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Structural investigation of mesoporous material made from silica-water-surfactant system by electron microscopy

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It has been discovered that ionic surfactants can be used as templates for the synthesis of silica-mesoporous materials, M41S and FSM-16. The synthesis methods open the new mesoporous materials to provide periodically arrayed spaces (cages and channels) of meso-scale region, and therefore they are attractive as hosts for making new low dimensional materials in their spaces. Recently, a series of new 3-dimensional (3-D) mesoporous materials has been reported at different conditions. In order to characterize the structure of the materials, powder X-ray diffraction (XRD) technique has been used mostly, because crystal sizes are so small. If the number of observed reflections in powder XRD pattern is small, crystal symmetry (space group) and sometime even the crystal system can not be uniquely determined. Therefore structural information must be supplied by other experiments. In this investigation, a method to determine the structure of mesoporous materials by using transmission electron microscopy, based on Electron Crystallography, was established and the structures of some mesoporous materials (3-D; SBA-6, SBA-1, SBA-16 and SBA-2, 2-D; MCM-41, FSM-16 and SBA-8) were characterized or determined.

TEM observations were performed with a 300 kV electron microsope (JEM-3010, Cs = 0.6 mm). Images were recorded by a Multiscan CCD camera (model 794, Gatan, size 1024 × 1024 pixels, pixel size 25 × 25 μm) at from 50 k to 100 k times magnifications using low dose conditions.

SBA-6 is one of the new 3-D mesoporous materials, and we determined the space group for this material to be Pm-3n. By Fourier sum of the crystal structure factors extracted from TEM images of the material, the 3-D electrostatic potential distribution was constructed. Using N_2 gas adsorption data, threshold in the potential density was estimated for differentiating amorphous wall and cavities, and 3-D structure was determined. SBA-6 has two kind of cavities with different sizes, A and B, arranged in A15 type (A_3B). Cavity A is surrounded by twelve B cavities and they are connected through window openings. By the same procedure, 3-D electrostatic potential distributions and structures of SBA-1 and SBA-16 were determined as shown in Table.

Table Structure of new mesoporous materials of SBA-6, SBA-1, SBA-16 and SBA-2.

Name	Space group	Types of pore shape and its arrangement	Pore diameter	Size of window
SBA-6	Pm-3n	cage, A15 type	A 73 Å B 85 Å	A-B 20 Å B-B 32 × 41 Å
SBA-1	Pm-3n	cage, A15 type	A 40 Å B 33 Å	A-B 3 Å B-B 15 × 23 Å
SBA-16	Im-3m	cage, BCC	95 Å	23 Å
SBA-2	hcp/ccp	cage, Closed pack		

MCM-41 is one of the 2-D mesoporous materials. We characterized the shape and diameter of the pore, the wall thickness, and the lattice parameter of MCM-41 synthesized by using surfactants with different alkyl-chain length. In addition, the growth mechanism of FSM-16 was studied and the structure determination of SBA-8 was performed.