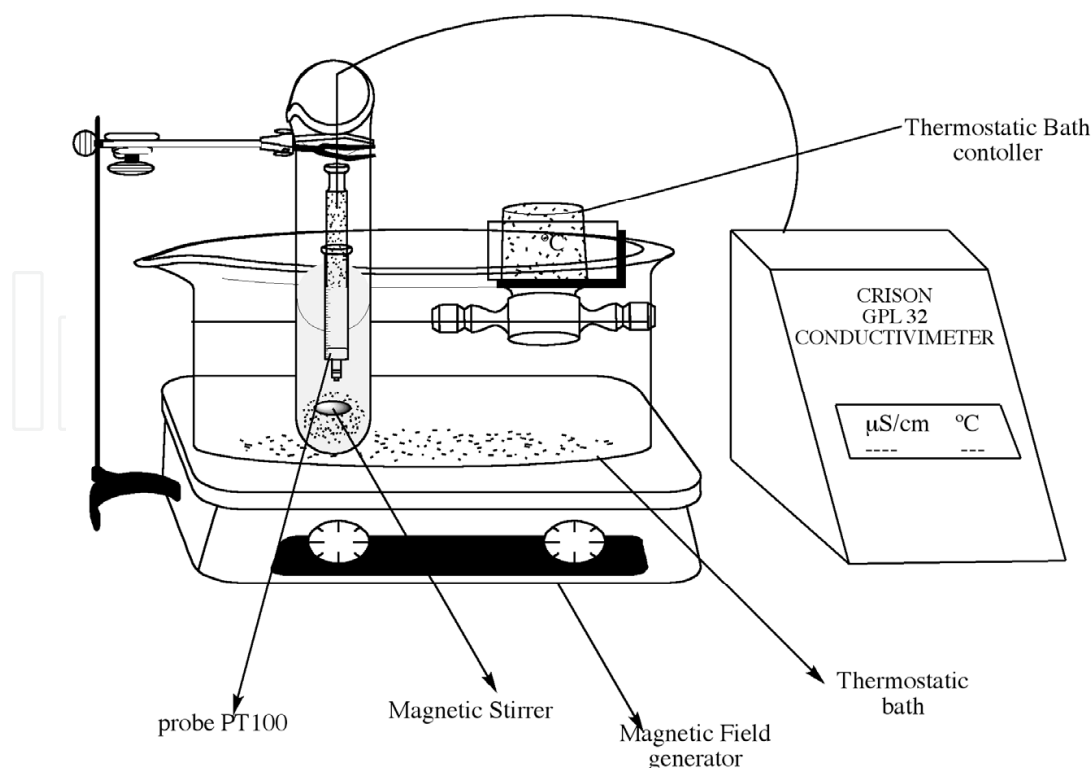

Fig. 1. Experimental equipment

The electrical conductivity was measured using a Crison GPL 32 conductiometer with a cell constant of 0.0109 cm^{-1}. The conductiometer was calibrated using standard solutions of KCl supplied by the apparatus manufacturer with following concentration and specific conductivity: [KCl]=0.0100 mol dm^{-3}, κ=1.143×10^{-3}Ω^{-1}cm^{-1} (κ=1143µScm^{-1}) at 25 $^{\circ}$C and [KCl]=0.100mol dm^{-3}, κ=1.288×10^{-2} Ω^{-1}cm^{-1}(κ=12.88 mScm^{-1}) at 25 $^{\circ}$C. Conductivity measurements were carried out using a thermostatic-cryostatic Teche TE-8D RB-5 to control the medium temperature, achieving an accuracy of ±0.1 $^{\circ}$C.

