Creation of Surface Mesopores in Macroporous Silica via Partial Pseudomorphic Transformation

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In this study, partial pseudomorphic transformation was employed to structure the pore walls of macroporous silica and to introduce surface mesopores. Cetyltrimethylammonium hydroxide (CTAOH) is used as structure directing agent (SDA) and macroporous silica (initial pore diameter ~140 nm and macropore volume ~0.96 cm³ g⁻¹) as silica source. A graded degree of partial pseudomorphic transformation between 0 to 80% was attained by adjusting the synthesis conditions (hydrothermal treatment time, silica to CTAOH ratio and concentration of CTAOH). The transformed material was analyzed by nitrogen physisorption at 77 K, water adsorption at 298 K, scanning electron microscopy (SEM), scanning transmission electron microscopy (STEM) and mercury porosimetry. The nitrogen physisorption at 77 K showed that specific surface area increased gradually from 16 m² g⁻¹ to 107 and 907 m² g⁻¹ and mesopore volume from 0 cm³ g⁻¹ to 0.1 and 0.82 cm³ g⁻¹ as the transformation degree increased from 10 to 80%. The creation of surface mesopores of ca. 4 nm in the macropore walls of the parent material was verified by nitrogen physisorption at 77 K and STEM. The water adsorption at 298 K exhibited the creation of ordered mesopores without the presence of substantial microporosity. Mercury porosimetry confirmed the preservation of initial macropore diameter, while SEM proved the preservation of the initial macroporous architecture and macroscopic shape of the parent material. The mentioned results revealed the pseudomorphic transformation of macroporous silica i.e., by maintaining the initial pore structure and macroscopic shape of the parent material into micellar templated silica (MTS), with hierarchical porosity, which could be a potential support for e.g. catalysts in supported ionic liquids. Such macropores will provide efficient mass transfer and surface mesopores will anchor the ionic liquid film to help forming the wetting layer on the surface and result into enhanced catalytic activity.