

STRUCTURE, MORPHOLOGY AND THERMAL BEHAVIOR OF SILVER-DOPED NANOPARTICLES FORMED IN $\text{La}_2(\text{SO}_4)_3$ AND LaCl_3 SYSTEMS

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Abstract

The formation of structures in systems of lanthanum sulphate and chloride in the presence of silver nitrate was studied by XRD, TG–DTA and SEM/EDS methods. TG–DTA analysis showed the formation of hexagonal structure $\text{La}(\text{OH})_3$ and its transformation into an unstable compound of monoclinic lanthanum (oxy)hydroxide in the temperature range 260 – 300°C, dehydroxilation of (oxy)hydroxide's lattice at temperatures of 420 – 440°C. The subsequent ordering of the lanthanum oxide structure and the destruction of the substances included in the system took place at temperatures of 600 – 830°C and the removing of silver clusters – at 920°C. It is shown that at $T = 400 - 600^\circ\text{C}$ the well-crystallized powders formation of lanthanum oxide La_2O_3 (space group $P3m1$ corresponds to a trigonal system) takes place, which includes elements of cubic phases La_2O_3 (space group $Ia3$) and LaO (space group F) in its structure. The presence of silver cations in the systems during the calcination process leads to their reduction on the surface of lanthanum oxide clusters. Morphological study indicates the heterogeneity of the powders and the distortion of the (hexagonal) shape of lanthanum oxide crystals to spherical that is typical for lanthanum chloride systems. In addition to the main elements (oxygen, lanthanum and silver if present in the system) the composition of the powders contains the elements that make up the anions of the initial salts (sulphur, chlorine, nitrogen), as well as the residual elements (sodium and potassium) of additional substances.

1. Introduction

Today, materials based on oxides and hydroxides of rare earth elements are widely used for the production of functional materials for technical and biomedical purposes. In particular, lanthanum oxide is used in the manufacture of optical glasses, phosphors, photocathodes, catalysts, light-emitting and laser materials, and hydrogen storage materials [1]. Lanthanum is introduced as an alloying additive in magnesium and aluminum alloys, it is used as a structure-forming agent [2]. Due to its catalytic properties, lanthanum oxide is used for the flameless methane combustion [3], in the process of graphite oxidation [4], for oxidative coupling of methane [5]. Also, lanthanum oxide is used to produce magnetic and electrical materials, automobile exhaust-gas convectors, ceramic superconductors, as indicated in the work [6]. Lanthanum hydroxide can serve as a catalyzer and sorbent. The loading ultralow amount of Pd

into lanthanum hydroxide structure enhances the catalytic activity for heterogeneous catalytic applications, namely, hydrogenation and cross-coupling reactions [7].

At the same time, silver-doped lanthanum oxide showed not only anti-oxidant activity, but also helped prevent agglomeration of silver particles. Ag–La₂O₃ composites exhibited certain scavenging ability against the hydroxyl radical and demonstrate an excellent antibacterial ability against the gram-positive *S. aureus* and gram-negative *E. coli* in vitro. In this connection, these composites are considered as a promising material for the production of an anti-oxidative and antibacterial agent in medical and food [8]. Preliminary studies have shown that silver-modified lanthanum oxide particles formed via chemical synthesis exhibit bactericidal activity and are considered as a potential material for creating antiviral protection agents [9 – 11].

It should be noted that when creating high-quality materials with adjustable properties, including those based on rare earths, an important issue is to study the phase transformations of their compounds under the influence of temperature, as indicated in [12]. In general, a number of works are devoted to the study of the processes of thermal conversion of lanthanum hydroxide and oxide [13, 14]. And, despite the fact that today the two-step mechanism of phase transformations $\text{La}(\text{OH})_3 \rightarrow \text{LaOOH} + \text{H}_2\text{O}$ and $\text{LaOOH} \rightarrow \text{La}_2\text{O}_3 + \text{H}_2\text{O}$ has been studied and proven by [15], the temperature of phase transformations, the composition and morphology of the obtained structures can be influenced by many factors, for example, the composition of the starting salts, auxiliary reagents, doping components. In particular, [16] shows that KOH and NaOH have different influences on crystallinity, morphology and thermal behavior of the obtained La(OH)₃ powders. Thus, despite the extensive study of the phase formation process in pure systems hydroxide–oxyhydroxide–lanthanum oxide, the introduction of dopants into the system can have a significant effect on the temperature of phase transformations, morphology and composition of the final reaction products.

