

**Preparation and Structural Characterization of Alkaline Earth–Iron(III)  
Layered Double Hydroxides and their Acrylate-Intercalated Derivatives**

*PhD Theses*

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## Introduction

Layered double hydroxides (LDHs) are brucite-like materials in which the divalent metal ions are partially substituted for trivalent ones. Thus, the layers contain octahedrally coordinated two- and trivalent metal ions, and the layers are positively charged. This charge is usually compensated by fully or partially hydrated inorganic anions. LDHs are versatile substances having many uses; they can serve as catalyst supports, catalysts, flame retardants, containers of biologically active compounds, to mention just a few. Although some are found in nature, for applications they are usually synthesized. The most frequently used representative is synthetic hydrotalcite, in which Mg(II) and Al(III) are the di- and trivalent cations, respectively. It is generally believed that LDHs are formed when the ionic radii of the di- and trivalent metal ions are close to each other. Respecting this dogma many LDHs were made, nevertheless, they were studied significantly less thoroughly than Mg(II)Al(III)-LDH.

These materials have anion exchange ability, thus, various hybrid materials may be prepared with useful functionalities. Originally, the aim of the researchers was to exchange the simple inorganic anions for robust isopoly or heteropoly acid anions to obtain durable catalysts with acid-base (most of the original LDHs are basic materials) and redox properties, of channel structure with molecular size openings, thus combining different chemical functionalities and shape selectivity. However, it turned out that the direct ion exchange was not an easy exercise. The feasible way proved to be exchanging the original anions to bulky organic anions, then, finishing the exchange with the large inorganic anions. More recently, it became clear that the intermediate step, *i.e.*, exchanging the small inorganic anions to bulky organic ones, may produce even more interesting and useful functional materials than the complete procedure.

At the start of the project we were able to prepare a material containing Ba(II) and Fe(III) ions under hyperalkaline conditions (in the presence of NaOH over 10 M concentration), which was proved to be of layered structure in spite of the significant difference in the ionic radii of the two ions [0.149 nm for Ba(II) and 0.069–0.079 nm for Fe(III)]. The accumulated knowledge briefly outlined above and this enlightening result initiated further work, like

1. exploring the experimental conditions and finding the limits under which Ba(II)Fe(III) LDHs are formed,

2. characterizing this novel material with as many tools as it is possible,
3. extending the synthetic efforts for the other members of the alkaline earth ions keeping Fe(III) as the trivalent constituent and to characterize them in as many ways as it is possible,
4. finding the most efficient method for the synthesis of inorganic–organic hybrid materials where the host material is Ca(II)Fe(III)-LDH and the guest anions are various acrylates,
5. characterizing the organic–inorganic hybrids with all available methods,
6. exploring the possibility of using the organic–inorganic hybrids as nanoreactors in [2+2] topotactic cyclisation reactions.

## Experimental

### *Materials and synthesis methods*

Concentrated NaOH (~20 M) stock solutions were prepared from Millipore MilliQ water and *a.r.* grade solid NaOH and their carbonate content was minimized. The exact concentration was determined through accurate density measurement. The solution was stored in an airtight, caustic resistant Pyrex bottle. This solution was kept carbonate-free, and was used for the making of the NaOH solution actually used in the synthesis (10 M, 5 M, 3 M, 2 M, 1 M depending on the material to be made). CaCl<sub>2</sub>, MgCl<sub>2</sub>, SrCl<sub>2</sub> (*puriss*), Sr(ClO<sub>4</sub>)<sub>2</sub>·3H<sub>2</sub>O, Ba(ClO<sub>4</sub>)<sub>2</sub>·3H<sub>2</sub>O (*p.a.* grade), FeCl<sub>3</sub>·6H<sub>2</sub>O (*puriss* special) or Fe(ClO<sub>4</sub>)<sub>3</sub>·xH<sub>2</sub>O (*p.a.* grade) and cc. HClO<sub>4</sub> (ca. 30 m/m%, *p.a.* grade) were used for the syntheses.

The Ca(Mg, Sr, Ba)Fe double hydroxides were prepared by the co-precipitation method *via* dropwise addition of the two metal salt solutions with various molar ratios to hot (*ca.* 80 °C), vigorously stirred and N<sub>2</sub>-blanketed NaOH solution. The precipitates formed were rapidly filtered until air dry in a practically CO<sub>2</sub>-free atmosphere, with the aid of a caustic resistant vacuum filter unit (Nalgene) equipped with an appropriate membrane (Versapor, 0.45 μm). The solid material was washed with small quantities of pure and hot NaOH with concentration identical to that used during the synthesis. The obtained crystals were kept at room temperature in a desiccator over dry SiO<sub>2</sub>.

