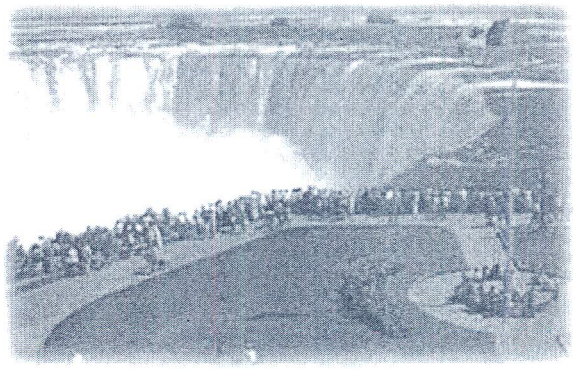

7th International Symposium

NANOPOROUS MATERIALS – VII

TECHNICAL PROGRAM

Niagara Falls, Canada
June 22 - 25, 2014

NANOPOROUS MATERIALS - VII
POSTER SESSION – II
(19:00 – 21:00, Monday, June 23, 2014)

MATERIAL SYNTHESIS/CHARACTERIZATION

Synthesis of MFI Zeolite Nanosponge with Uniform Mesopores by Seeding with Bulk Crystal in Surfactant-Directed Crystallization

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Introduction. Recently, Choi et al. reported the synthesis of 2.5-nm thickness MFI zeolite nanosheets using multi-ammonium surfactants as structure-directing agent (SDA) that could function in meso- and micro-length scales simultaneously.[1] The surfactant-directed MFI zeolite nanosheets are so far obtained as regular stacks of multiple layers supported by surfactant layers (called 'multilamellar MFI nanosheets') or disordered assemblies (called 'unilamellar MFI nanosheets). The mesopores between the zeolites layers in multilamellar MFI nanosheets were collapsed after removal of the surfactants layers. In case of unilamellar MFI nanosheets, the mesopores were retained after calcination but the size distribution of mesopore was very broad in the ranging from 5 to 25-nm. Here, we demonstrate that it is furthermore possible to synthesize the surfactant-directed MFI zeolite nanosponge possessing uniform mesopores by bulk crystal seeding.

Experimental section. The MFI zeolites were hydrothermally synthesized using, $C_{18}H_{37}$-N^+-$(CH_3)_2$-C_6H_6-$N^+(CH_3)_2$-C_6H_{13} as zeolite SDA. The synthesis gel was prepared as following the procedure of previous work for MFI zeolite nanosheets,[1] except for the addition of bulk MFI zeolite as the seeds. The added amount of seed was 5 wt.% of total silica amounts. A hydrothermal reaction at 150 °C for 2 days was employed for crystallization. The resulting zeolite is denoted by 'ZNS'.

Result and discussion. The ZNS exhibited a nanosponge-like morphology, in which 2.5-nm thick MFI zeolite layers were self-supporting each other. The hierarchically porous ZNS had high BET surface area (540 m^2 g^{-1}) and total pore volume (0.4 cm^3 g^{-1}). More importantly, the ZNS exhibited a very narrow distribution of mesopore, centered at 3.5 nm (Fig. 1). Such a pore textural property is a different from the unseeded control sample, showing broad distribution of mesopore diameters (5-25 nm). Furthermore, as a result of seeding, ZNS was rapidly obtained within 6 d of hydrothermal reaction, even at a Si/Al=15 of synthesis composition. The required time for crystallization of ZNS was three times shorter than that of unseeded control sample. Interestingly, the bulk MFI zeolite additives disappeared completely in the final product of ZNS. These implied that the added bulk crystals were acted as a seed after complete disintegration into sub-nanometer nuclei. Based on our results, we believe that the present strategy of seeding with bulk crystal can be extended to other synthesis of hierarchical zeolites.

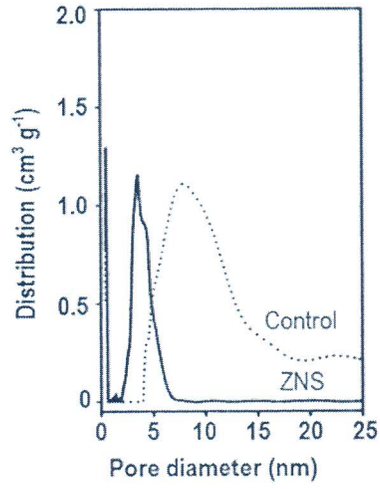

Figure 1. Pore size distributions of calcined ZNS and calcined control sample, which are analyzed using nonlinear density functional theory from adsorption branch of Ar isotherms.

[1] Choi et al. *Nature* **2009**, *461*, 246