In this connection, the purpose of this work is the comparative study of the phase composition, thermal behavior and morphology of the nanosized structures formed in pure and silver-bearing lanthanum chloride and lanthanum sulphate systems by the precipitation and calcination processes.

2. Research objects and methods

Particle' synthesis was performed via precipitation of LaCl₃ and La₂(SO₄)₃ chemically pure salt solutions in low alkaline medium in pure and Ag-containing systems. Hydrolysis regulator, nucleating and reducing agents were used as the adding substances to perform synthesis procedure. The concentration of AgNO₃ in the initial solutions was set based on 2, 4 and 5wt.% Ag in the nanoparticle's composition. The precipitates were rinsed by water and ethanol–water solutions several times and dried at T = 160°C. Wet precipitates were calcined at T = 400°C in 5 h, at T = 600 and 1000°C for 2 h.

The samples were visualized by scanning electron microscopy (SEM) method. Electron micrographs of the samples were recorded on a MIRA3 TESCAN scanning electron microscope. Determination of the phase composition of the obtained samples was performed by the method of powder X-ray diffraction phase analysis using DRON–3 device with radiation of the copper anode (Cu K_α). The scanning step was 0.05 – 0.10°, exposure – 4 s, range of 2θ angles – from 15 to 90°. Samples were taken at standard temperature. The International Powder Standards Committee (JSPDS International Centre for Diffraction Data 1999) database was used to

determine the phase composition. Calculation of the lattice parameters was performed according to standard methods. A simultaneous study of thermogravimetric and differential thermal properties (TG–DTA) of the composite particles were performed in the static air atmosphere by a derivatograph Q–1500D (Hungary). The parameters of the pattern recording were: the samples 200 mg were heated at the rate 10°C/min from 20 to 1000°C; the sensitivity was 20 mg; TG – 500, DTG – 500, and DTA – 250. The samples were placed into a corundum crucible and covered by a quartz beaker to create a homogenous temperature field.

3. Result and discussion

3.1. X-ray diffraction study

The main structure obtained in $\text{La}_2(\text{SO}_4)_3\text{--AgNO}_3$ system is lanthanum hydroxide $\text{La}(\text{OH})_3$ (#36–1481) but the next lyophilization of the precipitate at $T = 160^\circ\text{C}$ promotes the beginning of dehydroxylation process and lead to phase transformation of $\text{La}(\text{OH})_3$ into lanthanum oxides. Also, crystal lattice's reflexes of cubic body-centered $\text{Ia}3$ (#22–0369) La_2O_3 and face-centered F (#33–0716) LaO are seen in the XRD pattern (**Figure 1a**). At the same time, weak reflexes of silver and residual peaks of AgNO_3 salt (#8–0349) can be traced in the XRD pattern. The formation of trigonal $P3m1$ (#05–0602) La_2O_3 oxide is not excluded. Calcining the precipitate leads to an increase in the degree of crystallinity of the sample (**Figure 1b**).

It is significant that the precipitate heating obtained in a system of pure lanthanum sulphate at $T = 400^\circ\text{C}$ for 5 h does not lead to the formation of a well-crystallized precipitate of lanthanum oxide (**Figure 2b**). Only weak reflexes of La_2O_3 (#05–0602) and body-centered La_2O_3 (#22–0369) lanthanum oxides are seen in the XRD pattern. A powder of lanthanum hydroxide and oxide ($P3m1$) with a reduced silver cluster is formed, when 2wt.% Ag is injected into the system and the precipitate is heated at 400°C during 5 h (**Figure 1c**). The increase in the silver concentration to 4wt.% and the treatment the precipitate under the same condition leads to increase the crystallinity degree of the precipitate. Two main phases: trigonal La_2O_3 (#05–0602) and cubic Ag^0 (#4–0783) are present in the powder. Probably, silver clusters are reduced on the lanthanum oxide surface. In addition, the structural elements of face-centered LaO (#33–0716) are detected in small reflexes in the XRD pattern.