Percolation temperature has been obtained experimentally, observing the variation of conductivity with temperature (Figure 2) (Kim & Huang, 1986). Percolation threshold was obtained by analyzing the trace conductivity/temperature using the Boltzmann Sigmoidal Equation (BSE) proposed by Moulik (Hait et al., 2002b).
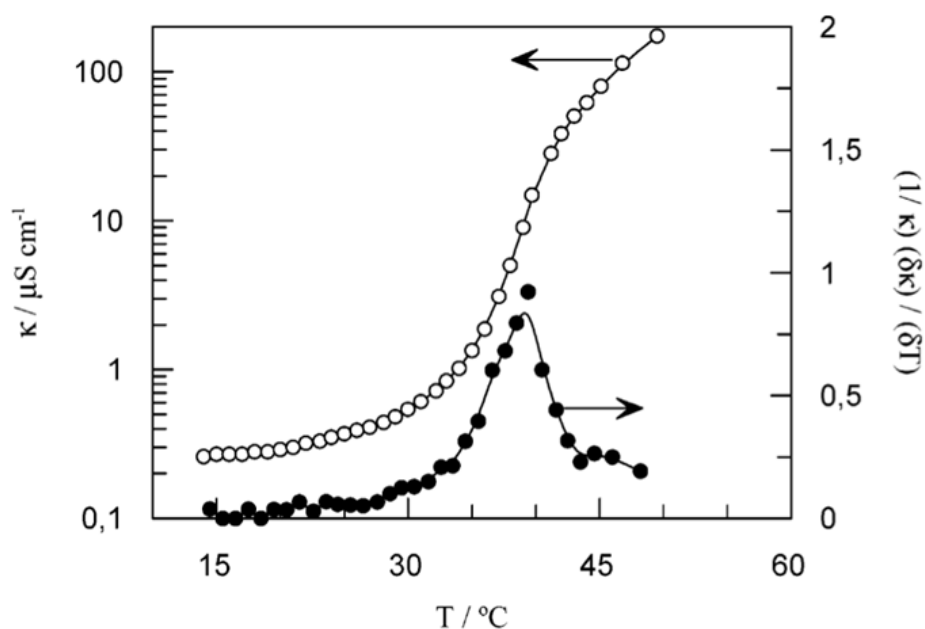
Fig. 2. (O) Determination of percolation temperature, T_p, by Moulik and Papelu procedure (Hait et al., 2002b). Solid line fits the adjustment of experimental data to SBE equation. (●) Determination of percolation temperature, T_p, by Kim and Huang procedure (Kim & Huang, 1986). In both cases adjustments correspond to w/o de AOT/iC$_8$/H$_2$O microemulsions ([AOT]=0.5M, W=22).
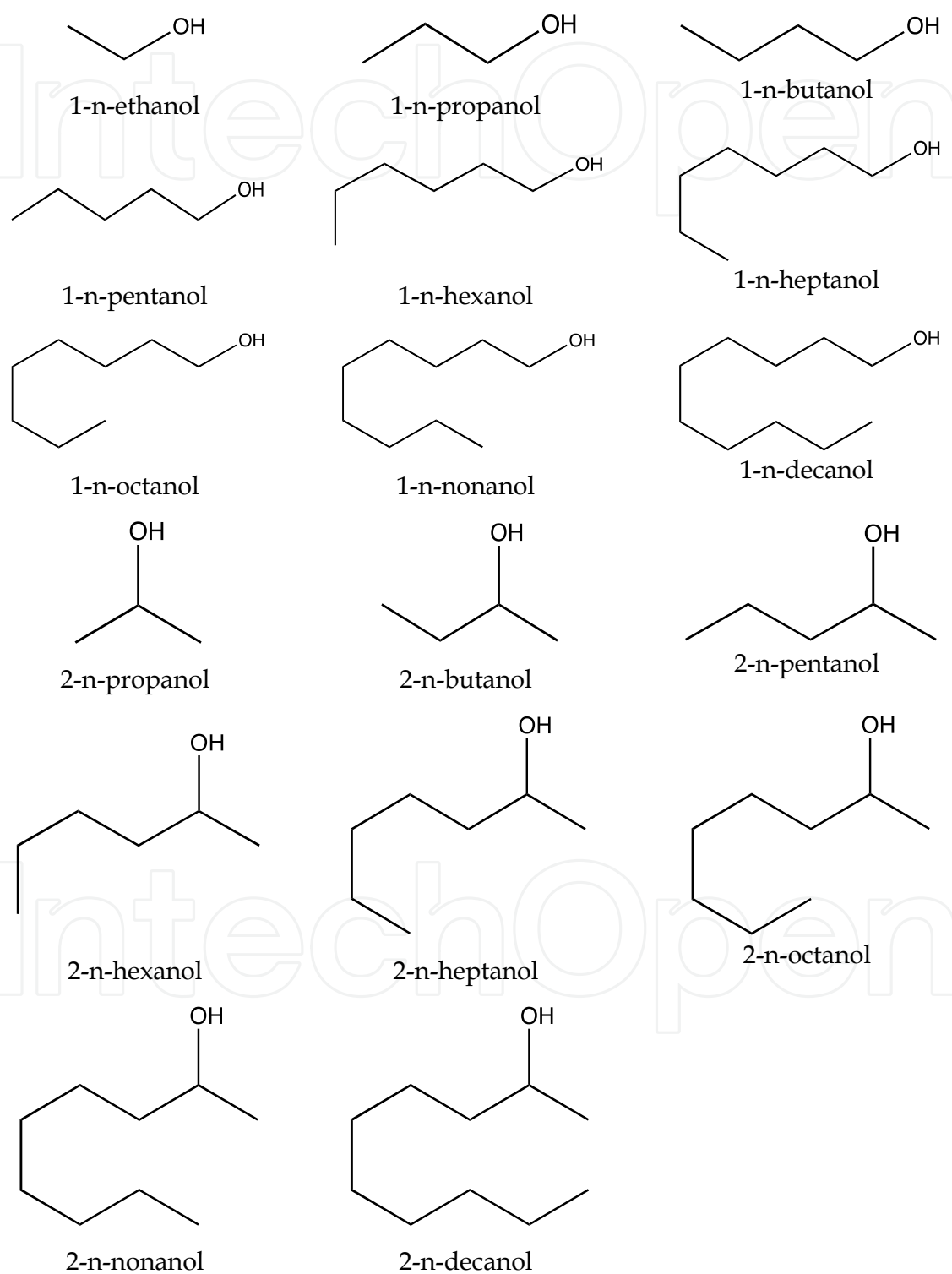
Determinations of maximum water capacity of microemulsion have been carried out by addition of appropriate volumes of water to sample with well-known AOT and isooctane quantities under agitation, and until permanent turbidity was observed. The samples thus prepared were stored at 25 $^{\circ}$C.

