

SYNTHESIS, STRUCTURE AND MORPHOLOGY OF SmMnO_3 - LaMnO_3 PEROVSKITE COMPOSITE

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Abstract: This article discusses the synthesis, phase composition, and structural morphology of the LaMnO_3 - SmMnO_3 double- phase manganite perovskite composite. The aim of synthesizing a two-phase manganite containing the rare earth elements samarium and lanthanum is to study the structural features and properties of the resulting products compared to classical perovskite manganites and single-phase samarium and lanthanum manganites. The Pechini method, based on sol-gel synthesis, was used to synthesize the product. The purpose of selecting this method was to obtain a product with high fineness and crystallographic purity. The phase composition and crystallographic parameters were analyzed using X-ray diffraction (XRD), Fourier-transform infrared (FTIR), and morphological methods to determine the formation of bonds between Me-O; scanning electron microscopy (SEM-EDS), scanning surface area, and elemental distribution. The synthesis was carried out based on sol-gel formation in the presence of metal nitrate hydrates. Phase analysis of the product showed that SmMnO_3 has the orthorhombic Pbnm (62) structure in accordance with PDF 00-025-0747, while LaMnO_3 corresponds to the rhombohedral space group R-3C (167) in accordance with PDF card No. 01-090-6505. It was established that the intense absorption bands in the FTIR spectrum lie within the range of $750\text{--}600\text{ cm}^{-1}$, indicating the formation of Mn–O bonds. SEM-EDS results show that the average particle size of the powdered product is $0.489\text{ }\mu\text{m}$, and the product was successfully characterized using the elemental distribution map.

Keywords: perovskite, manganite, Pechini method, XRD, SEM-EDS analysis

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1. Introduction

Perovskite manganites are a class of transition metal oxides with the general chemical formula ABO_3 ($RMnO_3$ for rare-earth variants), where the A-site is occupied by trivalent rare-earth cations (La^{3+} , Nd^{3+} , Sm^{3+}) or divalent cations of alkaline earth elements (Ca^{2+} , Sr^{2+} , Ba^{2+}), while manganese (Mn) ions occupy the B sites [1–3]. Their structure consists of a three-dimensional framework of MnO_6 octahedra, the vertices of which are shared, while the cation at the A-site occupies interstitial spaces [4–6]. These materials are model systems for studying strong electronic correlations, exhibiting a wide range of electronic and magnetic phases arising from complex interactions between spin, charge, lattice, and orbital structure [5,7]. Depending on their composition and temperature, they may exhibit ferromagnetism (FM), antiferromagnetism (AFM), or paramagnetism (PM) [5,8–10]. It is known for its phenomenon of giant magnetoresistance, in which electrical resistance decreases sharply under the influence of an applied magnetic field [2,9]. In contrast, it exhibits metal-insulator transitions and semimetallic properties, making it important for spintronics [7,9,11]. Physical properties of manganites are primarily determined by two competing exchange interactions involving manganese ions: double-exchange (DE) interaction and super-exchange (SE) interaction. DE interactions occur between Mn^{3+}/Mn^{4+} ions via oxygen 2p orbitals. Electron transitions between these mixed valence states contribute to both FM and metallic conductivity [2,9,11,12]. SE CAO interactions typically give rise to antiferromagnetic insulating states, examples of which are the stoichiometric compounds $CaMnO_3$ or $LaMnO_3$ [2,3,10].

