# DIFFERENT APPROACHES TO TRANSFORMATION OF SELECTED MINERALS INTO LAYERED DOUBLE HYDROXIDES

Karolina Rybka[*1] and Jakub Matusik[1]

[1]AGH University of Science and Technology, Faculty of Geology, Geophysics and Environmental Protection, Department of Mineralogy, Petrography and Geochemistry, al. Mickiewicza 30, Krakow, 30 059, Poland

Layered Double Hydroxides (LDH) are widely studied as promising materials in many different fields, such as catalysis, drug delivery and wastewater treatment. The LDH formula is the following: $[M^{II}_{1-x} M^{III}_{x} OH_2]_x + [A^{n-}]_{x/n} \cdot y\ H_2O$, where $M^{II}$ is a divalent metal, $M^{III}$ is a trivalent metal and A is an anion. The layered LDH structure generates positive charge balanced by the weakly bonded hydrated anions. This a reason for various applications of LDH as anion exchangers. The LDHs synthesis is straightforward, however, the excessive use of chemical reagents makes the whole process expensive. Currently many studies are devoted to obtaining LDH, where different materials serve as $M^{II}$ and $M^{III}$ sources. The use of abundant, easy accessible and cheap materials can significantly lower the final price of an adsorbent. Therefore in this work, Mg/Al LDH was synthesized with different contribution of minerals as precursors of Mg and Al: magnesite from Grochow deposit (Poland) and halloysite from Dunino deposit (Poland). Three variants of Mg/Al LDH were synthesized: reference sample (Mg/Al) obtained from chemical reagents ($MgCl_2 \cdot 6H_2O$ and $AlCl_3 \cdot 6H_2O$), sample obtained with magnesite as a Mg source (M/Al) and sample obtained via transformation of magnesite and calcined (750°C, 5h) halloysite (M/Hall). All substrates were dissolved to obtain Mg/Al solution. All materials were synthesized via co-precipitation method with the pH set and constantly controlled at 10. The precipitates were aged for 2 h, washed with redistilled water and dried at 60˚C. The starting minerals and the obtained materials were characterized by XRD, FTIR and SEM. All materials were dissolved in concentrated HCl and their chemical composition was determined through AAS measurements. Moreover, the stability of materials in pH range of 2-8 was investigated. For that, the materials were stirred in solutions with pH = 2, 4, 6 and 8 for 24 h, then the concentration of released elements was measured by AAS. The XRD pattern of raw magnesite sample showed the presence of quartz admixture. The content of Mg was equal to 25.7%. The XRD pattern of halloysite sample exhibited reflections characteristic for halloysite, kaolinite, quartz and iron minerals The chemical analysis revealed, that the content of Al was equal to 16.73% and Fe equal to 19.34%. Diffractograms of all synthesized materials showed peaks characteristic for LDH as compared to hydrotalcite standard. In the case of M/Al the additional formation of gibbsite was confirmed. The FTIR spectra of all materials showed bands in the regions 3700-3400 $cm^{-1}$ for OH stretching vibrations, 1540-1350 $cm^{-1}$ for the interlayer carbonates and <1000 $cm^{-1}$ for the lattice vibrations. The M/Al spectrum also showed a band at 3550 $cm^{-1}$ characteristic for Al-OH vibrations. The SEM images of obtained materials showed agglomerates of different size up to 100 µm. Moreover, in the M/Al images the grains of gibbsite were observed. The chemical analysis revealed, that the molar ratio $M^{II}/M^{III}$ was 2.8 and 5.6 for Mg/Al and M/Hall, respectively. Because of gibbsite in the M/Al, sample the Mg and Al content was not determined. The stability experiment showed, that the materials are stable in the pH range of 4-8. At pH = 2, the percent of released Mg was found to be: 8.85, 22.03 and 0.06% for the Mg/Al, M/Al and M/Hall, respectively.

This project was supported by the National Science Centre Poland, under a research project awarded by Decision No. 2017/27/B/ST10/00898.