
Redox Treatment of an Fe/Al Pillared Montmorillonite. A Mössbauer Study

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Abstract: Pillared structures with an interlayer opening of ~ 0.3 nm were obtained after successive heat treatments of the PILC precursor in reducing and oxidizing conditions. This precursor was prepared by reacting a Na^+ -montmorillonite with an intercalant containing Al and Fe oxo-hydroxides (Al/Fe = 1). Powder X-ray diffraction, elemental analysis, ^{57}Fe Mössbauer spectroscopy, catalytic activity measurements and surface area data were used to characterize the samples. On the basis of Mössbauer spectra taken at temperatures between 4.2 and 300 K, it is deduced that oxidizing steps produce Al substituted maghemite which converts into Al substituted magnetite upon reducing heat treatment. Firing the precursor in oxidizing atmosphere forms pillars of few nm in diameter. However, heating under reducing conditions yields pillars of smaller diameter. This later behaviour is maintained even after reheating the material in oxidizing atmosphere. From the temperature dependence of Mössbauer spectra it is deduced that the diameter of the Fe oxide particles in the pillars is smaller than 10 nm.

Key Words: Lepidocrocite • Maghemite • Magnetite • Modified montmorillonite • Mössbauer • PILC • Superparamagnetism.

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