Lanthanum manganite ($LaMnO_3$) is a typical perovskite oxide (ABO_3) characterized by a flexible structure, tunable electronic states, and magnetic properties. Stoichiometric $LaMnO_3$ is an antiferromagnetic insulator, but FE can be achieved by incorporating other metals into its composition or by introducing an excess or a small amount of oxygen [8,11]. These modifications are suitable for use in supercapacitors, electrocatalysis, MRI contrast agents, and environmental remediation [1,13,14]. Researchers have employed various strategies to overcome challenges such as particle agglomeration and low conductivity. One of these is the diversity of synthesis types and the choice of synthesis method. Hydrothermal synthesis is used to create $LaMnO_3$ nanoparticles, which are combined with MXene nanoparticles to improve charge transport and increase the surface area [1]. Mesoporous silica (SBA-15) is used to produce a nanoscale product, enhancing oxygen adsorption on the surface and creating more active sites for catalytic reactions. Sol-gel synthesis is a cost-effective and rapid method that often uses oxalic or citric acid as a precursor to produce fine magnetic nanoparticles [13,15,16]. Although doped $LaMnO_3$ perovskites are of great importance for the development of clean energy and electronics, issues related to the formation of secondary phases, electrolyte reactivity, and high-temperature stability have not yet been fully resolved.

$SmMnO_3$ is characterized by properties such as being one of the best-known perovskite manganites, exhibiting colossal magnetoresistance (CMR), and

possessing complex spin textures, making it a promising material for the development of next-generation sensor, memory, and spintronic devices [17,18]. SmMnO_3 crystallizes primarily in the orthorhombic perovskite structure (space group Pbnm). Substitution of samarium with other ions, such as strontium (Sr) [17], europium (Eu), or yttrium (Y), leads to significant changes in the lattice structure. Eu or Y dopants lead to lattice contraction and a decrease in lattice constants. Calculations of the Goldschmidt tolerance coefficient (0.9) and the cohesive energy confirm that the structure remains stable even at high doping levels (up to 25% for Eu and Y). Mechanical analysis shows that SmMnO_3 is flexible, as evidenced by a G/B ratio below 0.57. Y doping increases the material's hardness (Young's modulus), while Eu doping typically reduces it [19]. From highly efficient supercapacitors to multiferroic devices operating at room temperature, SmMnO_3 is a key product of innovation in materials science and functional electronics [18,20,21].

This study aims to synthesize the biphasic perovskite manganite LaMnO_3 - SmMnO_3 and to investigate the effect of the phase mixture on structural and morphological properties compared to the individual components LaMnO_3 and SmMnO_3 . Particular attention is paid to lattice distortion, phase formation, particle morphology, and the potential improvement in functional properties associated with the coexistence of orthorhombic and rhombohedral phases, using FTIR, XRD, and SEM-EDS methods.

2. Experimental part

Dual-phase perovskite manganites (LaMnO_3 - SmMnO_3) were first synthesized separately using the Pechini method [22] and weighed in a 1:1 ratio to obtain a homogeneous composite; they were then mixed in a ball mill in the presence of ethanol for 15 minutes. For the synthesis of LaMnO_3 , 10 mmol of lanthanum(III) nitrate $\text{La}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ (4.33 g) and 10 mmol of manganese(II) nitrate $\text{Mn}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$ (2.51 g) were used. To prepare SmMnO_3 , 10 mmol of samarium(III) nitrate $\text{Sm}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ (4.45 g) and 10 mmol of manganese(II) nitrate $\text{Mn}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$ (2.51 g) were used. The starting salts were dissolved in 10 mL of distilled water with constant stirring on a magnetic stirrer. Citric acid (1.92 g, 10 mmol) and ethylene glycol (0.93 mL, 15 mmol) were then added to the solutions in a molar ratio of metal: citric acid: ethylene glycol = 1:1:1.5. The resulting mixture was held at a temperature of 70-90 °C for 15 minutes until a gel-like mass formed. The resulting gel was dried at 200 °C for 5 hours, after which it was heat-treated in the temperature range of 600-1100 °C to form a perovskite structure. LaMnO_3 and SmMnO_3 powders were first prepared separately and then mixed in a 1:1 mass ratio to form a two-phase composite. To do this, 1 g of each powder was taken and mechanically ground in a ball mill in the presence of three drops of ethanol for 15 minutes to achieve a homogeneous phase distribution. The resulting wet powder was further calcined at 800 °C for 2 hours to remove residual organic components and increase the crystallinity of the composite. We

analyzed the two-phase perovskite using FTIR, XRD, and SEM-EDS to confirm that manganites had been obtained.