3. Results

3.1 Geometry of additives

The geometry of different additives employed in our study were optimized by MM2 (molecular mechanics), using a commercial program, CS ChemBats3D Pro 4.0, supplied by Cambridge Soft Corporation based on QCPE 395 (Burkert & Allinger, 1982; Allinger, 1977).

Chemical structures, of 1-n-alcohols and 2-n-alcohols studied, are shown in Scheme 3. Table 1 shows the values of chain length and head group area for each additive obtained from MM2 calculations.

1-n-ethanol   1-n-propanol   1-n-butanol

1-n-pentanol   1-n-hexanol   1-n-heptanol

1-n-octanol   1-n-nonanol   1-n-decanol

2-n-propanol   2-n-butanol   2-n-pentanol

2-n-hexanol   2-n-heptanol   2-n-octanol

2-n-nonanol   2-n-decanol

Scheme 3.

Additive	1-n-Alcohol	Number of C atoms	a_0 / $\breve{A}^2$	l_c / $\breve{A}$
	1-n-Ethanol	$1C_2$		2.16
	1-n-Propanol	$1C_3$		3.48
	1-n-Butanol	$1C_4$		4.84
	1-n-Pentanol	$1C_5$		6.14
	1-n-Hexanol	$1C_6$	1.34	7.30
	1-n-Heptanol	$1C_7$		8.70
	1-n-Octanol	$1C_8$		9.40
	1-n-Nonanol	$1C_9$		11.20
1-n-Alkanols	1-n-Decanol	$1C_{10}$		12.35
	2-n-Propanol	$2C_3$		2.16
	2-n-Butanol	$2C_4$		3.48
	2-n-Pentanol	$2C_5$		4.84
	2-n-Hexanol	$2C_6$	1.86	6.14
	2-n-Heptanol	$2C_7$		7.30
	2-n-Octanol	$2C_8$		8.70
	2-n-Nonanol	$2C_9$		9.40
2-n-Alkanols	2-n-Decanol	$2C_{10}$		11.20

Table 1. Values of critical length of chain and head group area obtained from MM2 calculations for each of the amphiphiles used as additives.

3.2 Influence of 1-n-alcohols upon the electric percolation

The effect of several 1-n-alcohols upon percolation phenomenon has been reported. The addition of these 1-n-alcohols (1-n-ethanol [$1C_2$], 1-n-propanol [$1C_3$], 1-n-butanol [$1C_4$], 1-n-pentanol [$1C_5$], 1-n-hexanol [$1C_6$], 1-n-heptanol [$1C_7$], 1-n-octanol [$1C_8$], 1-n-nonanol [$1C_9$], 1-n-decanol [$1C_{10}$]) to AOT/alkane/water microemulsions has been studied, analyzing the effect of additives concentration on percolation temperature. Chemical structures of these compounds are shown in Scheme 3.

Concentrations used were [C_n]= 0.05, 0.1, 0.25 and 0.5 M referred all of them to the water volume of the microemulsion. In some cases the concentrations range were not completed because of the addition of this additive concentration implies microemulsions destabilization. The percolation temperature values of each specific case were obtained keeping constant the composition of the microemulsion ([AOT]=0.5M and W= 22.2) and varying the additive concentration, as detailed in the experimental procedure section.