The hydroxylamine hydrochloric acid solution injection as a reducing agent into the system does not change the XRD pattern result. Crystal structures of trigonal La_2O_3 , face-centered LaO and cubic Ag^0 are clearly seen in the XRD pattern (**Figure 1c**). It is significant that, in the absence of silver nitrate, in the precipitate calcined at 400°C , the intensity of reflections is insignificant (**Figure 1d**). It's phase composition includes trigonal La_2O_3 (#05–0602) and body-centered La_2O_3 (#22–0369) formations. The following heating of the sample of pure $\text{La}_2(\text{SO}_4)_3$ system at 1000°C results in the formation only trigonal La_2O_3 (#05–0602) particles (**Figure 1e**), whereas trigonal La_2O_3 (#05–0602) and weak (residual) Ag^0 (#4–0783) clusters are formed in $\text{La}_2(\text{SO}_4)_3\text{--AgNO}_3$ system at the same heating temperature (**Figure 1f**).

The analysis the precipitates formed in pure LaCl_3 and Ag-bearing $\text{LaCl}_3\text{--AgNO}_3$ systems and calcined at 600°C indicates the complicated three phases formation in the powder trigonal La_2O_3 (#05–0602), cubic body-centered $\text{Ia}3$ La_2O_3 (#22–0369) and face-centered F LaO (#33–0716) (**Figure 1g**). In the presence of reducing agent the phase composition of the powder does not change. But when AgNO_3 was added in the initial precipitate the reflexes of silver phase Ag^0 (#4–0783) are appeared in the XRD pattern (**Figure 1h**).

