Hydrothermal synthesis of samarium-containing layered double hydroxide

S. Golovin, M. Yapryntsev

Institute of Pharmacy, Chemistry and Biology, Belgorod State National Research University, 308015, Russian Federation, Belgorod, Pobedy str., 85

Structure of layered double hydroxides (Mishra G., Dash B., Pandey S., Applied Clay Science 153 (2018) 172) [M²⁺_{1-x}M³⁺_x (OH)₂]^{x+} Basal spacing (d) Interlamellar space [Aⁿ⁻_{x/n}·yH₂O]^{x-} M²⁺/M³⁺

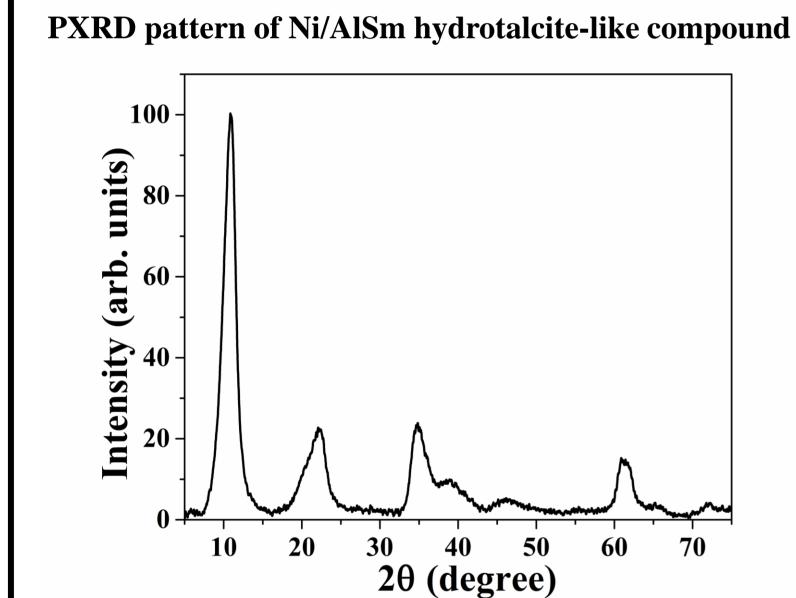
INTRODUCTION

Layered double hydroxides (LDH) consist of positive charged brucite-like layers, in which part of divalent cations are isomorphically substituted by trivalent ones. Positive charge of layers is balanced by anions located in interlamellar space. The general formula of LDH is $[\mathbf{M}(\mathbf{II})_{1-\mathbf{x}} \ \mathbf{M}(\mathbf{III})_{\mathbf{x}} \ (\mathbf{OH})_2]^{\mathbf{x}+} [\mathbf{A}^{\mathbf{n}-}_{\mathbf{x}/\mathbf{n}} \cdot \mathbf{y} \mathbf{H}_2 \mathbf{O}]^{\mathbf{x}-}$, where M(II) and M(III) are divalent and trivalent metal cations respectively, and $\mathbf{A}^{\mathbf{n}-}$ is n-valent anion. Incorporation of transition and rare earth metal cations, such as samarium, in layered structure may allow obtaining materials with specific optical, electronic and magnetic properties.

Among lanthanide containing LDHs samarium-doped materials are some of the rarest. Meanwhile, samarium loaded compounds were reported to demonstrate photocatalytic properties (Bellardita et al., Appl. Catal. B Environmental 104 (2011) 291).

