Preparation and Characterization of Montmorillonites Pillared by Cationic Silicon Species

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Abstract: The hydrolysis of 2-(2-trichlorosilylethyl)pyridine (TCSEP) or 3-aminopropyltrimethoxysilane (APTMS) yields a complex mixture of oligomers, the composition of which is a function of time. ^{29}Si NMR measurements show that the same oligosilsesquioxanes can be obtained from both compounds after prolonged hydrolysis. Ion exchange by montmorillonite with the pillaring solution obtained from TCSEP results in partial intercalation, with a noticeable fraction of the clay exchange sites occupied by protons not by the Si cationic species. Calcination of this pillared clay results in a mixture of phases showing 1.86 or 1.56 nm basal spacings. Pillaring of the same montmorillonite by the solution obtained by partial hydrolysis of APTMS results in a homogeneous solid with a basal spacing of 2 nm, which reduces to 1.65 nm upon calcination above 773 K. The microporous volume (pores < 1 nm) of these Siclays, determined after calcination at 773 K, 0.15 ml/g for the PILC obtained from TCSEP, and 0.186 ml/g for the PILC obtained from APTMS is comparable to that measured for Al or Zr pillared clays. A better thermal stability is observed for the clay pillared by APTMS, which retains a large microporosity (0.167 ml/g) up to 973 K in air. These Si-pillared clays show a small number of weakly acidic sites and some strongly acidic sites retaining ammonia up to 723 K, which are most likely localized on the clay layers.

Key Words: Cationic silicon complexes • Intercalation • Microporosity • Montmorillonite • Oligosilsesquioxanes • Pillared clays • Thermal stability

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