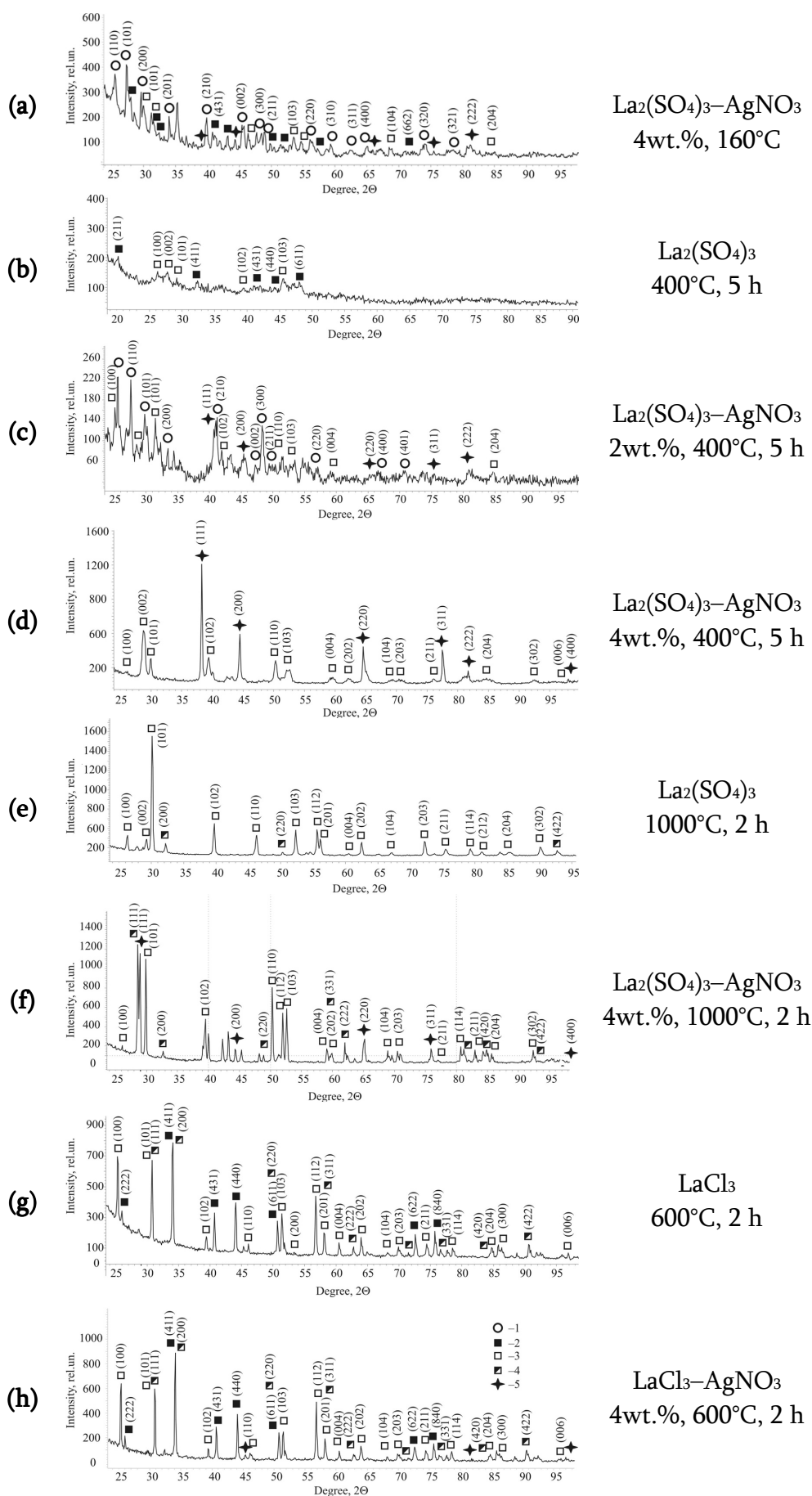


Figure 1. XRD-patterns of powders formed in $\text{La}_2(\text{SO}_4)_3$ and LaCl_3 systems. Mineral phases: 1 – $\text{La}(\text{OH})_3$ (P63/m), 2 – La_2O_3 (P3m1), 3 – La_2O_3 (Ia3), 4 – LaO (F) and 5 – Ag^0 (C).

The calculation of the crystal lattice parameter of body-centered $\text{Ia}\bar{3}$ La_2O_3 (#22–0369) by three planes (222), (400) and (431) shows its invariability for both systems: $a = 11.096$ nm for $\text{La}_2(\text{SO}_4)_3$ and $a = 11.322$ nm for LaCl_3 . This situation is typical for the LaO F phase, where calculation of parameter for (111), (220) and (311) planes show $a = 5.147$ nm for $\text{La}_2(\text{SO}_4)_3$ and $a = 5.082$ nm for LaCl_3 . Based on the obtained result, it can be assumed that these oxides are structural elements of trigonal lanthanum oxide, and not separate phases. At the same time, the calculation of the parameters of the crystal lattice of trigonal (hexagonal) lanthanum oxide La_2O_3 (#05–0602) showed their significant difference from the values declared in the JSPDS International Center for Diffraction Data 1999 ($a = 3.937$ and $c = 6.129$ nm), which may indicate a distortion of the crystal lattice of this structure.

3.2. Thermal-gravimetric analysis (TG–DTA)

Thermal gravimetric analysis of the powder obtained in the pure LaCl_3 system shows the first endothermic peak at $T = 270^\circ\text{C}$ and shoulder at $T = 300^\circ\text{C}$ in the DTA curve accompanied with weight loss at $T = 200^\circ\text{C}$ (DTG curve) that may be related to the formation of intermediate phase $\text{LaCl}(\text{OH})_2$ and $\text{La}(\text{OH})_3$ phase transformation into $\text{LaO}(\text{OH})$. The second effect of weight loss in DTG curve points to the dehydroxilation of lanthanum oxy(hydroxide) lattice and the formation of lanthanum oxide phases (endothermic peak at 390°C). The next shoulder in DTA curve at the temperature $710 - 735^\circ\text{C}$ may be related to transformation of the crystal lattice of lanthanum oxides ($\text{Ia}\bar{3}$ into $\text{P}3\text{m}1$) and oxidation of chlorides. Mass loss effect is described by DTG in the temperature range $750 - 860^\circ\text{C}$. Last small effect of mass loss in the DTG curve is detected at $T = 950^\circ\text{C}$ may be due to remove chloride. The total mass lost of the sample is 17.8%. In the presence of Ag (**Figure 2b**) the first effect of mass loss is not appeared but the second one belongs to corresponding endothermic reflexes at temperatures 260 and 440°C indicated the phase transformation of $\text{La}(\text{OH})_3$ into $\text{LaO}(\text{OH})$ and dehydroxilation of $\text{LaO}(\text{OH})$. The mass loss effect at 640°C may be corresponded to AgNO_3 destruction, whereas the mass loss effect at $T = 710 - 830^\circ\text{C}$ relates to chloride oxidation. The total mass loss equals 15.8%.

