

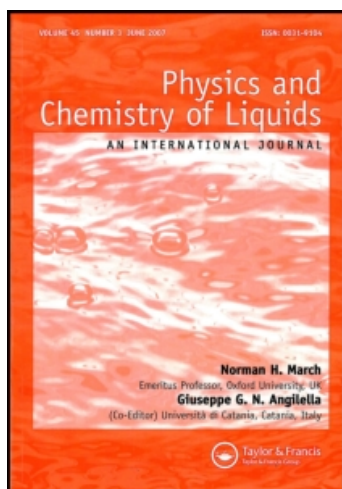
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D. H. Kurlat<sup>a</sup>; D. Zadunaisky<sup>a</sup>; M. S. Bidner<sup>a</sup>; H. Kostiria<sup>b</sup>

<sup>a</sup> Faculty of Engineering, University of Buenos Aires, Buenos Aires, Argentina <sup>b</sup> U. N. L. P,

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## Letter

### Physical Properties of Microemulsions

D. H. KURLAT, D. ZADUNAISKY, M. S. BIDNER

*Faculty of Engineering, University of Buenos Aires,  
Buenos Aires, Argentina.*

and

H. KOSTIRIA

*(U.N.L.P.)*

*(Received May 24, 1985)*

We have measured the interfacial tensions, vs. oil and brine, the electric conductivity and the magnetic susceptibility of microemulsions as a function of brine concentration and temperature.

The middle phase microemulsions exhibit the lowest interfacial tension, a sharp increase in the microemulsion/brine conductivity ratio and a maximum of the (diamagnetic) susceptibility.

The possibility of a percolative process and chemical or physical changes in the middle phase are discussed.

There is currently considerable interest in the study of the properties of microemulsions composed of oil-surfactant-water (brine) and cosurfactant. It is well known that, as the water (brine) concentration is changed the microemulsion passes from an oil-in-water microstructure of a water-in-oil one. Between the two phases, an intermediate one exists, which presents ultra-low interfacial tensions. The subject of this paper is to study how the formation of this phase is correlated with sudden changes of other physical properties: electric conductivity and magnetic susceptibility. It must be remarked that as far as we know, there are no previously reported experimental data of the magnetic susceptibility of these liquid systems.

Microemulsions were obtained by mixing brine oil-(dodecylbenzene), commercial dodecylbenzene sulfonate as surfactant, and normal amyl alcohol as cosurfactant. The overall water/oil volume ratio was 1:1; the surfactant/cosurfactant ratio was 6:4, and the surfactant-cosurfactant/total volume ratio was 1:10. When the concentration of sodium chloride increases, the liquid mixture changes from a two-phase system, (microemulsion in

the lower phase and oil) to a three phase system (brine, microemulsion in the middle phase and oil) and back to a two-phase system (brine and microemulsion in the upper phase).

We have measured the interfacial tension ( $\gamma$ ) vs. oil or brine, the electric conductivity ( $\sigma$ ) and the magnetic susceptibility ( $\chi$ ) as a function of brine concentration and temperature.  $\gamma$  experimental values were obtained using a spinning drop interfacial tensiometer ((1).

The electrical conductivity was measured with an a.c. current bridge (in the low frequency range in order to avoid polarization effects. The conductance cell was immersed in a thermostatic bath where temperature fluctuations were about  $\pm 0.1^\circ\text{C}$ . We must point out for  $t(^{\circ}\text{C}) \gtrsim 32^\circ\text{C}$  the microemulsions evolved from a clear fluid dispersion to a milky one, which indicates a change of the structure from microemulsion to macroemulsion. At the same time, in as much as this temperature range is reached, it is not possible to obtain the same conductivity values which had been obtained before.

We have not performed a careful analysis of these "hysteresis-like" phenomena indicative of liquid instability.

The magnetic susceptibility of these microemulsions was measured by the Faraday method (2). The apparent weight of the samples in a magnetic field was measured using a Cahn RTL millibalance; magnetic fields up to 10 KOe with a variable pole gap (2 to 4 cms.), were applied. The samples were heated by means of an electric furnace (temperature fluctuations were  $\pm 0.5^\circ\text{C}$ ).