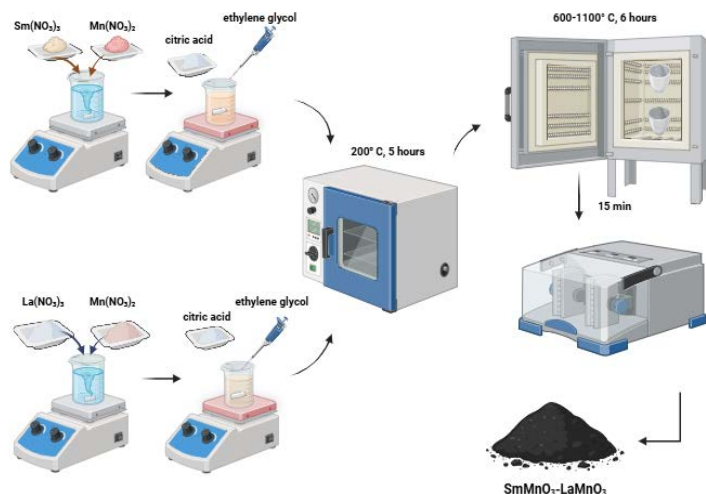


Figure 1 – Synthesis of a two-phase perovskite manganite.

Reagents used to prepare the two-phase product: manganese(II) nitrate ($\text{Mn}(\text{NO}_3)_2 \cdot x\text{H}_2\text{O}$, Buchs, Switzerland); lanthanum(III) nitrate hexahydrate ($\text{La}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$, TU 6-09-4676-83); citric acid ($\text{C}_6\text{H}_8\text{O}_7$, GOST 908-79); ethyl alcohol, 96% (medical grade) and ethylene glycol ($\text{C}_2\text{H}_6\text{O}_2$) (GOST 10164-75); samarium nitrate ($\text{Sm}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$, Ch.P, GOST 10928-90).

We used the following measuring instruments and methods: a laboratory agate (diameter-13 cm (130 mm)) (QIANGFU, ASIN, B0DNB1MQ28, China), an AM vibrating ball mill (China), and a “SNOL” laboratory furnace. The phase composition was determined using X-ray diffraction (Rigaku Miniflex 600 benchtop X-ray diffractometer (Rigaku, Tokyo, Japan)) and a field-emission scanning electron microscope (Thermo Scientific ChemSEM Axia, USA). IR spectroscopy was performed using a Bruker ALPHA FTIR spectrometer in the range of $400\text{--}4000\text{ cm}^{-1}$.

3. Results and discussion

3.1. XRD analysis

The formation of biphasic perovskite manganites was confirmed by determining the crystallographic parameters and phase composition of the resulting product using X-ray phase analysis (XRD). Measurements were performed using a Rigaku Miniflex 600 X-ray diffractometer, and the parameters of the crystalline phase were analyzed using the PDF-5 software.

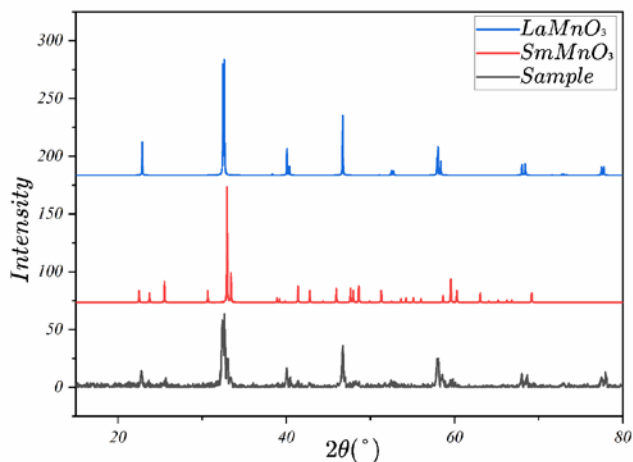


Figure 2 – LaMnO_3 - SmMnO_3 diffractogram.

