Facile Synthesis of Meso/Macroporous Materials in Highly Concentrated Emulsions with a Cubic Liquid Crystal

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Highly concentrated emulsions are characterized by an internal phase volume fraction larger than 0.74, which is the maximum packing of monodisperse spherical droplets [1,2]. Consequently, these emulsions have a compact foam-like structure, which consist in deformed and/or polydispersed droplets, separated by a thin film of continuous phase [1,2]. In our previous studies, silica porous materials were obtained by hydrolysing tetraethyl orthosilicate (TEOS) in the external phase of O/W highly concentrated emulsions, where this external phase was a liquid crystal [3]. However, ethanol released by TEOS hydrolysis produced emulsion instability and also obstructed the formation of ordered mesopores [4].

Very recently, a new simple one-step method has been developed to obtain SiO_2 monolithic materials with a bimodal meso- and macroporous pore-size distribution [5]. Sol-gel reactions were carried out in the continuous phase of highly concentrated emulsions with a cubic liquid crystal in this external phase, using a polyoxyethylene alkyl ether surfactant and containing a novel glycol-modified silane, tetra(2-hydroxyethyl) orthosilicate (abbreviated as THEOS). The hydrolysis and condensation reactions of this precursor have been carried out in basic pH, between pH 8.8 and pH 11.4. Interestingly, the ethylene glycol released during condensation reactions does not affect significantly the phase behaviour, and consequently the cubic liquid crystalline phase was stable during the sol-gel reactions. As a result, the cubic phase based emulsions could template the formation of meso/macroporous dual materials, which possess interconnected polydisperse macropores, between 1 and 5 μm, and cubic-ordered mesopores, with a narrow pore size distribution around 4 nm. Monoliths with a specific surface area higher than 550 m^2 g^{-1} and bulk density of 0.16 g cm^{-3} have been obtained.

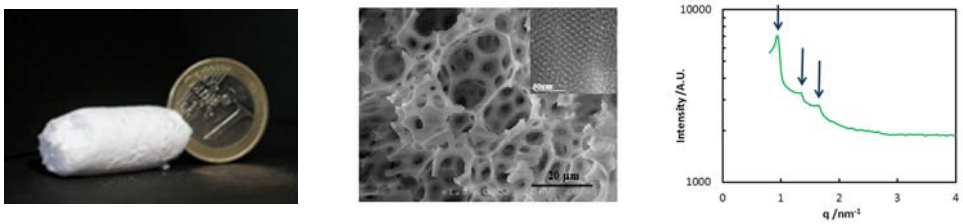

Fig. 1 Image at a macroscopic scale (left), mesoscopic and nano scale (centre) and small angle X-ray spectra (right) of a meso/macroporous dual material.

1. K. L. Lissant, 1966, The geometry of high-internal-phase-ratio emulsions, J. Colloid Interface Sci., 22, 462-468.
2. J. Esquena, C. Solans, In: Emulsions and Emulsion Stability; Taylor and Francis, New York, 2006.
3. H. Maekawa, J. Esquena, S. Bishop, C. Solans and B.F. Chmelka, 2003, Meso/macroporous inorganic oxide monoliths from polymer foams, Adv. Mater., 15, 591.
4. J. Esquena; J. Nestor; A. Vílchez; K. Aramaki; C. Solans, 2012, Preparation of mesoporous/macroporous materials in highly concentrated emulsions based on cubic phases by a single-step method, Langmuir, 12334.
5. J. Nestor; A. Vílchez; C. Solans; J. Esquena, 2013, Facile synthesis of meso/macroporous dual materials with ordered mesopores using highly concentrated emulsions based on a cubic liquid crystal, Langmuir, 432.