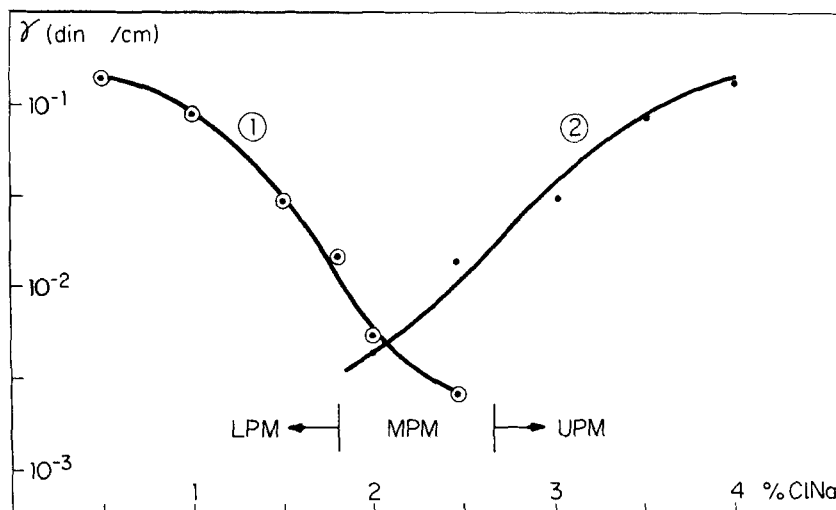


FIGURE 1a Interfacial tension of the microemulsions vs. oil (1) and vs. brine (2) at  $25^\circ\text{C}$  as a function of brine concentration (weight fraction). LPM: lower phase microemulsion; UPN: upper phase microemulsion.

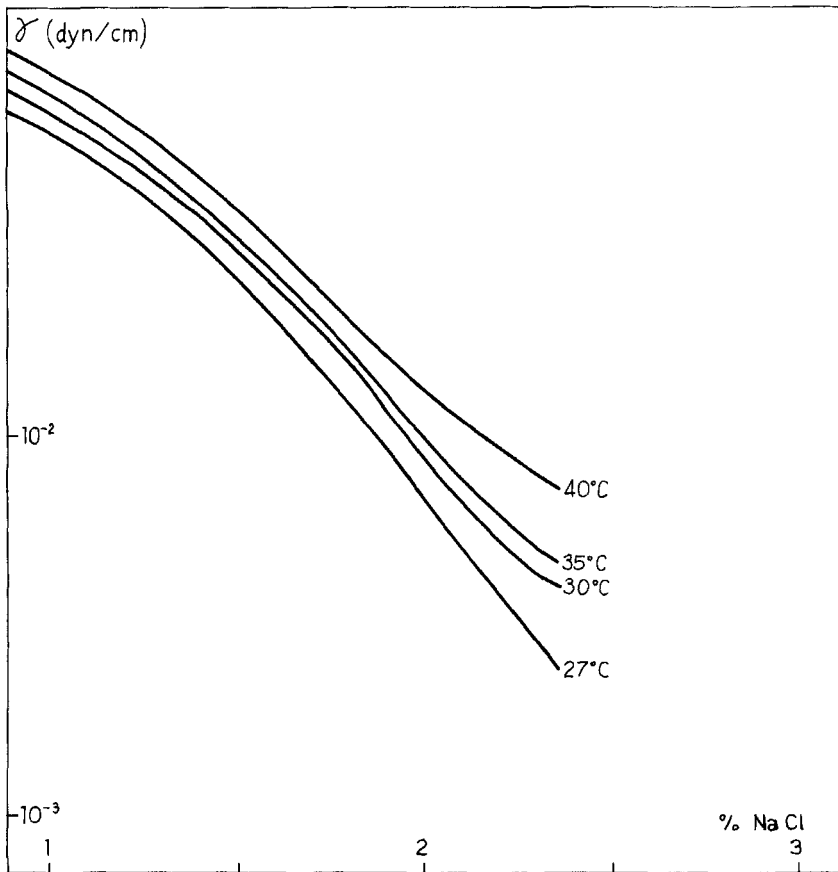


FIGURE 1b Interfacial tension of the microemulsion vs. oil as a function of brine concentration for different temperatures.

In Figure 1a, we plotted the interfacial tension of the microemulsion vs. water and oil respectively as a function of brine concentration and in Figure 1b, the former's behaviour for different temperatures. It has been suggested that, following the scaling theory of critical phenomena (3),  $\gamma \propto \Phi^s$  where  $\Phi$  is the volume fraction of brine in the microemulsion. From a log-log plot of  $\gamma$  vs.  $\Phi$  (taken from the 27°C data of Figure 1b) we have obtained  $s = 6.28$  (corr = 0.96).

The ratio of each microemulsion conductivity ( $\sigma_{M.E.}$ ) to that of the brine used to make the sample ( $\sigma_{NaCl}$ ) as a function of  $\Phi$  for  $t = 25^\circ\text{C}$  is plotted in Figure 2a. For  $\Phi < 0.4$ ,  $\sigma_{M.E.}/\sigma_{NaCl}$  is small, while for those concentrations corresponding to the middle phase microemulsion, this ratio shows a sharp increase.

