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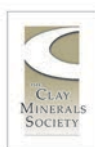


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Physico-chemical studies of Mg/Fe and Mg/Al Layered Double Hydroxides obtained via transformation of minerals

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Layered Double Hydroxides (LDH) represent a large class of phases which exhibit a brucite-like structure intercalated with anions. According to the general formula $[M^{II}_{1-x} M^{III}_x OH_2]^{x+} [A^{n-}]_{x/n} \cdot y H_2O$, the LDH structure consists of divalent (M^{II}) and trivalent (M^{III}) metals that form positively charged layers where the charge is balanced by interlayer anions. The LDH display a wide range of unique physical and chemical properties which makes them perfect candidates in a variety of industrial applications, including wastewater treatment [1]. These minerals can be rarely found in nature and represent a hydrotalcite-pyroaurite group [2], however their synthesis is easy to carry out in laboratory conditions. Nevertheless, the exclusive use of chemical reagents for their synthesis is relatively expensive. This implies a demand for other low-cost sources of metals e.g. metal-bearing minerals forming widespread deposits.

The aim of this research was to investigate the quality of Mg/Fe and Mg/Al LDH obtained by the transformation of magnesite [M] and dolomite [D]. These minerals were sources of M^{II} (Mg^{2+} and $Mg^{2+}+Ca^{2+}$, respectively) and $AlCl_3$ or $FeCl_3$ were used as a source of M^{III} . The M and D were dissolved in the appropriate chloride solution, where acidic hydrolysis enabled the release of M^{II} cations. This approach is an alternative to the use of strong acids e.g. HCl which is not desired in industry due to corrosive properties [3]. The solution containing M^{II} and M^{III} was added dropwise to the 2M NaCl solution with the pH=10 set by an aqueous NaOH and controlled constantly during the synthesis. The obtained slurry was aged (2 or 24 h), washed with water and dried at 60°C overnight. For comparison the samples of pure LDH from chemicals were synthesized in analogical conditions. Several variations of LDH in terms of M^{II}/M^{III} ration and ageing times were obtained.

The XRD confirmed presence of LDH in all samples as compared to the ICDD standards of hydrotalcite and pyroaurite. In the samples derived from minerals additional phases: gibbsite, calcite and akaganeite were formed. An increasing M^{II}/M^{III} molar ratio induced structural changes revealed by peak shifts in the XRD patterns. This was due to changes of unit cell parameters as well as change of interlayer distance. It was observed that an increase of ageing time affected the crystallinity of the obtained phases. The LDH morphology was depicted by the SEM and showed characteristic layered structures differing from the starting minerals. The surface chemistry and thermal behaviour of the materials varied as attested by DTA/TG and XPS methods.

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[2] Mills, S.J. et al. (2012) *Mineralogical Magazine* 76(5), 1289-1336.

[3] Mao, N. et al. (2018) *Applied Clay Science* 159, 25-36.