Ultrasonic treatment and sorption properties of Mg²⁺/Fe³⁺ layered double hydroxides

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Synthesis of Mg^{2+}/Fe^{3+}-NO_3^- 3:1 LDHs was performed as follows. To a solution of 19.23 g $Mg(NO_3)_2 \times 6H_2O$ and 10.1 g $Fe(NO_3)_3 \times 9H_2O$ in 50 ml of distilled water, a solution of 8 g NaOH and 2.12 g $NaNO_3$ in 50 ml of distilled water was added dropwise. Then the reaction mixture was treated by one of the following ways: heating at 348 K for 24 hours or for 72 hours, or ultrasonic treatment at 28 kHz for 5, 10 or 15 minutes. The resulting precipitate was washed 4 times using a centrifuge (6000 rpm, 5 min) and distilled water, and dried at room temperature to a constant weight.

An experiment to study the sorption properties of LDH Mg^{2+}/Fe^{3+}-NO_3^- was carried out at a temperature of 291 K. 10.00 g of 0.1000 N K_2CrO_4 solution was added to a mixture of 490.00 g of distilled water and 0.84 g of LDH. The resulting solution was closed with a rubber septum and an aluminum cap by crimper and stirred on a magnetic stirrer bar. Samples of the reaction mixture for subsequent photometric analysis were taken with a syringe at required periods of time. To remove residual LDH, the samples for analysis were rapidly filtered through a membrane filter.

The concentration of chromate in solutions the reaction mixtures was determined photometrically using the calibration graph method. Experimental solutions and a control solution were photometered on the KFK-3 device relative to distilled water in 1 cm cuvettes at a wavelength of 410 nm. During photometry of experimental solutions, the equilibrium optical density of A_e was determined, followed by the calculation of the equilibrium concentration of potassium chromate C_e.

The microstructure study (SEM) was carried out at the interdisciplinary resource Center in the direction of "Nanotechnology" of the St. Petersburg State University Science Park using a Carl Zeiss Merlin scanning microscope (Carl Zeiss AG, Oberkochen, Germany) at 10kV, SE2 detector, working distance 6.4 mm, pressure 9.4×10^{-7} mbar. Graphite deposition was carried out on the Getal PECS Model 682 device, the deposition layer is 10 nm.

The IR spectrum of LDH Mg^{2+}/Fe^{3+}-NO_3^- was recorded on the Shimadzu IRPrestige-21 spectrometer (Japan).

X-ray diffraction analysis was performed on a Dron-7 diffractometer. 2θ angular interval from 7° to 40° with a scanning step of $\Delta 2\theta = 0.02°$ and a shutter speed of 7 seconds per point. Cu Kα radiation (Ni filter) was used, which was subsequently decomposed into components Kα1 and Kα2 during the processing of the spectra.

Magnesium to iron mass ratio was analyzed by inductively coupled plasma mass spectrometry on a NexION 300D instrument (PerkinElmer Inc., Shelton, CT, USA). The mass ratio of nitrate ions in relation to the found mass ratio of magnesium and iron was determined by reverse dichromatometric titration (an excess of a standard Fe^{2+} solution was added to a solution containing nitrate ions, obtained by dissolving a suspension of a known amount of LDH in concentrated sulfuric acid. After completion of the reaction, the remaining iron was titrated with a standard $K_2Cr_2O_7$ solution).

