Structure Fingerprints of Ordered Mesoporous Silica

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Mesoporous materials present structural complexity, that together with the small volume of ordered domains, make it difficult the precise assignment of their structure. The combination of many different characterization techniques, like X-ray diffraction (XRD) or scattering (SAXS), transmission electron microscopy (TEM) and electron diffraction (ED) are necessary to properly determine the mesoporous structure. The use of different templates, like block copolymers (PEO-PPO-PEO; PEO-PBO-PEO) and methyl-ammonium compounds (CTAB), gives hexagonal and cubic silica structures having different pore's diameter and wall thickness. In this work, the optimized synthesis of hexagonal $P6_3/mmc$ SBA-15 and MCM-41 and, cage-like cubic *Fm3m* FDU-1 and *Im3m* SBA-16 are described. The samples were prepared with commercial TEOS and Cab-O-Sil silicon sources. The as-synthesized and calcined (at 540ºC) powders were analyzed by SAXS, TEM and N_2 gas adsorption, allowing a precise determination of the different material's structure, by the recognition of typical results.

The cubic structures present larger lattice parameter; the FDU-1 has a ~20 nm and, SBA-16 has a ~14 nm. The hexagonal SBA-15 and MCM-41 have a ~ 5 nm. Besides the analysis of the diffraction peaks, information on the non-ordered pores and micropores in the silica walls were also obtained from the SAXS data. The extent of pore's shrinkage effect, due to the calcination process, was also analyzed.

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