In the following figures 3 to 6 the effect of above-mentioned concentrations of 1-n-alcohols can be observed. As we can see in Figures 3 and 4, the observed behaviour of 1-n-alcohols upon the percolation temperature presents a decrease in percolation temperature when increasing the additive concentration studied, it occurs for alcohols which the number of carbon atoms of the alcohol studied is lower than 3. By contrast, the behaviour would be reversed when the alcohol studied has a number of carbon atoms higher than 3, as we quote in Figures 5 and 6. Numerical values of percolation temperature obtained for each alcohol and each concentration are listed in Table 2.
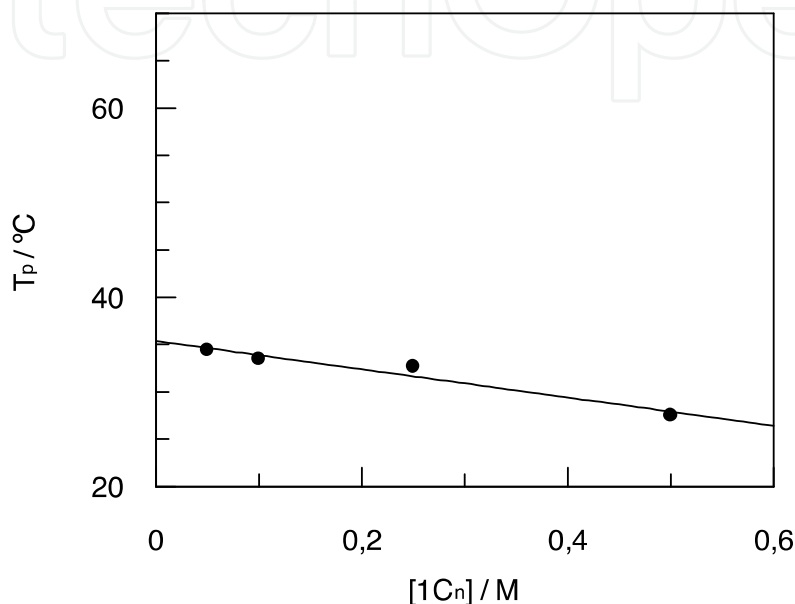

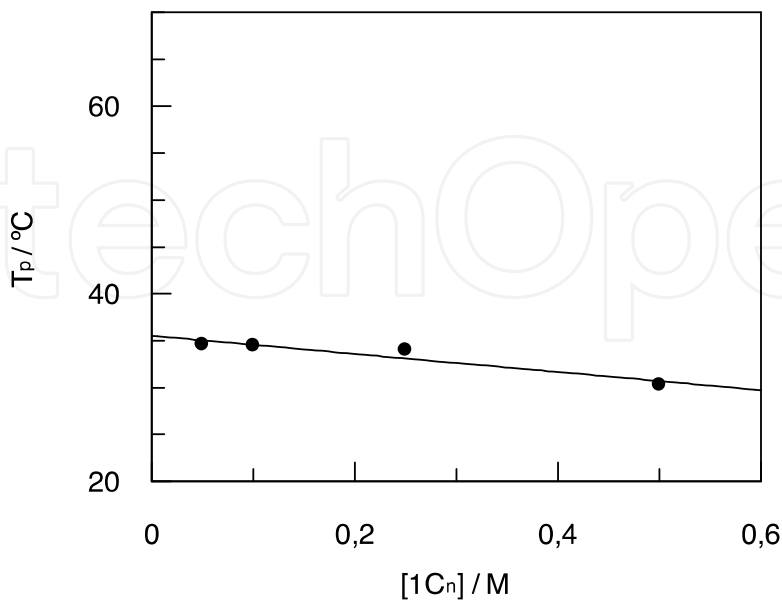Fig. 3. Influence of $1C_2$ upon electric percolation temperature of AOT/iC$_8$/H$_2$O microemulsions

