
Dehydroxylation of Dioctahedral Phyllosilicates

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Abstract: Mössbauer spectroscopy of dioctahedral phyllosilicates showed that on dehydroxylation iron which originally occupied M(2) and M(1) sites became, respectively, 5- and 6-coordinated. The 6-coordinated sites are very distorted. No migration of cations occurs in the course of heating the specimens for 1– 3 hr at 600° – 700° C.

By using a combination of several physicochemical methods, different successive stages of the dehydroxylation process could be distinguished: (1) migration of protons; (2) localized dehydroxylation of individual associations without significant change in the overall configuration of the octahedral sheets; and (3) loss of most of the hydroxyl groups with concomitant changes in the cell dimensions. Penetration of Li into the octahedral sheets does not affect the course of the reaction, but reduces the dehydroxylation temperature and the stability of the products.

Dehydroxylation was preceded by or associated with the oxidation of any divalent iron present. Fe³⁺ derived from Fe²⁺ was indistinguishable by Mössbauer spectroscopy from iron initially present in the trivalent form. High concentrations of Fe lower the dehydroxylation temperature and reduce the stability of the dehydroxylate to the extent that partial disintegration may precede complete dehydroxylation.

Key Words: Dehydroxylation • Iron • Montmorillonite • Mössbauer spectroscopy • Oxidation • Smectite

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