Poster Presentation

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In-situ high-temperature diffraction studies of ion-exchanged umbite

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Microporous materials such as zirconium silicates have the potential to be of importance in the nuclear industry for the selective uptake of cationic radionuclides and environmental pollutants. The structural behaviour of these materials at elevated temperatures is of interest for two reasons, the first is the densification of the exchanged materials prior to long term storage and the second is the formation of new porous phases which may have increased ion exchanged affinity for certain cations. The work presented here focuses on the umbite system. Umbite is a naturally occurring microporous zirconium titanium silicate found in northern Russia and synthetic analogues, $K_2ZrSi_3O_9 \cdot H_2O$, can be prepared using hydrothermal methods. It has an orthorhombic cell with a = 10.2977(2)Å, b = 13.3207(3)Å and c = 7.1956(1)Å. The ion-exchange of umbite with cations such as rubidium, caesium and strontium and the structures of the resulting exchanged materials have been studied. Exchanges with certain cations were found to cause a change in crystal system to a monoclinic cell. Recently Rocha and co-workers found that synthetic umbite will undergo a topotactic transformation when heated 910°C to form a new microporous zirconium silicate (AV-15) with the formula K₂ZrSi₃O9·2H₂O, but to date no in-situ work has been carried out on this phase transition. In this work the high temperature structural behaviour of five umbite samples with different exchanged cations (K⁺, Na⁺, Mg²⁺ Ca²⁺and Cu²⁺) was studied up to a temperature of 1000°C. All samples behaved very differently, indicating that the nature and location of the charge balancing cation plays an important part in determining which high temperature phases are formed. Certain general trends were observed, with group 1 cations the samples remain crystalline to high temperatures. With group 2 cations dense phases are formed at high temperatures and with transition metal cations there is a loss of crystallinity at low temperature.

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