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Synthesis of mesoporous silica MCM-41 from sodium silicate: the effect of CTAB:Si ratio

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The template synthesis is interesting process among methods of synthesis porous silica gels. Pore size can be controlled by adding the surfactant that leads to ordered silica assembling. Silica with structure of MCM-41 has regular porous structure, which is assembled by lingering open cylindrical channels with diameter of 2-4 nm. It has high values of specific surface area (over 1000 m^2/g), high thermal stability and wide range of potential applications in sorption, as support for heterogeneous catalysts. However, the application of MCM-41 is limited by high cost of material because expensive tetraethoxyortosilane (TEOS) and cetyltrimethylammonium bromide (CTAB) is used generally as precursor of silica and surfactant-temple, respectively. Sodium silicate ($Na_2O \cdot nSiO_2$) is another possible cheaper precursor of silica, however, template synthesis of mesoporous silicates from sodium silicates is not enough studied.

The present work is devoted to the study the dependence of the structure and properties of the material MCM-41, synthesized from sodium silicate, from the conditions of synthesis. Special attention is given to effect of CTAB/Si ration on structure of MCM-41 with low CTAB concentration (CTAB/Si<0.4). The MCM-41 was synthesized with composition of $525(H_2O):X(CTAB):1(SiO_2)$, X = 0.025–0.4. MCM-41 was obtained in water at pH ≈12 without catalyst by mixing of CTAB solution and sodium silicate solution with following by hydrothermal treatment at 110 °C during 24 h. Then, materials were washed by distilled water and calcined at 540 °C during 10 h. The structure of MCM-41 was studied by low-temperature N_2 adsorption using 3Flex analyzer (Micromeritics, US). The specific surface area (SBET) of the MCM-41 calculated from the absorption branch of isotherm in the range of relative pressures from 0.02 to 0.20. Pore size distributions were calculated with using of BJH method, Horvath-Kawazoe and DFT.

It was shown that silica with structure of MCM-41 can be obtained from sodium silicate. The adsorption-desorption isotherms for prepared samples have a high adsorption step at relative pressures of 0.2-0.35 that confirm the formation of silicate with structure of MCM-41 with narrow pore size distribution. The MCM-41, synthesized with ration CTAB/Si = 0.05 and 0.075, are characterized by narrowest pore size distribution from 3.2 to 3.9 nm (width at half-height pore size distribution W1/2=0.19 nm). With growth of ratio CTAB:Si from 0.025 to 0.4 the specific surface area increase from 1120 to 1384 m^2/g, that is attributed with pore size decrease.

Thus, it was shown that synthesis of MCM-41 with well-ordered structure may be realized with using of sodium silicate as silica precursor and small amount of CTAB. Growth of CTAB amount above CTAB/Si>0.1 leads to increased SBET but the ration CTAB/Si=0.05 is optimal for synthesis of MCM-41 with narrowest pore size distribution.