The analysis of thermal behavior of pure lanthanum sulphate system indicated deep reflex of mass loss at $T = 360^\circ\text{C}$ and endothermic peak at $T = 370^\circ\text{C}$ corresponded to sulphate destruction. The processes of phase transformation lanthanum hydroxide into lanthanum oxyhydroxide and lanthanum oxyhydroxide destruction carried out at $T = 230$ and 420°C correspondingly. The next reflexes seen at $T = 600 - 610^\circ\text{C}$ can indicate the formation of lanthanum oxide phase. The total mass loss is 13.7%. Two endothermic reflexes on the DTA curve of the powder obtained in $\text{La}_2(\text{SO}_4)_3\text{--AgNO}_3$ system (**Figure 2d**) indicates the formation of $\text{LaO}(\text{OH})$ phase from $\text{La}(\text{OH})_3$, dehydroxilation of its crystal lattice at a temperature of $\sim 300^\circ\text{C}$ and probable destruction of sulphates at a temperature of $\sim 340^\circ\text{C}$. The phase interaction of lanthanum oxide with silver ends at the $T \sim 400^\circ\text{C}$. The endothermic peak at $T = 900^\circ\text{C}$ can be attributed to the change in the crystalline modification of lanthanum oxide as well as melting silver. The DTG curve shows a two-stage weight loss, which characterizes the destruction of lanthanum and silver hydroxides (250°C) and the removal of sulphates ($\sim 340^\circ\text{C}$), respectively. Destruction of nitrate is observed at $T = 380^\circ\text{C}$. According to the TG, the total weight loss is 21.6%. The preliminary preparation the samples were subjected to heat treatment at $T = 160^\circ\text{C}$, hence, there is no reflex on the DTG curve, which characterizes the loss of adsorption-bound water.