Thermogravimetric analysis was performed on a TA Instruments TGA Q500 device using a heating rate of 5 °C per min in the temperature range from 30 °C to 850 °C.
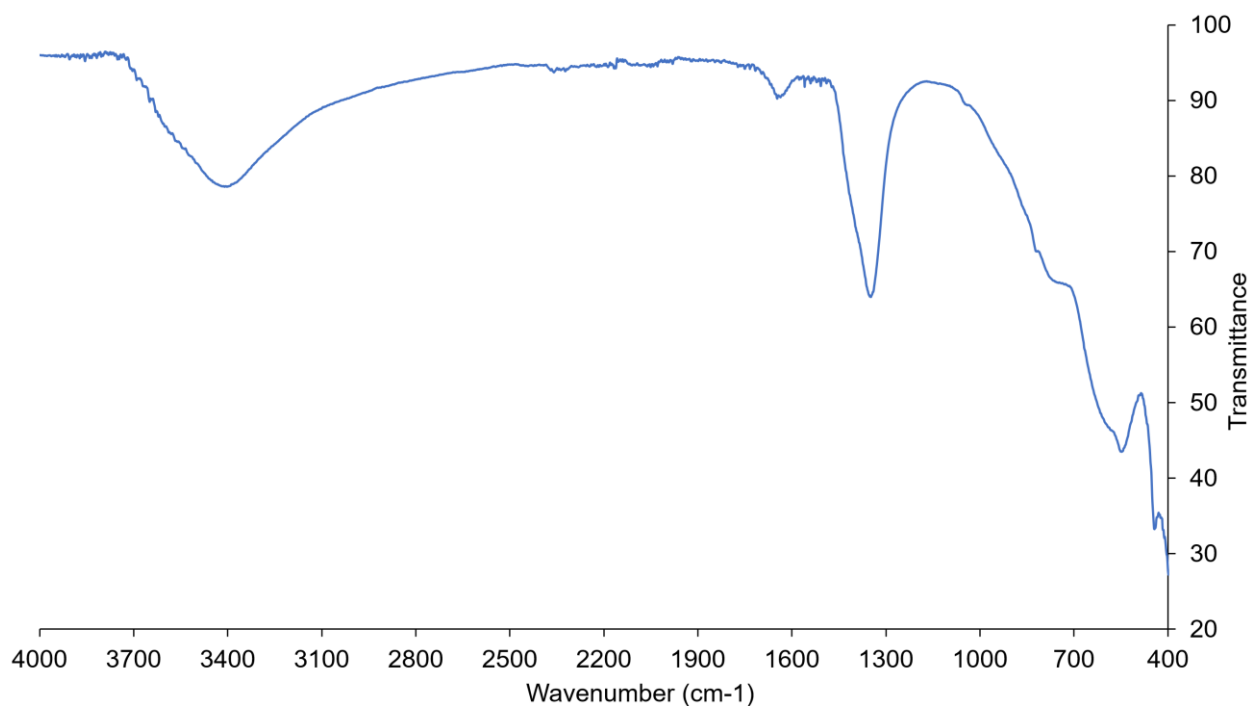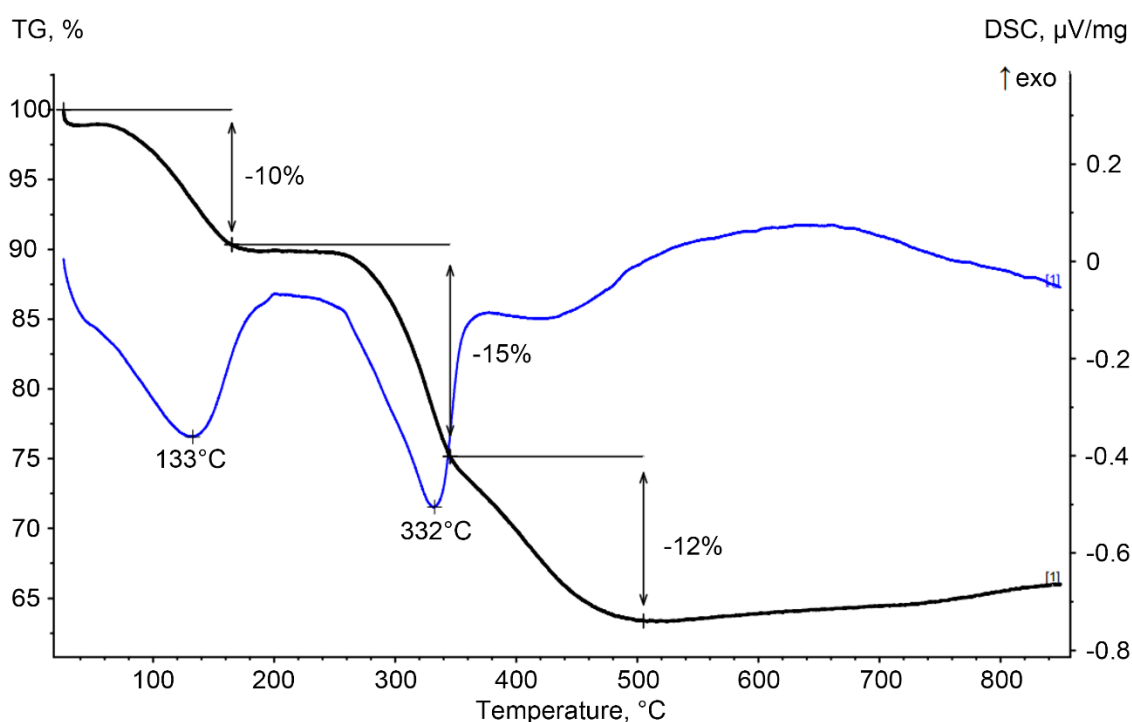

