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Microwave-assisted rapid synthesis of Si-MCM-41

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Synthesis of mesoporous molecular sieves of uniform pore size in the range of 15 to 100 Å has elicited considerable interest for their potential applications in catalysis and selective adsorption of gases and liquids. MCM-41, a hexagonal array of unidimensional parallel channels with diameters 15 to 100 Å, is prepared hydrothermally using a broad spectrum of surfactants as templates under a wide range of synthetic conditions (temperature, *p*H, reaction time and gel compositions). Hydrothermal synthesis of MCM-41 is mostly reported to take several days for completion. The use of microwaves for the synthesis of inorganic porous solids like zeolites has been reported to reduce the synthesis time significantly. Recently, Chen-Guey Wu and Thomas Bein have reported the synthesis of Al-MCM-41 using temperature control microwave ovens within about 1 h with tetramethylammonium silicate and Hi Sil T600 as a source of silica and $C_{16}H_{33}N(CH_3)_3$ Cl/OH (60% of OH anion) as templating agent.

We report, in the present study, rapid synthesis of hexagonal mesoporous silica MCM-41 by irradiating precursor gels with microwaves for different time intervals (10–60 min). The samples were characterized by powder X-ray diffraction (XRD) and BET by nitrogen adsorption at 77 K. Properties of these samples were compared with the MCM-41 prepared by the conventional hydrothermal method (100°C for 144 h). No significant difference was observed in the specific surface area of samples prepared under either microwave or conventional hydrothermal conditions. X-ray diffraction data revealed that samples prepared by the conventional hydrothermal method showed maximum value of net integral intensity of *d*(100) reflection and minimum value of full width at half maximum (FWHM). However a sample prepared with 40 min microwave irradiation time yielded maximum crystallinity amongst samples prepared under microwave irradiation.