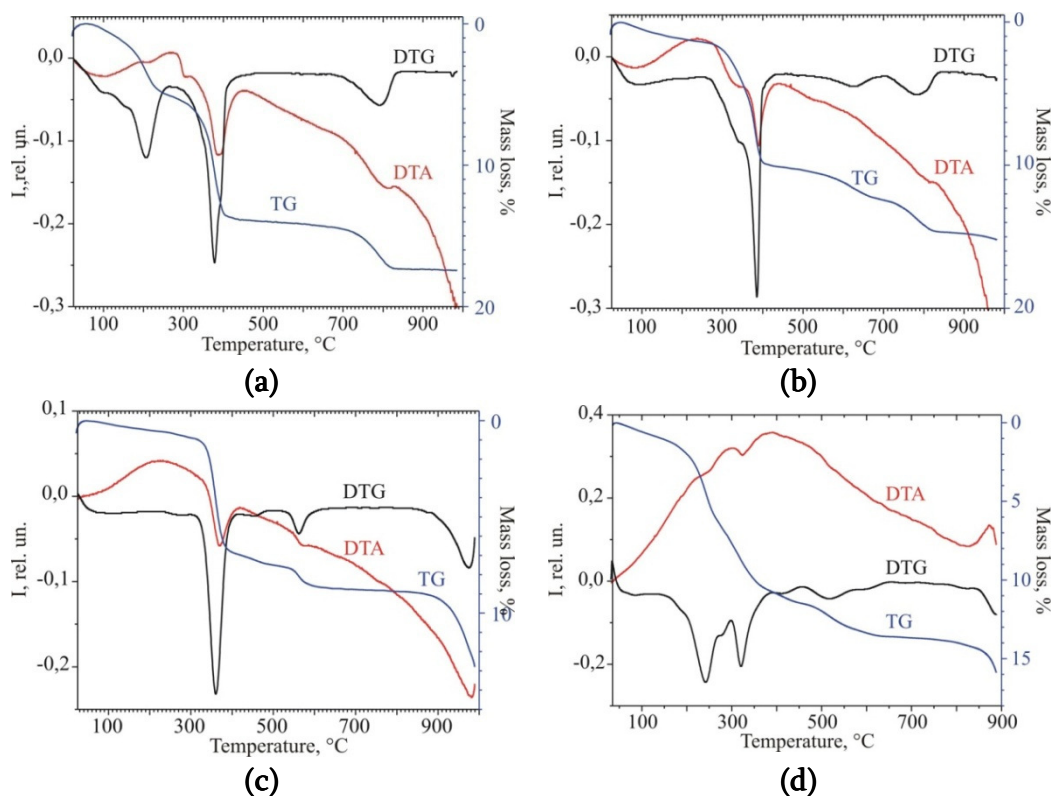


Figure 2. TG–DTA curves of resuspendable precipitates formed in lanthanum-containing systems: **(a)** LaCl_3 , **(b)** $\text{LaCl}_3\text{--AgNO}_3$, **(c)** $\text{La}_2(\text{SO}_4)_3$ and **(d)** $\text{La}_2(\text{SO}_4)_3\text{--AgNO}_3$.

3.3. SEM–EDS analysis

The visualization of the samples was performed using scanning electron microscopy. The SEM images of the sediments and calcined powders are present in **Figure 3**. Analysis of the $\text{La}_2(\text{SO}_4)_3$ system unambiguously indicates that at a relatively low calcination temperature of the samples, the powders are characterized by a heterogeneous composition and imperfect structure (**Figure 3a**). The degree of crystallinity and homogeneity of the powder increases in the presence of silver (**Figures 3b – 3e**). As the silver concentration increases, silver clusters appear on the surface of lanthanum oxide crystals (**Figure 3e**). Crystals of lanthanum oxide formed in the lanthanum chloride system are distinguished by smoothed edges and a shape tending to spherical (**Figures 3f – 3k**). Moreover, the structures of the chloride system are more homogeneous in comparison with the structures of the sulfate system.

EDS spectra are present in **Table 1**. The obtained data indicate that the samples are partly homogeneous in chemical composition. Carrying out the synthesis procedure without introducing additional substances promotes the structures' formation with containing the main elements: oxygen, lanthanum and, accordingly, silver when it is introduced into the initial suspension.

As additional elements included in inorganic salt anions are identified sulphur or chlorine, as well as potassium and sodium, which act as a precipitant and nucleating agents. In this case, the use of a reducing agent (hydroxylamine hydrochloric acid) leads to an increase in the number of elements that are included in the powder, and influences its quality for subsequent usage (e.g., in biomedicine) consequently.