Figure 2 shows synthesized two-phase perovskite manganites and the diffraction patterns of samarium manganite and lanthanum manganite. The sharpness of the diffraction peaks and the retention of peaks characteristic of the dual phase in the composite indicate the formation of the product. Samarium manganite crystallizes in orthorhombic space group $Pbnm$ [62] according to PDF 00-025-0747, while lanthanum manganite forms rhombohedral $R\text{-}3C$ (167) structure according to PDF 01-090-6505 (table 1).

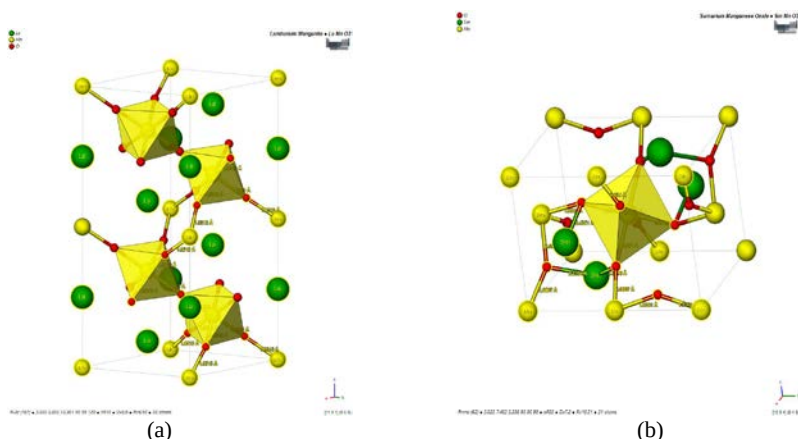


Figure 3 – Phase structures of LaMnO_3 (a) and SmMnO_3 (b).

Figure shows the crystal structure of the perovskite-like compounds LaMnO_3 and SmMnO_3 . In both phases, manganese atoms coordinate with oxygen atoms to form MnO_6 clusters and a 3D perovskite lattice. The lattice exhibits significant octahedral distortion due to the fact that the radius of the Sm^{3+} ion is smaller than

that of the La^{3+} ion, and also because LaMnO_3 structure is relatively symmetric, resulting in the formation of a rhombohedral structure.

Table 1 – Results of crystallographic quantitative analysis

№	Name of compound	Crystal System	a, Å	b, Å	c, Å	V, (Å ³)	Z	PDF Number	Space Group	Density $\rho_{\text{x-rays}}$ g/cm ³
1	SmMnO_3	Orthorhombic	5.825	7.483	5.358	233.55	4	00-025-0747	Pbnm (62)	7.20
2	LaMnO_3	Rhombohedral	5.511	5.511	13.396	352.27	6	01-090-6505	R-3c (167)	6.84

Table 1 lists the crystallographic parameters of two-phase product. Lattice parameters of SmMnO_3 are $a = 5.825$ Å, $b = 7.483$ Å, and $c = 5.358$ Å, with a unit cell volume of 233.55 cm³. Density of the X-ray structure is 7.20 g/cm³. Lattice parameters of the LaMnO_3 compound are $a = 5.511$ Å, $b = 5.511$ Å, and $c = 13.396$ Å, with a unit cell volume of 352.27 Å³. Density of the X-ray structure of this compound is 6.84 g/cm³.

3.2. FTIR analysis

FTIR spectroscopic analysis was performed using a Bruker ALPHA spectrometer (Ettlingen, Germany) at room temperature with a spectral resolution of 1 cm⁻¹. Measurements were taken in the frequency range of 4000 – 400 cm⁻¹ using KBr pellets.