Figure S1 Typical IR spectrum of the LDH.

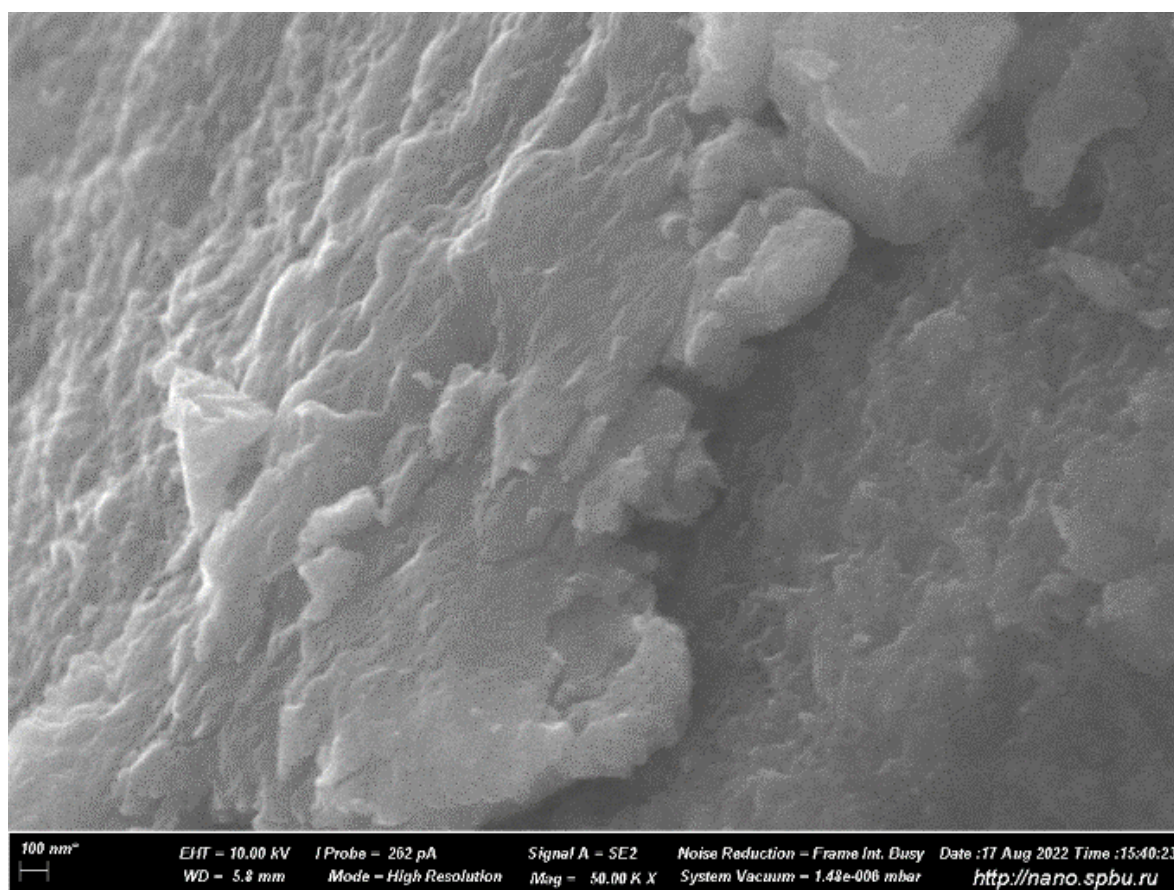**Figure S2** TGA/DSC curves of LDH Mg^{2+}/Fe^{3+}-NO_3^-.

Figure S3 SEM image of LDH $Mg^{2+}/Fe^{3+}-NO_3^-$.