Synthesis and characterization of saponite clays: Part 2. Thermal stability

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ABSTRACT

Synthetic saponites prepared at atmospheric pressure and at 90 °C within 20 hours with Mg²⁺, Zn²⁺, Ni²⁺, Cu²⁺, and/or Co²⁺ in the octahedral sheets have been thermally treated under reducing and nonreducing conditions with or without the presence of steam. The thermal stability of saponite in air is investigated at temperatures up to 900 °C. The nature of the octahedral cation largely determines the thermal stability of the saponite, which increases in the order Zn²⁺, Co²⁺, Mg²⁺, to Ni²⁺ from 450 to 800 °C. The stability against reduction of the octahedral cations increases in the order Cu²⁺, Ni²⁺, to Co^{2+} from 150 to 600 °C. The recrystallization of saponite at high temperatures is usually topotactic, resulting in the formation of either pyroxene or olivine types of minerals. The reduction of saponites also depends on the nature of the octahedral cation. Cu2+-containing saponites are most easily reduced, followed by Ni²⁺-containing saponites. The difficult reduction of Co-saponites is possibly related to the thermal breakdown of the saponite structure. Mg- and Zn-saponites do not show any reduction up to 800 °C. Incorporation of Mg²⁺ in Co²⁺- or Ni²⁺-containing saponites enhances the resistance to reduction. A fully reduced Ni-saponite shows a high dispersion of small Ni particles (10 nm) throughout the sample, whereas a fully reduced NiMg-saponite consists of large Ni particles (up to 60 nm) clustered together. The influence of steam on Mg-saponite is small, although incorporation of Zn²⁺ corresponding to an Mg/Zn ratio of 2 into the octahedral sheets results in a decrease of the hydrothermal stability. The drop in the stability is evidenced by a decrease in crystallinity and in a movement of Al atoms from sites within the saponite structure to non-framework positions.