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Co-MCM-41介孔分子筛的水热合成与稳定性

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摘 要: 以硅酸钠、氯化钴等无机盐为原料, 通过水热法合成含有不同Co含量的六方介孔分子筛Co-MCM-41。采用X射线粉末衍射、红外光谱、等离子发射光谱、程序升温还原、透射电子显微镜和 N_2 吸附-脱附等技术对样品的物化性能进行表征, 研究所合成的Co-MCM-41的稳定性。

研究表明: 在水热条件下合成4种不同Co含量的Co-MCM-41介孔分子筛, 其比表面积为 $809.8\text{--}1257.2\text{ m}^2/\text{g}$, 平均孔径为 $2.7\text{--}2.8\text{ nm}$ 。随着介孔分子筛中Co含量的增加, 介孔分子筛的比表面积、孔体积变小, 介孔有序性变差。所合成的含Co介孔分子筛经 $750\text{ }^\circ\text{C}$ 焙烧3 h后介孔结构被破坏, 经 $100\text{ }^\circ\text{C}$ 水热处理5 d后样品虽然具有介孔结构, 但介孔有序性差。

关键字: Co-MCM-41介孔分子筛; 水热法; 合成; 稳定性; 表征

Stability and hydrothermal synthesis of Co-MCM-41 mesoporous molecular sieves

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Abstract: Hexagonal Co-MCM-41 mesoporous molecular sieves with different contents of cobalt were synthesized by hydrothermal method and using sodium silicate and cobalt chloride as raw materials. The physicochemical properties of the samples were characterized by means of X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FT-IR), inductive coupled plasma (ICP) technique, temperature programmed reduction (TPR), transmission electron microscopy (TEM) and N_2 physical adsorption, respectively, to investigate the thermal and hydrothermal stabilities of the Co-MCM-41 samples. The experimental results show that Co-MCM-41 mesoporous molecular sieves are successfully synthesized under hydrothermal condition, with specific surface area in the range of $809.8\text{--}1257.2\text{ m}^2/\text{g}$ and average pore size in the range of $2.7\text{--}2.8\text{ nm}$. Specific surface area and pore volume of the synthesized Co-MCM-41 mesoporous molecular sieve decrease with the increase of cobalt content doped, and the mesoporous ordering becomes poor. The results of thermal and

hydrothermal tests show that the mesostructure of Co-MCM-41 mesoporous molecular sieve after calcination at 750 °C for 3 h is damaged. On the other hand, the framework of Co-MCM-41 mesoporous molecular sieve still retains after hydrothermal treatment at 100 °C for 5 d, but the mesoporous ordering is poor.

Key words: Co-MCM-41 mesoporous molecular sieve; hydrothermal method; synthesis; stability; characterization

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