SYNTHESIS PROCEDURE

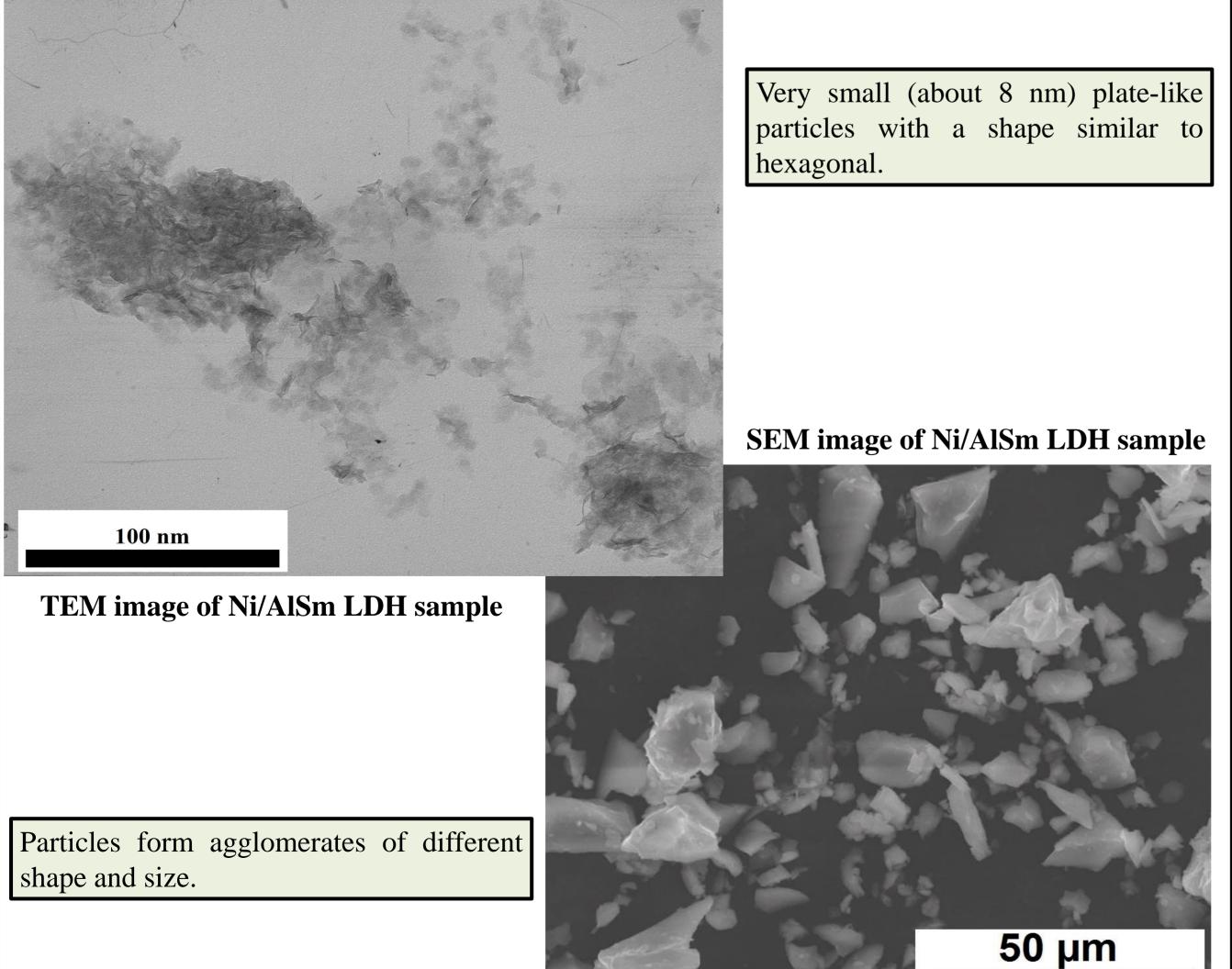
The Ni/AlSm hydrotalcite-like material was obtained by coprecipitation method followed by hydrothermal treatment. Reagents were taken in proportions to form the compound with molar ratio M(II)/M(III) = 3 and trivalent cations molar ratio $Sm^{3+}/(Al^{3+} + Sm^{3+}) = 0.05$. During the synthesis, the aqueous solution of corresponding nitrates was mixed under vigorous stirring with solution of sodium hydroxide NaOH. Then the resulting mixture was transferred into autoclave reactor for 30 h at 120 °C. The synthesized material was centrifuged, washed with distilled water and dried.

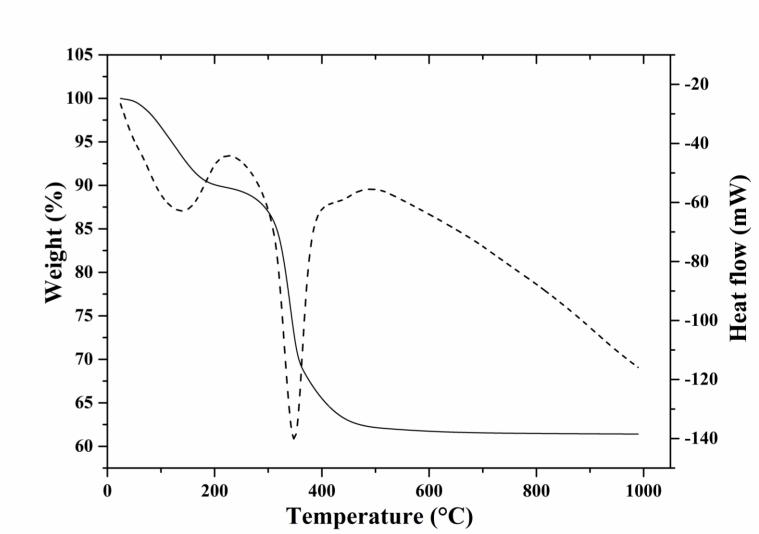


	Unit cell parameters		
	a, Å	c, Å	
Hydrotalcite	3,04	22,90	
Ni/Al-LDH	3,04	23,13	
Ni/AlSm-LDH	3,06	23,86	

EDX data on elemental composition of Ni/AlSm hydrotalcite-like compound

	Molar ratio of cations			Me ²⁺ /Me ³⁺ ratio	Percentage of aluminum
Sample	Co ²⁺	Al ³⁺	Sm ³⁺		ions substitution, %
Ni/AlSm	0,750	0,238	0,012	3	4,8





TGA-DSC curves of Ni/AlSm hydrotalcite-like compound (solid curve refers to "Weight" and the dashed one to "Heat flow")

Conclusion

Synthesized Ni/AlSm hydrotalcite-like compound seems single phase, but its crystallinity is mediocre. To the best of our knowledge, it is the first time when LDH with such composition is presented. The presence of samarium is proved by results of EDX analysis. Cations molar ratios are almost the same as predetermined. Morphology of the obtained sample is investigated with transmission and scanning electron microscopy. Thermal behavior of the compound is quite typical for hydrotalcite-like structures.

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