Fig. 4. Influence of $1C_5$ upon electric percolation temperature of AOT/iC$_8$/H$_2$O microemulsions with [AOT]= 0.5 M and W= 22.2
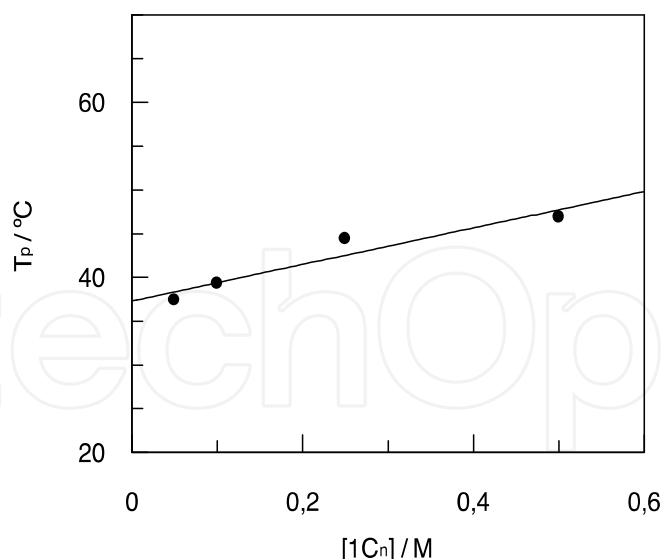
Fig. 5. Influence of $1C_7$ upon electric percolation temperature of AOT/iC_8/H$_2$O microemulsions with [AOT]= 0.5 M and W= 22.2
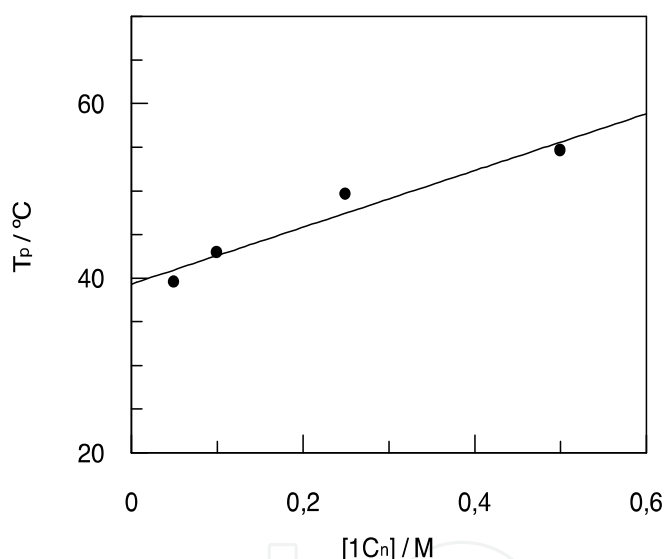

Fig. 6. Influence of $1C_{10}$ upon electric percolation temperature of AOT/iC_8/H$_2$O microemulsions with [AOT]= 0.5 M and W= 22.2

3.3 Influence of 2-n-alcohols upon the electric percolation

As in the previous section, the effect produced by different 2-n-alcohols (2-n-propanol [$2C_3$], 2-n-butanol [$2C_4$], 2-n-pentanol [$2C_5$], 2-n-hexanol [$2C_6$], 2-n-heptanol [$2C_7$], 2-n-octanol [$2C_8$], 2-n-nonanol [$2C_9$], 2-n-decanol [$2C_{10}$]) to AOT/alkane/water microemulsions upon percolation have been analyzed. Chemical structures of these additives are shown in Scheme 3.

In this case, additive concentrations used were [C_n]= 0.05, 0.1, 0.25 and 0.5 M referred all of them to the water volume of the microemulsion. As quote above, in some cases the concentration ranges were not completed. In this case, an increase in percolation temperature as a function of chain length of the alcohol is observed for all alcohols and all concentration ranges (Table 2).

It must be underlined that this behaviour contrasts with the one that is observed in the case of 1-n-alcohols (vide supra), where there is not a decrease of the percolation temperature with increasing of additive concentration for chain length values lower than 3.

4. Discussion

4.1 Influence of concentration upon electric percolation

In Table 2 we present the values obtained for electric percolation temperature of AOT/iC_8/water microemulsions in the presence of each alcohol. In Figures 7 and 8 the influence of the concentration of $1C_n$ and $2C_n$ on the percolation temperature AOT/iC_8/water microemulsions are depicted. A linear trend can be observed in percolation temperature with the concentration of additives such as 1-n-alcohols and 2-n-alcohols. A comparison of these two graphs allows us to report a different behaviour between 1-n and 2- n-alcohols,
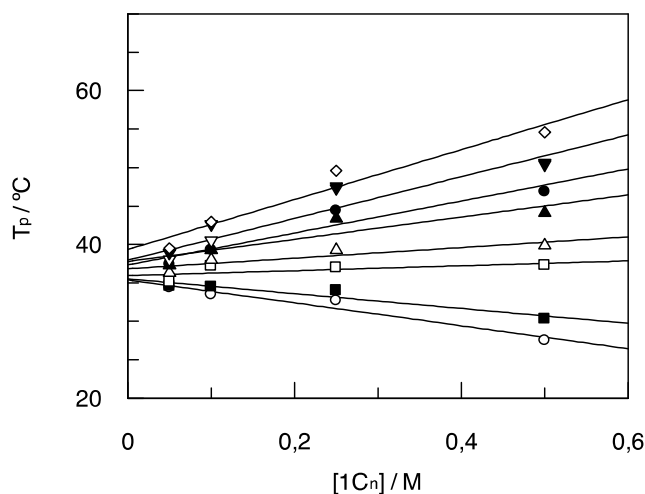