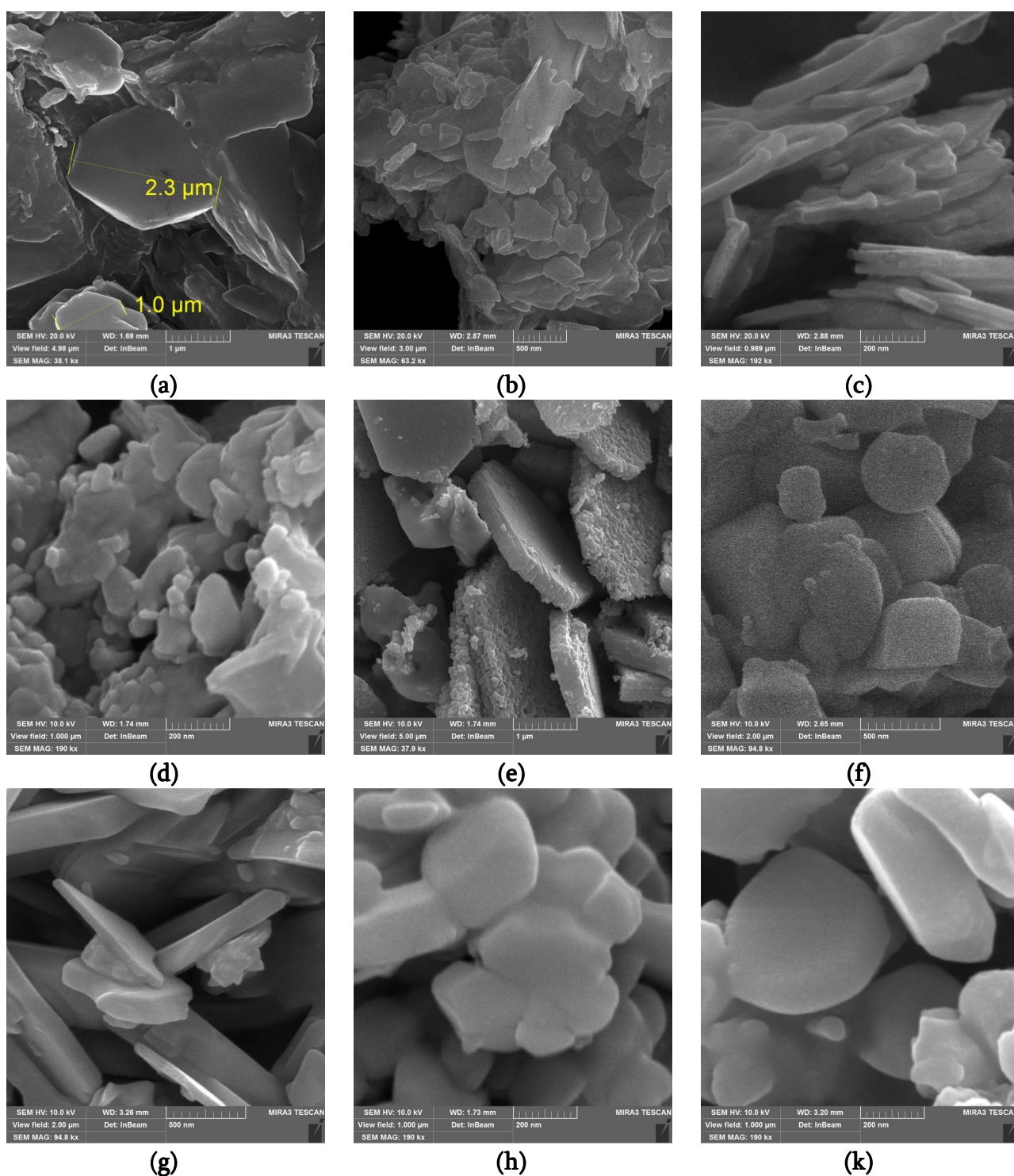


Figure 3. SEM images of powders formed in $\text{La}_2(\text{SO}_4)_3$ systems (400°C , 5 h): (a) $\text{La}_2(\text{SO}_4)_3$, (b) $\text{La}_2(\text{SO}_4)_3\&\text{Ag}(2\text{wt.}\%)$, (c) $\text{La}_2(\text{SO}_4)_3\&\text{Ag}(4\text{wt.}\%)$ and (d) $\text{La}_2(\text{SO}_4)_3\&\text{Ag}(4\text{wt.}\%)$ under reductive conditions and (e) $\text{La}_2(\text{SO}_4)_3\&\text{Ag}(>5\text{wt.}\%)$ under reductive conditions; and in LaCl_3 systems (600°C , 2 h): (f) LaCl_3 , (g) LaCl_3 under reductive conditions, (h) $\text{LaCl}_3\&\text{Ag}(4\text{wt.}\%)$ and (k) $\text{LaCl}_3\&\text{Ag}(4\text{wt.}\%)$ under reductive conditions.

Table 1. EDS distribution of chemical elements (wt.%) in nanopowder compositions formed in $\text{La}_2(\text{SO}_4)_3$ and LaCl_3 systems.

