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# Mixed-layered structure formation during *trans*-vacant Al-rich illite partial dehydroxylation

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## Abstract

The <1 μm fraction of a *trans*-vacant 1*M* illite (RM30) was studied by conventional and synchrotron X-ray diffraction (XRD) techniques, combined with thermogravimetric (TG, DTG) methods to investigate the structural transformation of illite at different temperatures and degrees of dehydroxylation ($D_T$). The oriented specimens preheated at 300 and 680 °C correspond to the non-dehydroxylated ($D_T$ = 0) and completely dehydroxylated ($D_T$ = 100%) 1 *M* illite structures. Deviation of the basal reflection positions from rationality, expressed by the coefficient of variation of *d*(00*l*) values, progressively increase from 0.05 at $D_T$ = 4%, to 0.14 at $D_T$ = 51%, and then decrease to 0.06 at $D_T$ = 95%. Similarly, for each 00*l* reflection the full width at half-maximum (FWHM) shows a bell-like evolution with increasing preheating temperature. Both of these features are characteristic for mixed-layered structures.

The experimental profiles of 00*l* reflections from the oriented partially dehydroxylated specimens perfectly matched the profiles from XRD pattern simulations calculated in terms of a mixed-layered structure in which the non-dehydroxylated (ND) and completely dehydroxylated (CD) illite layers are interstratified with a strong tendency to segregation. The content of the CD layers in the modeled mixed-layered structures of the preheated specimens show a significant linear correlation with the corresponding $D_T$ values ($R^2$ = 0.99).

In random powder XRD patterns collected with synchrotron radiation, the preheated specimens show a distinctive trend in the unit-cell parameters. However, the accuracy in determination of the unit-cell parameters at first decreases up to $D_T$ = 61% and then increases with a further increase in $D_T$. The evolution of FWHM values of individual *hkl* reflections is also similar to that observed for each 00*l* peak from oriented sample preparations. The unexpected evolution of the unit-cell parameters during progressive dehydroxylation is explained by the interstratification of ND and CD layers in illite.

The formation of the mixed-layered structures during Al-rich 1*M* illite dehydroxylation is in agreement with the prediction from the kinetic model of partially dehydroxylated dioctahedral 2:1 clay structures where dehydroxylation of each portion of the initial OH groups corresponds to a zero-order reaction that is independent of the structural and chemical composition. The reaction is homogeneous and during partial dehydroxylation of the illite structure, ND layers transform into the CD layers without formation of an intermediate phase. A layer-by-layer dehydroxylation mechanism is suggested for thermally induced illite structural transformation.

**Keywords:** Illite, dehydroxylation, XRD, modeling, structural features, DTG, synchrotron