Fig. 7. Influence of the concentration of $1C_n$ on the percolation temperature AOT/iC_8/water microemulsions. [AOT] = 0.5 M, W = 22.2.
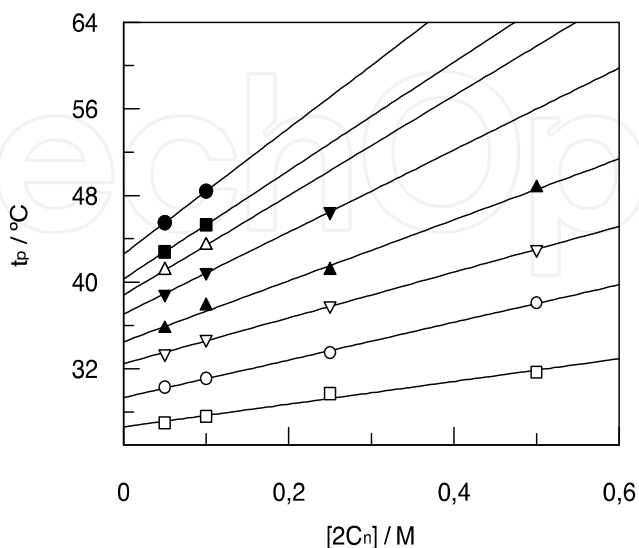

Fig. 8. Influence of the concentration of $2C_n$ on the percolation temperature AOT/iC_8/water microemulsions. [AOT] = 0.5 M, W = 22.2.

although in them, the slope of the growing dependence occurs by increasing the value of n. In both cases, as it was reported, the percolation temperature increases linearly with additives concentration. However, in the case of 1-n-alcohols, and for values of n<3 a negative slope it is observed, i.e. increasing alcohol concentration is a decrease in temperature of percolation (Figure 7). For values of n=3 the percolation temperature is almost independent of the chain length (Figure 7), ultimately for values of n>3 the behaviour is completely analogous between 1-n and 2-n-alcochols (Figure 7). In the case of 2-n-alcohols, an increase in percolation temperature as a function of chain length of the alcohol is observed at a constant additive concentration (Figure 8). This behaviour contrasts with the one that is observed in the case of 1-n-alcohols (Figure 8), where there is not a decrease of the percolation temperature with increasing of additive concentration for chain length values lower than 3. This behaviour would be associated with the polarity of the molecules and hence to the different distributions among the three different domains of the microheterogeneous system and their insertion at the interface as a co-surfactant.

$1C_n$	$[1C_n]$ / M	T_p / ^oC
$1C_2$	0.05	34.4
	0.10	33.5
	0.25	32.7
	0.50	27.5
$1C_3$	0.05	34.6
	0.10	34.5
	0.25	34.0
	0.50	30.3
$1C_4$	0.05	35.1
	0.10	37.2
	0.25	37.0
	0.50	37.3
$1C_5$	0.05	36.3
	0.10	38.0
	0.25	39.3
	0.50	39.9
$1C_6$	0.05	37.3
	0.10	39.3
	0.25	43.4
	0.50	44.1
$1C_7$	0.05	37.4
	0.10	39.3
	0.25	44.4
	0.50	46.9
$1C_8$	0.05	38.0
	0.10	40.5
	0.25	47.3
	0.50	50.4

$1C_n$	$[1C_n]$ / M	T_p / °C
$1C_9$	0.05	38.9
	0.10	42.6
	0.25	47.5
	0.50	50.6
$1C_{10}$	0.05	39.5
	0.10	42.9
	0.25	49.6
	0.50	54.6
$2C_n$	$[2C_n]$ / M	T_p / °C
$2C_3$	0.05	27.0
	0.10	27.6
	0.25	29.7
	0.50	31.7
$2C_4$	0.05	30.3
	0.10	31.1
	0.25	33.5
	0.50	38.1
$2C_5$	0.05	33.4
	0.10	34.7
	0.25	37.8
	0.50	43.0
$2C_6$	0.05	35.7
	0.10	37.8
	0.25	41.1
	0.50	48.7
$2C_7$	0.05	38.9
	0.10	40.9
	0.25	46.5
	0.50	m/r
$2C_8$	0.05	41.1
	0.10	43.4
	0.25	m/r
	0.50	m/r
$2C_9$	0.05	42.8
	0.10	45.3
	0.25	m/r
	0.50	m/r
$2C_{10}$	0.05	45.5
	0.10	48.4
	0.25	m/r
	0.50	m/r

