

Characterization of tungstophosphoric acid supported on MCM-41 mesoporous silica by using n-hexane cracking, benzene adsorption, and x-ray diffraction.

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Abstract

MCM-41 with all-silica composition was synthesized with unit cell parameters $a=b=60\text{\AA}$. The material was characterized by X-ray powder diffraction and benzene adsorption. Combinations of unit cell parameter, benzene adsorption and crystal density determination have given values for the pore diameter and wall thickness in the ab -plane of the unit cell. The unit cell size shrank upon impregnation of the material with tungstophosphoric acid. The catalytic activity of tungstophosphoric acid (HPW) supported on the all-silica mesoporous MCM-41 material has been evaluated for the cracking of n-hexane. The catalyst showed a very high activity of n-hexane cracking at a temperature as low as 200°C , at very low space velocity. However, the catalyst deactivated rapidly due to coke deposition on the acid sites. At higher temperature the activity declined, most likely because of the heteropoly-acid thermal instability. Both X-ray powder diffraction and infra-red spectroscopy have shown evidence for the heteropoly-acid instability starting at 300°C . The type of the solvent used in the impregnation affected the dispersion of HPW on the MCM-41 material as indicated by benzene adsorption measurement, but it did not influence the catalytic performance for n-hexane cracking. In the case of aqueous solution, the benzene adsorption was lower than that of the parent MCM-41 material. The reduced adsorption capacity was probably due to the presence of the HPW at the pore mouth of the cylindrical channels, causing narrowing and blocking of some sections of the channels. Moreover, the adsorption isotherm showed a micropore characteristic supporting the pore narrowing proposal. In the case of using methanol solvent for the impregnation, the full adsorption capacity was retained, indicating a much higher dispersion of the HPW throughout the channels of the MCM-41 material. The benzene adsorption isotherm also showed the characteristic of mesoporosity and the adsorption capacity was not appreciably reduced. Characterization of the spent catalyst after n-hexane cracking test showed additional reduction in the benzene adsorption capacity for the aqueous impregnated catalyst. Again the methanol-impregnated catalyst had almost a full adsorption capacity.