Porous silica with complex dual morphology, prepared with a novel silica precursor in highly concentrated emulsions

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Porous inorganic oxides are usually prepared by sol-gel synthesis in the presence of surfactant systems, which act as templates for the control of the pore size and morphology. In a previous work, we obtained materials with dual meso/macroporous structures, by hydrolizing tetraethyl orthosilicate (TEOS) in the external phase of O/W highly concentrated emulsions, where this external phase was a liquid crystal [1]. However, ethanol released by TEOS hydrolysis produced emulsion instability and also obstructed the formation of ordered mesopores.

In the present work, silica with bimodal pore size distribution was obtained by a new simple method [2]. A novel hydrophilic precursor, tetra(2-hydroxyethyl) orthosilicate (abbreviated as THEOS) was hydrolized in the continuous phase of highly concentrated emulsions with a cubic liquid crystal in this external phase stabilized with a polyoxyethylene alkyl ether surfactant.

Highly concentrated emulsions possess volumes of dispersed phase higher than 74%, maximum packing for monodispersed spherical droplets [3,4]. Therefore, these emulsions consist of densely-packed droplets, separated by a thin film of external phase [3-5]. In our studies, a cubic liquid crystal was present in these thin films that surround the oil droplets. The hydrolysis and condensation reactions of the precursor were carried out in the cubic liquid crystal, at slightly basic pH.

Interestingly, the structure of the cubic liquid crystalline phase, was stable during the sol–gel silica synthesis. As a result, mesoporosity was formed, replicating the same morphology of the cubic liquid crystal. Moreover the presence of emulsion oil droplets allowed to obtain a interconnected macroporous texture, with morphology similar to that of foams. Consequently, materials that simultaneously possessed mesopores and macropores, were obtained. The mesopores were small (around 4 nm in size), highly monodisperse, well-ordered and with cubic symmetry. In the other hand, the macropores were much larger (with pore sizes between 1 and 5 µm) polidisperse and highly interconnected, replicating the morphology of the highly concentrated emulsion droplets. Monoliths with a specific surface area around 500 $m^2\ g^{-1}$ and bulk density of 0.16 $g\ cm^{-3}$ were been obtained.

References

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