m/r: no stable microemulsion

Table 2. Continued

4.2 Influence of chain length upon electric percolation

In Figure 9 one can see the effect exerted by the hydrocarbon chain length of the 1-n-alcohols upon percolation temperature at a constant concentration of additive. Similar behaviour has been found for 2-n-alcohol. Taking into account the obtained results we highlight two aspects, the first is the fact that the slope of the experimental curve of t_p *vs* n grows with increasing additive concentration. This result is consistent with the one obtained when studying the effect of additive concentration upon the percolation threshold for each one of the alcohols studied.
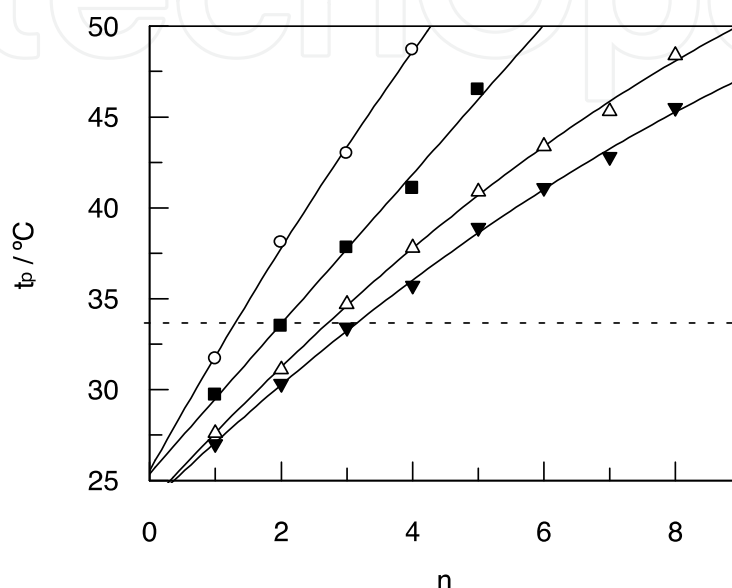
Fig. 9. Influence of *n* (number of carbon atoms) upon t_p in the addition of $[1C_n]$ to $AOT/iC_8/H_2O$ microemulsions [AOT]= 0.5 M, W= 22. [1Cn]=0.05 M, [1Cn]=0.1 M, [1Cn]=0.25 M & [1Cn]=0.50M

This behaviour observed would be due to the solubilisation of the alcohol between the interface and the aqueous phase in the case of low concentrations and low chain lengths; the majority solubilisation of the alcohol at the interface in the case of moderate concentrations of additive and moderate and high chain lengths; and the partial solubilisation of the alcohol at the organic phase in the case of high concentrations.

4.3 Comparison of results

As quote above, a different behaviour between both alcohol families was reported. In both cases as it was reported the percolation temperature increases linearly with additives concentration and the growing dependence of percolation temperature with alcohol concentration occurs by increasing the value of n. However, in the case of 1-n-alcohols, there are three different behaviours as a function of alcohol chain length (for n<3 implies a negative slope, for values of n=3 the percolation temperature is almost independent of the chain length and, for n>3 the slope is positive).

This behaviour would be associated with the polarity of the molecules and hence to the different distributions among the three phases of the microemulsion and their insertion at the

interface as a co-surfactant. In this sense to rationalize this behaviour, it is necessary to know the polarity of these molecules. The values of the logarithm of the partition coefficient between 1-n-octanol and water of these molecules are shown in Table 5. This parameter is accepted as a plausible way to quantify the polarity of a molecule. This parameter is often accepted as a plausible way to quantify the polarity of a molecule. The observed differences in polarity between 2-n-alcohol and 1-n-alcohols can be noticed for example for a chain length of n=2, 2-n-propanol has a logP=016 while the 1-n-etanol its analogue has a logP=- 0.19.

Other point that must be underlined is the fact that there are two different head-groups with different head-group area, because the 1-n-alcohols would present a head-group area of 5.64 $Å^2$, versus 10.86 $Å^2$ head-group area of the 2-n-alcohols. Thus, the correlation between the logP values and the effect exerted on percolation temperature of the system is shown in Figure 10. Analogous correlation ship can be plotted for 2-n-alcohols.