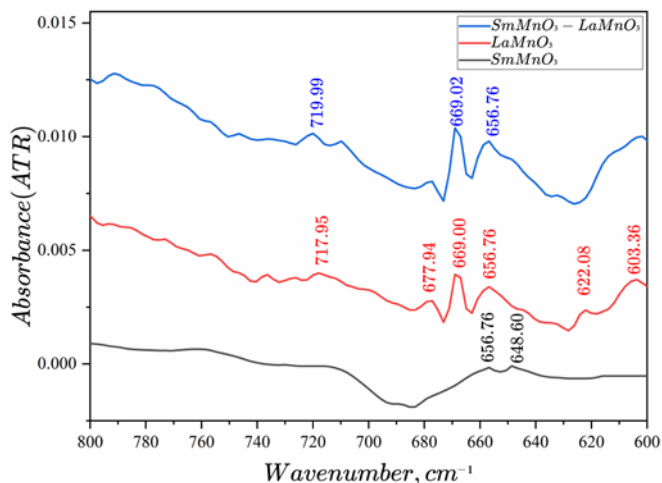


Figure 4 – IR spectrum of LaMnO_3 , SmMnO_3 , and LaMnO_3 - SmMnO_3 .

Figure 4 shows the FTIR spectrum of LaMnO_3 , SmMnO_3 , and LaMnO_3 - SmMnO_3 samples. The correspondence between Mn-O bonds and vibrations in 800 – 600 cm⁻¹ absorption region explains the formation of the perovskite

structure. LaMnO_3 compound is identified by absorption bands at 717.95, 677.94, 669.00, 656.76, 622.08, and 603.36 cm^{-1} . SmMnO_3 compound was observed near 656.76 and 648.60 cm^{-1} . In $\text{LaMnO}_3\text{-SmMnO}_3$ composite, an increase in the intensity of the bands in the regions of 719.99, 669.02, and 656.76 cm^{-1} can be observed.

3.2. SEM-EDS analysis

SEM-EDS analysis was performed using a Thermo Scientific ChemSEM Axia (USA) in a 99.999% pure nitrogen (N_2) atmosphere. The quantitative and qualitative elemental composition was determined by SEM and EDS analysis.

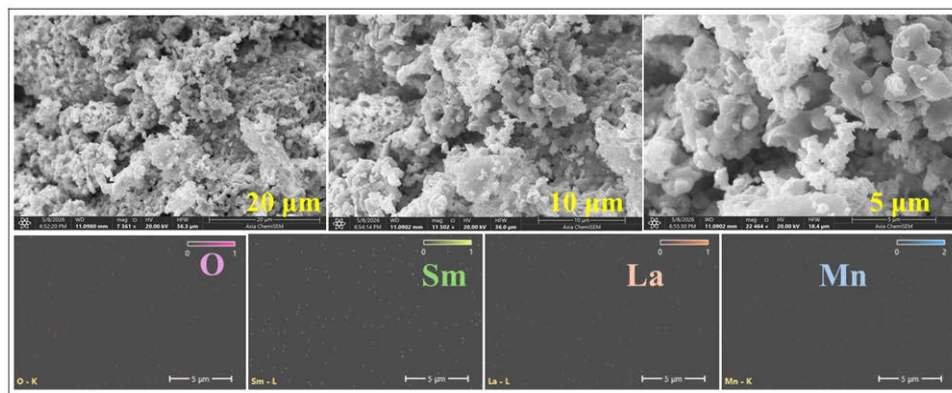


Figure 5 – SEM images of the surface of $\text{LaMnO}_3\text{-SmMnO}_3$ at 20, 10, and 5 μm , and elemental distribution.

Figure shows SEM images and EDS elemental distribution maps of the $\text{LaMnO}_3\text{-SmMnO}_3$ perovskite composite at sizes of 20, 10, and 5 μm . The SEM images show that the product is agglomerated and has a fine-grained morphology. The EDS map indicates that the constituent elements are evenly distributed and confirms that the composite has a homogeneous structure.

