Colloidal nanoparticles of gelled oil – elaboration process and characterization

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The aim of this study was to elaborate a stable dispersion of organogel nanoparticles in water. Organogels are soft materials, which result from the immobilization of an organic liquid in a three-dimensional network by a gelator. The organogels in this study were obtained from an organic oil and a low molecular-mass organic gelator (LMOG). These organic gelators have several advantages over polymer gelators because of their greater flexibility of use and their gelling capacity even in very small quantities. LMOG based organogels are usually prepared by dissolving the gelator in an oil at high temperature and then cooling the solution below its characteristic sol-gel transition temperature (Tgel). Tgel is empirically defined as the temperature below which flow is not discernible. It depends on the concentration of the gelator, the properties of the oil (polarity, viscosity, etc.) and, in some cases, the conditions of cooling.

As the LMOG, we chose 12-hydroxystearic acid (HSA), an efficient and well known gelator largely used in cosmetic formulations and dicaprylyl ether, an emollient oil also used in cosmetics.

The first step in the preparation of the aqueous dispersion was based on the gelation of dicaprylyl ether oil by 12-hydroxystearic acid. Consequently, we began the study of the physicochemical properties of the organogel by gaining an understanding of the gelation process, which is the basis of gelled nanoparticle preparation. According to the literature, HSA molecules self-assemble in some organic liquids via intermolecular hydrogen bonds between hydroxyl and carboxyl functional groups. At nanometric scale, the self-assembly leads to fibrils which themselves assemble at large scale into an interconnected scaffold of fibers. Scanning Electron Microscopy (SEM) observations of the organogel obtained from dicaprylyl ether with 15 wt % HSA confirmed the same kind of fibrous structures (Fig. 1).

After organogel elaboration, aqueous dispersions were prepared in two successive steps. First, the melted organogel (T > Tgel) was emulsified in water by sonication in presence of an emulsifying or stabilizing agent (CTAB or PVA 80). Then, after sonication, the emulsion was cooled at room temperature (T < Tgel) and gelation transformed the oil droplets into gelled nanoparticles, dispersed in water. Another great advantage of this process is that the emulsifying agent, for example CTAB, can easily functionalize the particles by simple adsorption on their surface (Fig. 2a).

Once the dispersion had been elaborated, we characterized the particles and studied the influence of the concentration of different ingredients (oil, gelator, surfactant) on their aqueous dispersions and their aging. This investigation showed the importance of the amount of oil and surfactant on the size of the nanoparticles and the stability of their dispersions. The TEM observation of stable dispersions showed spherical particles with a mean diameter of 250 nm in accordance with the DLS measurements (Fig. 2b). Compared of corresponding organogel, the gel-sol phase transition of dispersions stabilized with CTAB indicates a perturbation of the melting process (Fig. 3a). This effect was confirmed by a comparative study of the Tmelt (gel-sol transition temperature) of a dispersion prepared with a polymer (PVA 80) that does not favour the perturbation of the melting process (Fig 3b).

Also, a comparative study of the aging of the emulsion and the corresponding nanoparticle dispersion studied by photodensitometry showed a greatly enhanced stability (over 45 days) after gelation. All these results demonstrate the interest of these gelled nanoparticles and their aqueous dispersion for the preparation of new formulations for cosmetic applications or smart nanoparticles for drug delivery.

References:

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Figures:

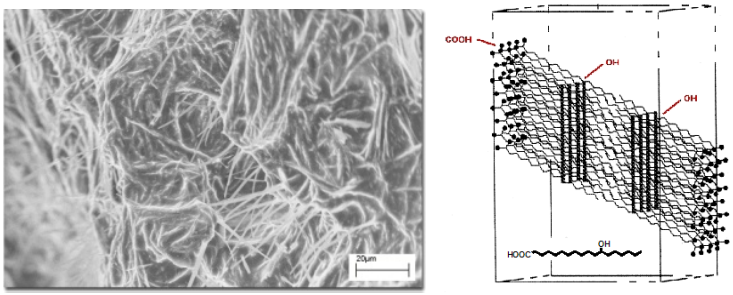
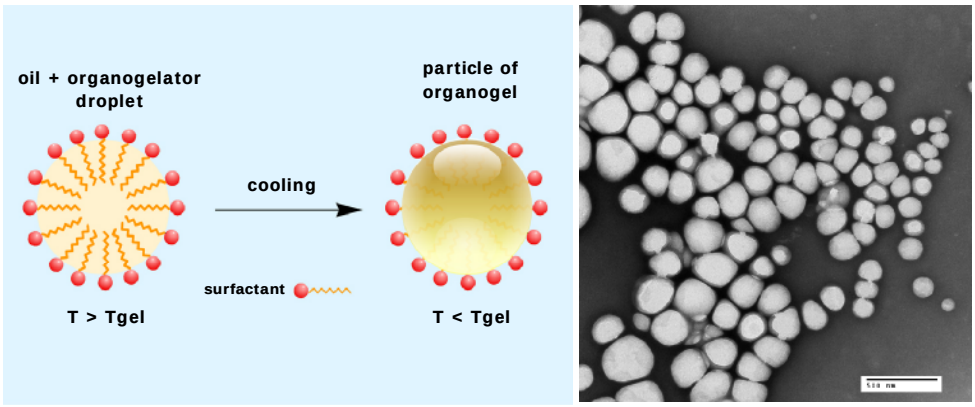
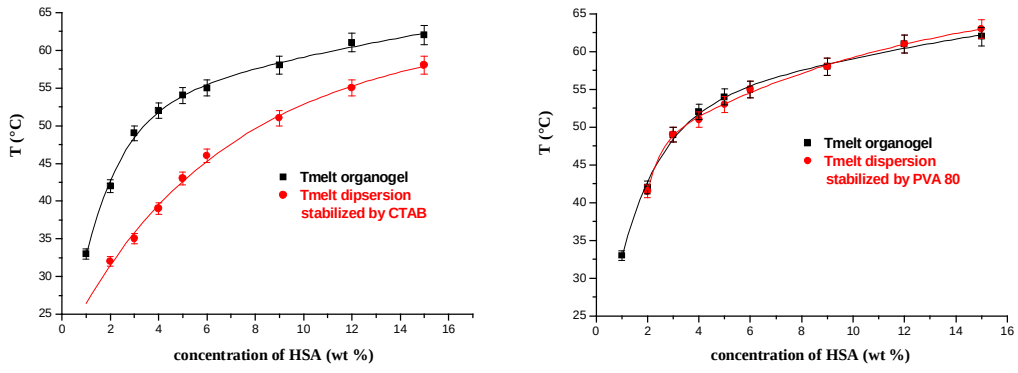


Figure 1 : SEM micrograph of the organogel (15 wt % HSA and dicaprylyl ether).



3a **3b**

Figure 2 : Elaboration concept of gelled nanoparticle aqueous dispersions (**3a**) and SEM micrograph of the particles (**3b**).



3a **3b**

Figure 3 : Gel-sol phase transitions of the organogel and the corresponding dispersion stabilized by CTAB (1.6 wt %; **3a**) and by PVA (1.6 wt %; **3b**).