
Acid Hydrolysis of Octahedral Mg²⁺ Sites in 2:1 Layered Silicates: An Assessment of Edge Attack and Gallery Access Mechanisms

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Abstract: The acid hydrolysis products of trioctahedral fluorohectorite and phlogopite have been investigated by XRD, MAS NMR spectroscopy and nitrogen BET surface area analysis in an effort to assess the relative importance of edge attack and gallery access mechanisms. A dramatic loss of X-ray crystallinity and the formation of Q³ and Q⁴ SiO₄ sites accompanied the depletion of Mg²⁺ from the octahedral sheet of both 2:1 layered structures. Depending on the extent of hydrolysis, the products derived from fluorohectorite exhibited surface areas up to 208 m²/g, whereas phlogopite hydrolysis products gave values <20 m²/g. The dramatic differences in surface areas were not related to differences in hydrolysis mechanisms. ¹⁹F MAS NMR studies indicated that the hydrolysis of fluorohectorite occurred primarily by an edge attack mechanism equivalent to the hydrolysis pathway for phlogopite. A gallery access mechanism contributed to the hydrolysis of fluorohectorite only at the later stages of octahedral Mg²⁺ depletion. Solvation effects appeared to be important in determining the surface areas of the reaction products derived from the swelling (fluorohectorite) and non-swelling (phlogopite) precursors.

Key Words: Acid hydrolysis • Fluorohectorite • Mechanism • Phlogopite

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