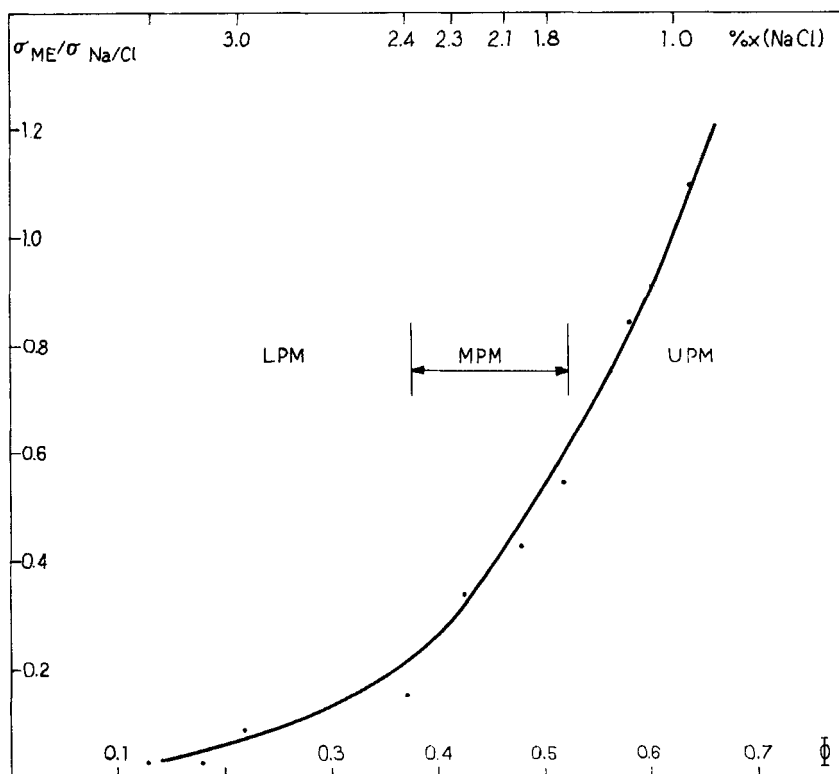


FIGURE 2a Ratio of microemulsion conductivity ( $\sigma_{M.E.}$ ) to that of the brine used to make the sample ( $\sigma_{NaCl}$ ) as a function of brine volume fraction ( $\Phi$ ) for 25°C. LPM, MPM, UPM defined for Figure 1a.

In Figure 2b we can see a linear dependence of  $\ln \sigma_{M.E.}$  as function of the inverse of absolute temperature.

We didn't observe any variation of conductivity values in the low frequency range ( $50 \text{ Hz} \leq \omega \leq 1000 \text{ Hz}$ ). The magnetic susceptibility shows a weak dependence on the concentration of the solute NaCl—see Figure 3—exhibiting a maximum of diamagnetism for the middle phase microemulsion, correlating with the relative maximum in the conductivity at the same concentration. Concerning the variations with temperature, we noted a slight increase of diamagnetism with temperature. We must say that the results lacked reproducibility for  $t(^{\circ}\text{C}) \geq 32^{\circ}\text{C}$  in agreement with the behaviour observed when measuring the electric conductivity. We also measured the magnetic susceptibility of the chemical components of the microemulsion (with a 2% of NaCl). The results are seen in Table I.