### *Characterization methods*

Thermal analytical measurements were performed using a Setaram Labsys derivatograph working under N<sub>2</sub> flow at 2 °C/min heating rate.

Powder X-ray diffraction (XRD) patterns of the air-dried and heat-treated solid samples were registered in the  $2\Theta = 5\text{--}55^\circ$  range on a Rigaku Miniflex II instrument, using CuK $\alpha$  ( $\lambda = 1.5418 \text{ \AA}$ ) radiation. Reflection positions were determined *via* fitting a Gaussian function. Reflection positions were found to be reproducible within  $0.05^\circ$  ( $2\Theta$ ), therefore the uncertainty of the basal spacing was estimated as  $\pm 0.1 \text{ \AA}$ .

The topology, approximate composition and the elemental map of the sample were examined with scanning electron microscope (Hitachi S-4700 microscope, 10 kV acceleration voltage) coupled to a Röntec QX2 energy dispersive X-ray fluorescence spectrometer.

<sup>57</sup>Fe Mössbauer spectra of the samples were recorded with conventional Mössbauer spectrometers (Wissel and Ranger) in transmission geometry at the temperature of liquid nitrogen (78 K). A <sup>57</sup>Co/Rh  $\gamma$ -radiation source of 3.109 Bq activity was used.

Fe–K-edge (7112 eV) X-ray absorption spectra (XANES/EXAFS) were measured on beamline I811 at the MaxLab facility, Lund, Sweden. The station is based on a superconductive multipole wiggler radiation source connected to the 1.5 GeV MAX II storage ring. X-ray radiation in the 2.4–12 keV energy range can be obtained from this system. The maximal flux on the sample at 9 keV is  $5 \times 10^{11}$  on a  $0.5 \times 0.5$  mm surface area. Measurements were performed at the Fe–K-edge (7112 eV) in the fluorescent mode. The sample holder was made of Teflon and its window was Kapton (polyimide) ribbon. Due to the high iron content of the sample, it had to be diluted with boron nitride. All works were done in a dry box. The analysis of the near-edge spectrum was performed with the help of the EXAFSPAK and the FEFF packages allowing us to obtain quantitative data on the local coordination environment of the Fe(III) ion.

The mode of intercalation was studied by FT-IR spectroscopy. Spectra were taken on a Bruker 66V FTIR spectrometer (unapodized resolution  $0.25 \text{ cm}^{-1}$ ) and coupled to a Bruker Hyperion 3000 IR microscope. For a spectrum 256 scans were collected. The spectra were

baseline corrected, smoothed if it was necessary and the spectrum of the pristine LDH was subtracted. All these operations were performed with OPUS program package.

The photochemical reactions were initiated by irradiating the host–guest materials with a MAX-302 Xenon light source (Asahi Spectra) working in the 250–400 nm wavelength range for 5 to 90 minutes. The reactions were performed with the solid intercalated compounds in the absence of the LDH as well.

The sizes of the intercalated carboxylate ions were determined after performing full geometry optimization with the PM3 semiempirical quantum chemical method included in the Hyperchem 8.0 molecular modeling package.

### 3. New scientific results

**1. The synthesis of a novel layered double hydroxide, Ba(II)Fe(III)-LDH that is, invalidating the dogma that the di- and trivalent ions in LDHs must have similar ionic radii.**

1.1 It has been verified by various methods (powder XRD, <sup>57</sup>Fe Mössbauer spectroscopy, SEM–EDX elemental maps, TG–DTG) that the co-precipitation of the aqueous solutions of Ba(II) and Fe(III) salts only leads the layered double hydroxides under hyperalkaline conditions ( $c_{\text{NaOH}} \geq 10 \text{ M}$ ) in the 2–4:1 Ba(II):Fe(III) range.

**2. It was possible to obtain quantitative structural data about Ba(II)Fe(III)-LDH regarding both the coordination of the Fe(III) ion in the first coordination sphere and the Fe–O(Ba, Fe, O) distances in the first to third coordination spheres by the XAS method.**

2.1 The coordination environment of the Fe(III) was found to be octahedral in the LDH, while from 453 K and above the LDH structure deteriorated and from now on, the environment gradually turned tetrahedral. Changes are seen in the second and third coordination sphere first, then, at 523 K the first coordination sphere also altered significantly indicating the collapse of the layered structure.

2.2 The Fe–O distance in the LDH is ~200 pm and ~400 pm in the first and third coordination spheres, respectively. In the second coordination sphere Fe(III) and Ba(II) ions are found at ~300 pm.

**3. Optimal conditions were found for the formation of layered double hydroxides when Mg(II), Ca(II) were the alkaline earth ions under normal alkaline conditions.**

3.1 Many conditions were tried and it was found, verified by XRD, TG-DTG, SEM, SEM–EDX elemental maps that using a wide range of Mg(II)[Ca(II)]:Fe(III) ratios the method keeping the final pH at 13 in the co-precipitation reaction and maintaining CO<sub>2</sub>-free environment provides with the optimum conditions for LDH synthesis.

