Poster

NANO-EMULSION FORMATION IN IONIC SURFACTANT SYSTEMS BY THE PHASE INVERSION COMPOSITION METHOD (PIC): OPTIMIZATION THROUGH AN EXPERIMENTAL DESIGN

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Nano-emulsions are colloidal dispersions formed by a liquid phase dispersed in another one in form of droplets. What makes them very interesting is the extremely small droplet size they have, in the range 20-500 nm, reason why they may appear transparent or translucent. They are thermodynamically unstable systems, and consequently, their properties depend on the preparation method. The low-energy preparation methods, in which the temperature or the composition are maintained constant, make use of the phase transitions that take place during the emulsification process as a result of a change in the spontaneous curvature of the surfactant[1]. As a result, it is not necessary to apply a high mechanical energy.

The formation of nano-emulsions stabilized with non-ionic surfactants has been widely reported in the bibliography[2,3]. However, there are few studies about the preparation of nano-emulsions stabilized with ionic surfactants by low energy or "spontaneous" methods[4,5]. In this work, a method to obtain nano-emulsions in ionic surfactant systems by the phase inversion composition method is presented. The aim of this new method is the use of a fatty acid as a cosurfactant, which is progressively neutralized during the emulsification process due to the continuous addition of an alkaline watery solution, obtaining the corresponding salt, which acts as the ionic surfactant.

The ionic system chosen was the anionic system water / potassium oleate-oleic acid-$C_{12}E_{10}$ / hexadecane. Potassium hydroxide solutions were added to the mixtures formed by water, hexadecane, $C_{12}E_{10}$ and oleic acid, being this last one component partially neutralized into potassium oleate during the emulsification process. The results obtained were compared with those obtained in the cationic system water / oleylamine chloride-oleylamine-$C_{12}E_{10}$ / hexadecane. In this system, chloride acid solutions are added to the mixtures formed by water, hexadecane, $C_{12}E_{10}$ and oleylamine. The oleylamine is partially neutralized into oleylamine chloride along the emulsification paths. The final water composition was 80% w/w in all cases.

Experimental design[6] was used to optimize the composition variables oleic acid/$C_{12}E_{10}$ or oleylamine/$C_{12}E_{10}$ ratios (depending on the system) and oil-surfactants ratios, at constant preparation conditions. Nano-emulsion droplet size has been selected as the main response as it determines whether an emulsion is a nano-emulsion or not.

The results of this study show that droplet size as a function of oleic acid/$C_{12}E_{10}$ or oleyalmine/$C_{12}E_{10}$ ratios and oil-surfactants ratio, can be approximated by quadratic equations, which develop the response surfaces represented in figure 1.

It can be seen from this graphics that there is an optimal oleic acid or oleylamine/$C_{12}E_{10}$ ratio in both systems. Referring to the oil/surfactant ratio, a linear behaviour is observed; the lower the O/S ratio, the smaller the droplet size in both systems.

Trying to explain these results, the phases present through the different emulsification paths were determined at 25ºC. It has been found a cubic liquid crystalline zone with Pm3m structure, which expands to higher hexadecane/surfactants ratios and makes narrower as the oleic acid or oleylamine/$C_{12}E_{10}$ ratio increases. Reaching or not the equilibrium in this phase during the emulsion formation process allows to explain the results obtained in the experimental designs. The smallest droplet sizes are obtained when the cubic phase is wider.

Finally, it has to be said that in the system using oleylamine chloride as ionic surfactant, the droplet size of the nano-emulsions obtained is smaller than with the other system, due to the fact that the cubic phase zone is wider in the system W / oleylamine chloride - $C_{12}E_{10}$ - oleylamine / hexadecane than in the system water / potassium oleate-oleic acid-$C_{12}E_{10}$ / hexadecane.

References:

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Figures:

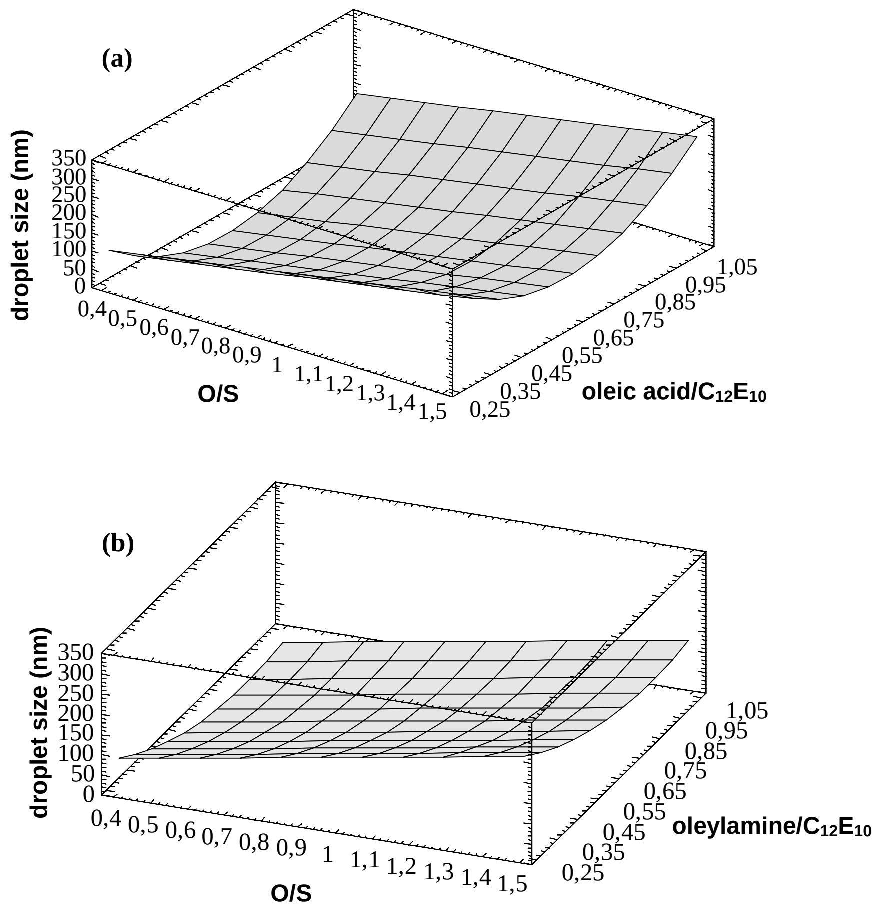

Figure 1. Response surfaces: droplet size as a function of the composition variables for the ionic system with potassium oleate as anionic surfactant **(a)** and for the ionic system with oleylamine chloride as cationic surfactant **(b)**.