
Syntheses of Disordered and Al-rich Hydrotalcite-Like Compounds

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Abstract: Hydrotalcite-like compounds, $[\text{Mg}_{1-x}\text{Al}_x(\text{OH})_2]^{x+} [x\text{X}^- \cdot n\text{H}_2\text{O}]$, where $\text{X}^- = \frac{1}{2}\text{CO}_3^{2-}$ or OH^- , were prepared by hydrothermal syntheses at $P_{\text{H}_2\text{O}} = 100\text{ MPa}$ and $T = 100^\circ - 350^\circ\text{ C}$. Starting materials were MgO , $\gamma\text{-Al}_2\text{O}_3$, H_2O , and $\text{MgC}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$. The synthesis depended on temperature, pressure, the $\text{Al}/(\text{Al} + \text{Mg})$ ratio x , and the CO_2 content of the starting material. Previously an Al content of $x = 0.33$ was thought to be the upper limit in these double-layer compounds, but by using pressure the Al-content was increased to $x = 0.44$. Up to $x = 0.33$, a_0 decreased linearly to about 3.04 \AA , but for $x \geq 0.33$, a_0 remained nearly constant at this value. For the synthesized products the layer thickness c' varied between 7.40 and 7.57 \AA in contrast to the natural phases wherein c' varies from 7.60 to 7.80 \AA . At higher temperatures CO_2 -free syntheses, i.e., those without Mg-oxalate, resulted in a disordered hydrotalcite-like phase. The transition temperature between the ordered and the disordered hydrotalcite-like phase depended on the Al-content, x .

Clays and Clay Minerals; October 1986 v. 34; no. 5; p. 507-510; DOI: [10.1346/CCMN.1986.0340502](https://doi.org/10.1346/CCMN.1986.0340502)

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