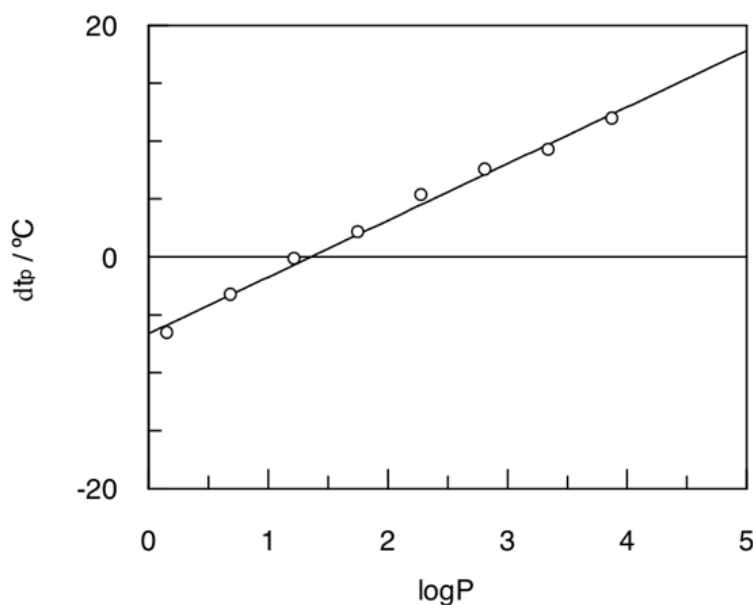| $2C_n$	Log P
$2C_3$	0.160
$2C_4$	0.691
$2C_5$	1.223
$2C_6$	1.754
$2C_7$	2.285
$2C_8$	2.817
$2C_9$	3.348
$2C_{10}$	3.879

Table 3. logP values for several used 2-n-alcohols

Fig. 10. Influence of hydrocarbon chain length of 2-n-alcohol upon the percolation temperature of AOT/iC$_8$/H$_2$O microemulsions. [AOT]= 0.5 M and W= 22.2, [2Cn]= 0.05 $dt_p = t_p^{additive} - t_p$

5. Conclusions

The investigation of the behaviour of 1-n-alcohols on the percolation temperature showed that for the values of n<3 the percolation temperature decreases with increasing the concentration of alcohol added, while, on the contrary, values of n>3 the behaviour would be reversed.

In the same way we have proceeded to study the behaviour of 2-n-alcohols on percolation temperature, obtaining different effects: at low concentrations and short chain lengths we observed an advancement of the percolation temperature, while alcohols with moderate and long-chain lengths showed that there is a delay in the temperature of percolation. This behaviour is clearly different to the results observed for 1-n-alcohols

It must be remarked that the behaviour observed for these two families of additives is quite different to the corresponding one of other amphiphilic molecules studied previously in our research team, as n-alkyl amines (García-Río et al., 2000), sodium alkyl-sulphates (García-Río et al., 2005) or n-alkyl acids (Cid-Samamed et al., 2008)

The behaviour of these studied systems is justified differently depending on the polarity of the molecules and the differences in solubility of the same in the various domains of microemulsions: the solubilisation of the alcohol between the interface and the aqueous phase at low concentrations and low chain lengths; solubilisation of the alcohol takes place mainly at the interface for moderate concentrations of additive and moderate and high chain lengths; and the partial solubilisation of the alcohol at the organic phase in the case of high additive concentrations.

6. Acknowledgment

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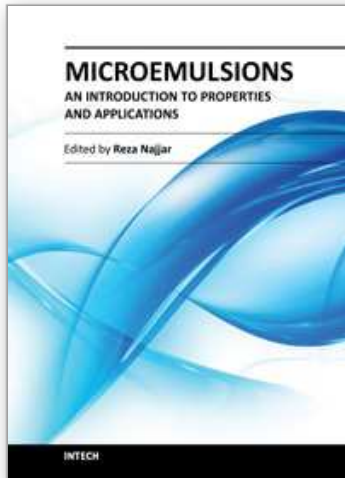
Microemulsions - An Introduction to Properties and Applications

Edited by Dr. Reza Najjar

The rapidly increasing number of applications for microemulsions has kept this relatively old topic still at the top point of research themes. This book provides an assessment of some issues influencing the characteristics and performance of the microemulsions, as well as their main types of applications. In chapter 1 a short introduction about the background, various aspects and applications of microemulsions is given. In Part 2 some experimental and modeling investigations on microstructure and phase behavior of these systems have been discussed. The last two parts of book is devoted to discussion on different types of microemulsion's applications, namely, use in drug delivery, vaccines, oil industry, preparation of nanostructured polymeric, metallic and metal oxides materials for different applications.

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