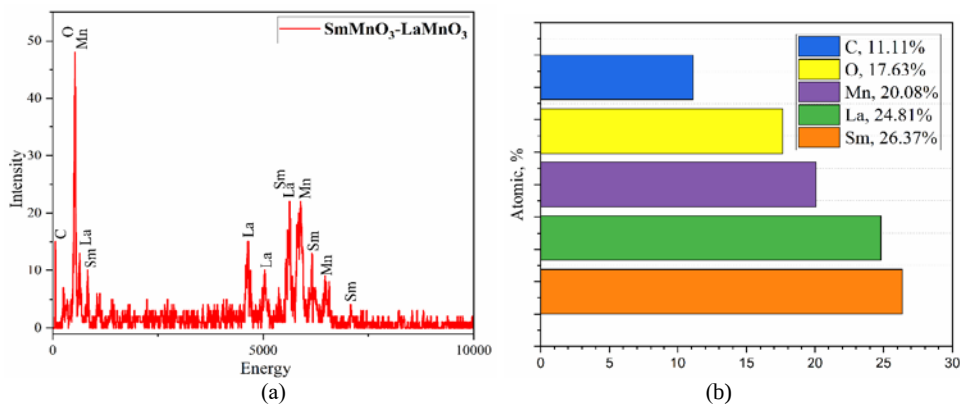


Figure 6 – SEM-EDS diagram (a) and atomic percentage, % (b) of $\text{LaMnO}_3\text{-SmMnO}_3$ composite.

EDS diagram shows the composition of the composite, confirming the presence of La, Mn, O, and Sm; the atomic composition of these elements serves as evidence of the composite's formation. The presence of carbon in the diagram is also explained by the fact that it was detected by placing the sample on a carbon label in scanning electron microscope.

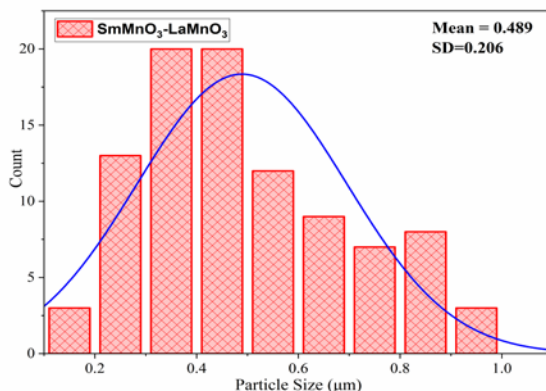


Figure 7 – Average particle size of LaMnO₃-SmMnO₃ at 5 μm.

Figure 7 shows that the average particle size of the two-phase manganite in the SEM range of 5 μm was 0.489 μm, with a standard deviation of 0.206 μm. The primary particle sizes ranged from 0.1 to 1.1 μm. The measurements were taken using ImageJ software.

4. Conclusion

LaMnO₃-SmMnO₃ composite was synthesized using the Pecchini method. Crystallographic and phase characteristics of the product were determined by X-ray analysis. Based on the results, the a, b, and c parameters, the space group, and the crystal system types were determined. The orthorhombic *pbnm* samarium manganite (62) crystallizes in the space group, while the lanthanum manganite exhibits a rhombohedral *R-3C* (167) structure. The FTIR results corresponded to Mn-O bond vibrations in the absorption range of 800–600 cm⁻¹. Surface area and elemental distribution were analyzed using SEM-EDS data. The average microparticle size in the 5 μm range was 0.489 μm. The results presented confirm the formation of a two-phase perovskite manganite composite.

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SmMnO₃-LaMnO₃ ПЕРОВСКИТТИ МАНГАНИТТИҢ СИНТЕЗІ, ҚҰРЫЛЫСЫ ЖӘНЕ МОРФОЛОГИЯСЫ**Нурбекова М.А., Матаев М.М., Сарсенбаева* З.Б., Абдраймова М.Р., Көшкімбаева А.Қ.***«Қазақ ұлттық қыздар педагогикалық университеті» КеАҚ, Алматы, Қазақстан*

