SYNTHESIS AND CHARACTERIZATION OF NANOSTRUCTURED IRON OXIDE IN ZEOLITE MATRICES

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ABSTRACT

The formation of iron oxide nanoparticles within the sodalite cages and supercages of Na-Y zeolites was investigated. Ferrous ions were introduced as guest cations into the host zeolite through a vacuum-assisted ion exchange procedure. The formation of zeolite-iron oxide (ZIO) system was facilitated by addition of differing concentrations of NaOH, and accelerated by heating at 60°C and addition of hydrogen peroxide. Energy dispersive X-ray spectroscopy (EDS) studies showed a significant amount of iron in the exchanged samples. X-ray diffraction (XRD) results showed the gradual phase change to iron oxide as well as diminishing zeolite peaks at increasing concentrations of NaOH. At lower concentrations of NaOH solution, the zeolite matrix was retained but the precipitated iron oxide was too small to be detected. Several peaks were identified in the product, which corresponded to maghemite, as well as hematite, indicating that the product is a mixed oxide of iron. Calculation of the crystallite size using Scherrer's equation yielded an average diameter of 13.4 nm. Under scanning electron microscopy (SEM), the ZIO particles have a cluster-like appearance, different from the acicular particles obtained from free precipitation. Transmission electron microscopy (TEM) images of the ZIO specimens showed the particles to be less than 50 nm. The adsorption isotherm of the ZIO products is characteristic of materials with a high specific surface area, with a specific surface area of 785 m^2/g, determined using the B.E.T. method. The mean pore diameter of pure zeolite was calculated to be 7.4 nm, using the BJH method, while the size of iron oxide particles precipitated within the internal pores of zeolite is estimated to be 7.1 nm. Magnetic response measurements obtained from an alternating gradient magnetometer (AGM) shows magnetization values up to 10.2 emu/g at the maximum applied field of 10 kOe. The absence of hysteresis loops indicates superparamagnetism.

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