Organic Derivatives of Attapulgite—I. Infrared Spectroscopy and X-Ray Diffraction Studies

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Abstract: Highly significant differences are observed between the methyl derivatives of attapulgite, produced when this mineral is reacted with $(CH_3)_2Si(OC_2H_5)_2$, in the presence or in absence of HCl. In the first case, the corresponding infrared spectra show characteristic absorption bands due to the $Si(CH_3)_2$ radicals at 1260, 850 and 800 cm^{-1} as well as a shoulder at 960 cm^{-1}, the latter assigned to silanol groups. The 850 cm^{-1} frequency which is usually exhibited by trimethylsilicon compounds is also detected when —O—$Si(CH_3)_2$ radicals are grafted in the silicates, but only if HCl is present in the reaction. Neither this band nor the 960 cm^{-1} shoulder appear in the spectrum of the derivative synthetized in absence of HCl. A comparative study by both i.r. spectroscopy and X-ray diffraction does not reveal structural modifications in attapulgite after it has been methylated in absence of HCl. However, although the i.r. spectra of the HCl-methylated derivatives, prepared at different periods, do not indicate substantial structural perturbations, X-ray diffraction patterns show a gradual weakening of the peaks due to attapulgite, as reaction time increases; the intensity of the (110) order reflection is drastically reduced after a 165 hr attack. The most viable mechanism for the grafting of the dimethylsiloxy units in attapulgite is through the HCl induced silanol sites. When the reaction is taking place in anh. benzene medium (absence of HCl), dimethyldiethoxysilane may be hydrolyzed by a fraction of water contained in attapulgite; the hydrolysis products which do incorporate on the surface of the silicate are identified by i.r. spectroscopy.

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