Institute of Chemical Engineering

Crystallization of ts-1 zeolite in skeleton of titania-silica hierarchical monoliths

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Summary: The paper shows the results of preliminary research on transformation of an amorphous phase of titania-silica skeleton of porous monolithic materials into crystalline structure of the TS-1 zeolite. The materials subject to transformation were characterized by hierarchical micro- and macro-pore structure and high dispersion of titanium atoms in the silica skeleton. The concentration of Ti in all materials was 2 wt.%. The crystallization process was carried out in aqueous solution of tetrapropylammonium hydroxide (TPAOH) at 175 oC in autoclave. The impact of TPAOH and silica concentration and crystallization time on the final crystal structure of the materials was investigated. Low temperature adsorption of nitrogen, scanning electron microscopy (SEM) and diffraction of X-ray were employed to study properties of the materials. The original form of the cylindrical monoliths has only been partially preserved when low concentration of TPAOH (8 wt.%) and short time of crystallization (24 h) were used. Under higher concentration (16 wt.%) and for longer time (60 h) fine cylindrical microcrystals were obtained, and presence of TS-1 zeolite was confirmed by XRD analysis. The monolithic materials contained two phases, i.e. the crystalline structure characteristic for TS-1 zeolite and an amorphous one. SEM images showed quite well-developed spherical microcrystals with diameter ca. 5.8 nm in the skeleton and well-preserved macroporous structure. The changes in structural parameters of transformed materials were observed. The size of micropores present in silica skeleton

of the pristine material decreased from 1.5 nm to 0.94 nm. A reduction of the specific surface area (SBET) was also detected.

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