**4. Sr(II)Fe(III) LDHs could not be prepared irrespective to the alkalinity of the precipitating medium.**

4.1 Varying the concentration of NaOH used for precipitation from normal alkalinity (2 M) to hyperalkaline (20 M) conditions and the Sr(II):Fe(III) ratios in 2–6:1 range resulted

in the formation of double hydroxides (attested by SEM–EDX elemental maps), but not of layered structure (indicated by XRD measurements).

**5. The dehydration-rehydration method was found to be suitable for the preparation of many novel acrylate–Ca(II)Fe(III)-LDHs and the possible arrangement of the interlayer ions were determined with the combination of molecular modeling and powder XRD measurements.**

5.1 Probing various methods (direct ion exchange, co-precipitating the organic and the inorganic components, dehydrating the LDH host and rehydrating it in the presence of the organic anion), the last one, the dehydration-rehydration method was found to be the most effective in the preparation of acrylate (*E*-phenylpropenoate, *E*-3(4'-nitrophenyl)propenoate, *E*-3(2',5'-difluorophenyl)propenoate, *E*-3(4'-chlorobenzoyl)propenoate, *E*-2-cyano-3-phenylpropenoate, *E*-3(4'-imidazolyl)propenoate or *E*-3(2'-thienyl)propenoate)–Ca(II)Fe(III) LDH nanocomposites verified by XRD, TG-DTG measurements, SEM–EDX elemental maps and FT-IR spectroscopy.

5.2 The possible arrangements for the organic anions are suggested on the basis of basal spacings from XRD measurements, the estimate of layer thickness and the dimensions of the quantum chemically optimized structures of the acrylate ions.

**6. Using the acrylate–Ca(II)Fe(III) LDHs as nanoreactors, photoinitiated topotactic [2+2] cyclization reactions could be performed with many representatives [*E*-phenylpropenoate–, *E*-3(4'-nitrophenyl)propenoate–, *E*-3(2',5'-difluorophenyl)propenoate– or *E*-3(2'-thienyl)propenoate–Ca(II)Fe(III)-LDHs] resulting in cyclobutane derivatives within the layers of the host material.**

6.1 The progress of the cyclization reactions could be followed by monitoring the disappearance of C=C vibration in the 1560–1670 cm<sup>-1</sup> wavenumber range. The mere fact of that cyclization took place indicated that there were domains where the intercalated anions were in close vicinity to each other and in proper arrangement.

6.2 Instrumental measurements attested that the *E*-3(4'-chlorobenzoyl)propenoate, the *E*-3(4'-imidazolyl)propenoate and the *E*-2-cyano-3-phenylpropenoate anions were intercalated in Ca(II)Fe(III) LDH, however, their arrangements and/or their distances were not suitable for the topotactic reaction to proceed, otherwise seen in the solid state in the absence of the LDH host.



#### **4. Practical use of the results**

The results presented here is of fundamental nature. Nevertheless, the concept of nanoreactor in organic–inorganic host–guest systems may be of more general use performing more selective and effective chemical reactions decreasing the burden of unwanted side products on the environment.

## 5. Papers directly related to the Theses

1. **Srankó, D.**, Pallagi, A., Pálincó, I., Kuzmann, E., Canton, S.E., Walczak, M., S. Sipos, P.:  
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2. **Srankó F.D.**, Sipiczki, M., Bajnóczy, G.É., Pálincó, I., Sipos, P.:  
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6. **Srankó, D.**, Sipiczki, M., Bajnóczy, É.G., Darányi, M., Kukovecz, Á., Kónya, Z., Canton, S.E., Norén, K., Sipos, P., Pálincó, I.:  
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7. **Srankó, D.**, Sipiczki, M., Kukovecz, Á., Kónya, Z., Sipos, P., Pálinkó, I.:  
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## 6. Presentations at conferences directly related to the Theses

1. **Srankó, D.**, Pallagi, A., Pálinkó, I., Kuzmann, E., Canton, S., Walczak, M., S. Sipos, P.:  
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2. Sipiczki, M., **Srankó, D.**, Kukovecz, Á., Kónya, Z., Sipos, P., Pálinkó I.:  
Preparation, characterisation and some reactions of organocatalysts immobilised between the layers of a layered double hydroxide,  
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*27th European Crystallographic Meeting (ECM 27)*, Bergen (Norway), 2012. (poster presentation)

full papers, total: 20

cumulative impact factor, total: 29.267

independent citations, total: 51

related to the topic of the theses: 11

related to the topic of the theses: 5.571

related to the topic of the theses: 8