

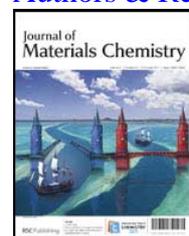
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Activation and local structural stability during the thermal decomposition of Mg/Al-hydrotalcite by total neutron scattering

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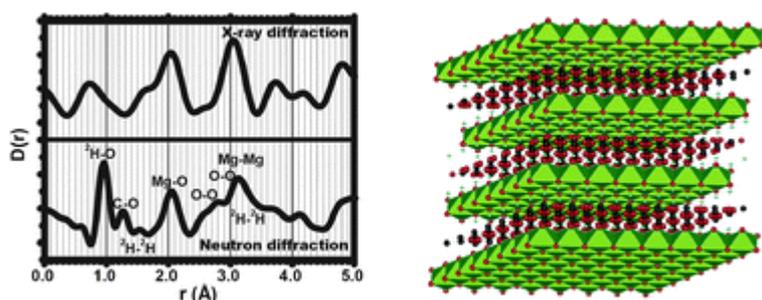
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The activation of synthetic hydrotalcite, the carbonated layered double hydroxide (LDH) with ratio $Mg^{2+}/Al^{3+} = 3$ and structural formula $[Mg_6Al_2(OH)_{16}]^{2+} \cdot CO_3^{2-} \cdot n(H_2O)$, has been investigated using neutron and X-ray diffraction. *In situ* neutron diffraction was used to follow the structural phase transformations during the thermal decomposition (calcination) of hydrotalcite under vacuum in the temperature range 298–723 K, and during which the residual gas evolved by the sample was analysed by mass spectrometry. Detailed structural information of the LDH and mixed metal oxides (MMOs) was extracted from both the Bragg peaks and the total scattering. These two analysis techniques provide complementary insight into the relevant transition mechanisms, since Bragg diffraction originates from long-range periodicities within the samples while total scattering reveals the subtleties of the local atomic environment. This latter information is particularly important for our understanding of catalytic activity since it elucidates the local metal coordination. We find that, during the calcination process, the local environment around the metal centres is robust, as the various stages during the phase transition have identical local structures. The implications of these new results for the nature of the MMOs is discussed in relation to the well-studied, reverse, rehydration reaction, and the high propensity of trivalent Al ions to migrate to tetrahedrally-coordinated lattice sites.



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