Spectrum	Element, wt. %								Total
	O	S	Cl	K	Na	Ag	La	N	
La ₂ (SO ₄) ₃ (400°C, 5 h)									
1	25.71	—	—	—	25.71	—	62.95	25.71	100
2	31.38	—	—	—	31.38	—	55.41	31.38	100
La ₂ (SO ₄) ₃ &Ag(4wt. %) (400°C, 5 h)									
3	40.14	12.09	—	0.35	—	0.25	47.17	—	100
4	39.31	14.57	—	0.47	—	1.05	44.60	—	100
5	35.62	11.81	—	0.62	—	1.26	50.70	—	100
La ₂ (SO ₄) ₃ &Ag(4wt. %) (400°C, 5 h, reductive conditions)									
6	15.67	0.80	14.63	1.03	—	1.56	66.31	—	100
7	18.22	1.01	14.46	0.84	—	1.94	63.53	—	100
8	16.74	2.17	11.27	1.04	—	1.68	67.10	—	100
LaCl ₃ (600°C, 2 h)									
9	7.25	—	21.09	—	—	—	71.66	—	100
10	7.73	—	21.30	—	—	—	70.97	—	100
11	7.52	—	20.36	—	—	—	72.13	—	100
LaCl ₃ (600°C, 2 h, reductive conditions)									
12	5.82	—	17.68	0.77	—	—	75.72	—	100
13	5.90	—	17.06	—	—	—	77.05	—	100
LaCl ₃ &Ag (600°C, 2 h)									
14	4.70	—	23.06	—	—	2.50	69.74	—	100
15	6.22	—	19.95	—	—	3.70	70.13	—	100
LaCl ₃ &Ag (600°C, 2 h, reductive conditions)									
16	15.67	0.80	14.63	1.03	—	1.56	66.31	—	100
17	18.22	1.01	14.46	0.84	—	1.94	63.53	—	100
18	16.74	2.17	11.27	1.04	—	1.68	67.10	—	100

Thus, our studies have shown the effect of the anionic composition (sulfate and chloride) of the initial lanthanum-containing solution and the temperature of subsequent treatment of the precipitate on the phase composition, temperature of phase transformations, and morphology of lanthanum oxide powders.

In general, the introduction of silver nitrate into the system promotes an increase in the degree of crystallinity of the precipitate and the formation of lanthanum oxide particles with silver clusters reduced on its surface.

It was shown by XRD that weakly crystallized particles of lanthanum hydroxide are formed upon lyophilization of the suspension at $T = 160^\circ\text{C}$. Further temperature treatment of the precipitate leads to dehydroxylation of the hydroxide and its transformation into the oxide phase.

4. Conclusion

The study of the phase formation process in pure systems and doped (2 – 5wt.%) with silver systems of lanthanum chloride and lanthanum sulphate showed an increase in the degree of crystallinity of the samples obtained in the presence of silver. While the precipitate of the pure lanthanum sulphate system after calcination at 400°C for 5 h is characterized by weak crystallinity, the injection of 4wt.% silver leads to the formation of well-crystallized lanthanum oxide (space group P3m1, trigonal system), including the structural elements of the cubic phases La₂O₃ (Ia3) and LaO (F), that distorts the lanthanum oxide crystal lattice and is reflected in the morphology of particles. Under these conditions, silver is reduced on the La₂O₃ surface in the form of metal clusters. The reducing agent adding to the systems does not change the phase composition of the samples, while an increase in the calcination temperature to 1000°C leads to the silver clusters melting.

The composite structure formation based on lanthanum oxide doped with silver was recorded at T = 400 – 600°C after calcining the precipitate for 2 – 5 h. Thermogravimetric analysis showed that the temperature of the phase transformation in the series lanthanum hydroxide–oxyhydroxide–oxide varies depending on the anionic composition of the initial lanthanum salt and the presence or absence of silver: it equals 260 – 300°C for La(OH)₃–LaO(OH), 420 – 440°C for dehydroxilation of (oxy)hydroxide's lattice, and 600 – 830°C for ordering of the lanthanum oxide structure. The morphological study of the powders showed a distortion of the crystal lattice of lanthanum oxide and the amorphization of some of its crystal faces. The lanthanum chloride system is distinguished by smoothing the edges and approaching the particle shape to spherical. SEM–EDS analysis shows the additive elements presence (in addition to the basic elements La, O and Ag, when it was introduced into the system) included in the anionic species and which were used as nucleating agents and reducing agents.

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