Түйіндемe: Бұл мақалада LaMnO₃-SmMnO₃ қос манганитті перовскитті композиттің синтезі және фазалық құрамы мен құрылыс-морфологиясы қарастырылады. Құрамында самарий және лантан сирек элементтері бар қос фазалы манганитті синтездеудегі мақсат алынған өнімдерді классикалық перовскитті манганиттерден және бір фазалы самарийдің және лантанның манганиттерімен құрылыс мен қасиеттерін ерекшеліктерді зерттеу. Өнімді синтездеу үшін золь-гельді синтезге негізделген Печини әдісі қолданылды. Бұл әдісті таңдаудағы мақсат ұсақтығы жоғары және кристаллографиялық таза өнім алуға бағытталды. Фазалық құрамы мен кристаллографиялық параметрлерін рентген фазалы талдау (XRD), Me–O арасындағы байланыстың түзілгенін анықтау үшін Фурье-ИК (FTIR) және морфологиясын; беттік өлшемі мен элементтік таралуын сканерлеуші электронды микроскоп (SEM-EDS) әдістері арқылы талданды. Синтез үшін металл нитраттарының гидраттары қатысында золь-гель түзуге негізделе жүргізілді. Өнімнің түзілгенін фазалық талдау жүргізіп құрамындағы SmMnO₃ PDF 00-025-0747 сәйкес орторомбалы Pbnm (62) пішінді, ал LaMnO₃ PDF №01-090-6505 картасына сәйкес ромбоэдрлік R-3c (167) кеңістіктік топқа сәйкес екендігі анықталды. FTIR мәндері бойынша интенсивті жұтылу жолақтары 750-600 см⁻¹ аралығында жатқаны анықталып, Mn – O байланыстың түзілгенін көрсетеді. SEM-EDS нәтижелері бойынша ұнтақ өнімнің орташа беттік өлшемі 0,489 μm екені және элементтердің таралу картасы арқылы өнімнің сәтті түзілгенін көрсетеді.

Түйінді сөздер: перовскит, манганит, Печини әдісі, XRD, SEM-EDS талдау

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СИНТЕЗ, СТРУКТУРА И МОРФОЛОГИЯ ПЕРОВСКИТНОГО КОМПОЗИТА SmMnO₃-LaMnO₃**Нурбекова М.А., Матаев М.М., Сарсенбаева* З.Б., Абдраймова М.Р., Көшкімбаева А.Қ.***НАО «Казахский национальный женский педагогический университет», Алматы, Казахстан*

Резюме: в этой статье рассматривается синтез и фазовый состав, и строительно-морфология двойного манганитового перовскитного композита LaMnO₃-SmMnO₃. Целью синтеза двухфазного манганита, содержащего редкоземельные элементы самарий и лантан, является изучение особенностей строения и свойств полученных продуктов из классических перовскитных манганитов и манганитов однофазного Самария и лантана. Для синтеза продукта использовался метод Печини, основанный на синтезе Золя-геля. Цель выбора этого метода была направлена на получение продукта с высокой тонкостью и кристаллографической чистотой. Фазовый состав и кристаллографические параметры были проанализированы с помощью методов рентгенофазного анализа (XRD), Фурье-ИК (FTIR) и морфологии для определения образования связи между Me – O; электронного микроскопа (SEM-EDS), сканирующего размер поверхности и распределение элементов. Для проведения синтеза проводились на основе образования золь-геля в присутствии

гидратов нитратов металлов. Фазовый анализ образования продукта показал, что SmMnO_3 имеет орторомбальную форму Pbnm (62) в соответствии с PDF 00-025-0747, а LaMnO_3 соответствует ромбоэдрической пространственной группе $R\text{-}3\text{C}$ (167) в соответствии с картой PDF №01-090-6505. Установлено, что интенсивные полосы поглощения по значениям FTIR лежат в пределах $750\text{--}600\text{ см}^{-1}$, что указывает на образование связи $\text{Mn} - \text{O}$. Результаты SEM-EDS показывают, что средний размер поверхности порошкообразного продукта составляет $0,489\text{ мкм}$, и продукт был успешно сформирован с помощью карты распределения элементов.

Ключевые слова: перовскит, манганит, метод Печини, XRD, SEM-EDS анализ

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