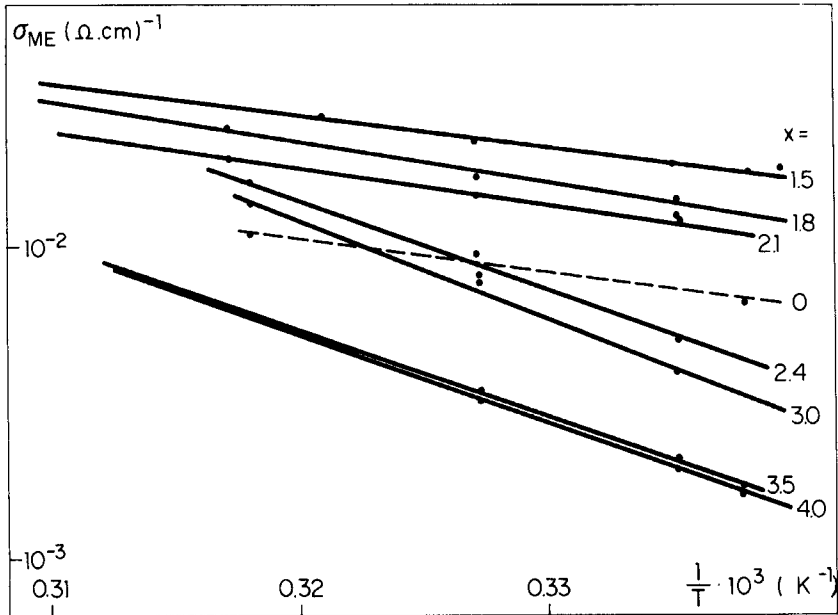


FIGURE 2b  $\ln \sigma_{M.E.}$  as a function of inverse absolute temperature, for brine concentrations,  $\times 0; 1.5; 1.8; 2.1; 2.4; 3.0; 3.5; 4.0$ .

It has been argued that, (4) (5), the sudden increase in the ratio  $\sigma_{M.E.}/\sigma_{NaCl}$  is indicative of a percolative behaviour, since for volume fractions of brine less than a critical value, ( $p_c$ ), (“percolation threshold”) there is no continuous path of brine, so that the charge carriers are isolated and cannot form an “infinite” (conductive) cluster. Strictly, this model implies a value  $\sigma = 0$ , just at the threshold percolation, and for  $p > p_c$ ,  $\sigma \neq 0$  (6). It must be pointed out that measured values of  $\sigma_{M.E.}/\sigma_{NaCl}$  vs.  $\Phi$  for the upper phase are not negligible compared to those corresponding to the middle and lower phases.

TABLE I  
Magnetic susceptibility of the chemical components of the microemulsions

Component	$\times 10^{-6}$ emu/g
H <sub>2</sub> O (with 2% ClNa)	0.77
Surfactant	0.70
Amyl alcohol	0.89
Dodecylbenzene	0.85

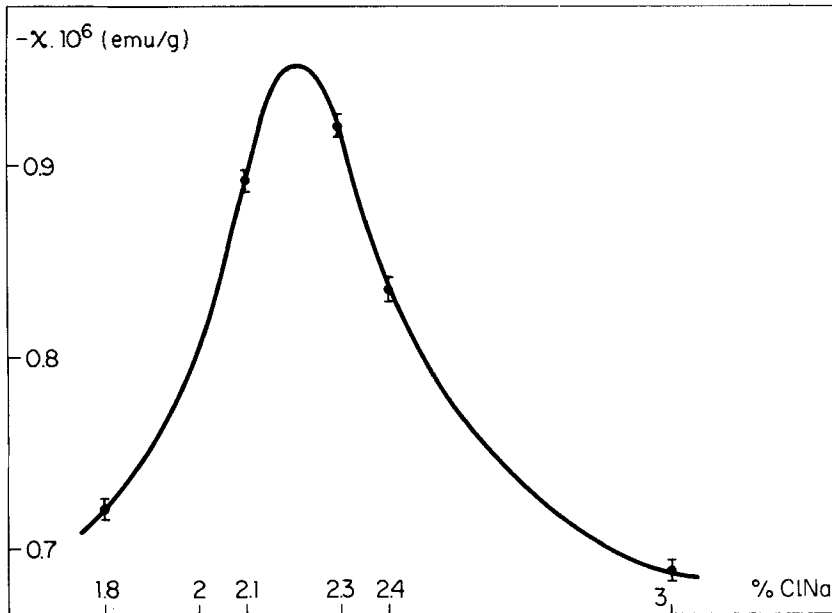


FIGURE 3 Magnetic susceptibility of microemulsions as a function of brine concentration (weight fraction).

Of course, this is an open question and following Lagües,<sup>7,8</sup> Brownian motion should be taken into account, to obtain an improvement to this theoretical approach.

Knowing the magnetic susceptibility of the chemical components of microemulsion, we can compute the susceptibility of the molecular mixture Wiedemann's additivity law.<sup>9</sup>

$$\chi = \sum_i c_i \chi_i$$

where  $C_i$ ,  $\chi_i$  are the weight fractions and the susceptibilities respectively for each component. We obtain:

$$\chi = 0.802 \times 10^{-6} \text{ e.m.u.s}$$

This result is intermediate among the experimental results for the different microemulsions. The deviations can be due either to the variation of the magnetic susceptibility of the brine with the concentration, or they can be assigned to chemical or physical changes in the mixtures.

We measured the magnetic susceptibility of these aqueous solutions for salt concentration ranging up to 3%, finding no dependence on concentra-

tion. Then the variation of the magnetic susceptibility of microemulsions with the concentration of NaCl should be associated with the structural changes they undergo in the concentration range 0–3% and which are obviously exhibited by the different place the microemulsion phase occupies in the liquid system.

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