Synthesis of Mesoporous SiO_2 and TiO_2 Nanoparticles by Miniemulsion Technique

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Micelle-templated mesoporous materials are of great interest in many fields of chemistry for example as catalyst support and sorption media because of their large internal surface area. Here we report a novel approach to the synthesis of spherical mesostructured SiO_2 nanoparticles. These materials have been prepared by sol-gel processing of a water-soluble ethylene glycol-modified precursor (tetrakis(2-hydroxyethyl)orthosilicate, EGMS) by the indirect miniemulsion technique. This process of emulsification is of advantage because it produces small monodisperse precursor droplets, which are transformed into the final SiO_2 particles by hydrolysis and condensation. Every droplet acts as a nanoreactor without changing its size. Therefore the final particle size can be easily controlled by the droplet size; the morphology of the particles can be tuned in a wide range.

The benefit of glycol-modified silanes compared to the commercially available tetraalkoxide precursors is their high water solubility. This is particularly important for preparing mesoporous materials by templating of a liquid crystal aqueous phase. With the use of EGMS the structuring surfactant can be directly dissolved in the dispersed phase without the addition of a co-solvent such as ethanol.

The organic-inorganic precursor and the templating amphiphile (CTAB) were solubilized in acidic media and dispersed as small droplets in a hydrophobic phase comprising a non-ionic block cololymer (Poly(ethylene-co-butylene)-b-poly(ethylene oxide) by ultrasonation. The obtained powders were calcined and characterized by (small-angle) X-ray diffraction, transmission and scanning electron microscopy. Small structured silica particles ranging from 60 nm to 120 nm have been prepared. With the template of CTAB hexagonal mesoporous silica has been generated with a unit of repetition of about 4 nm. Using other precursors for example bis(2-hydroxyethyl)titanate (EGMT) also TiO2 nanoparticles in the same size range could be synthesized.