

SYNTHESIS AND PHYSICO – CHEMICAL CHARACTERISTICS OF COMPLEX FERRITE $\text{CrNaFe}_2\text{O}_5$

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Abstract: *Introduction.* The article discusses for the first time the synthesis, radiographic and electron microscopic examination of $\text{CrNaFe}_2\text{O}_5$, synthesized using the high – temperature Sol-gel method. The phase of complex ferrites was synthesized using Sol-Gel synthesis method at high temperature. Initially, the structure of $\text{CrNaFe}_2\text{O}_5$ composite ferrite was researched by X-ray phase analysis and SEM, the syngony type, elementary cell parameters, radiographic and pycnometric densities and elemental analysis were defined: $\text{CrNaFe}_2\text{O}_5$ -a=5.0289, b= 5.0289, c=13.6938 Å , $\rho_{\text{x-ray}}= 5.327 \text{ g/sm}^3$, $\rho_{\text{pycn.}}=5.331 \text{ g/sm}^3$. A comparative analysis of the connection between the parameters of the Crystal cell of the initial materials and of the obtained complex ferrites was carried out. Through a scanning electron microscope, microsystems were taken from different parts of $\text{CrNaFe}_2\text{O}_5$ type crystallite, the elemental composition of crystals was analyzed, and the general type of surface layer of complex ferrite was displayed. As a result, the fact that the compound consists of a single phase, the clarity of its construction was determined by the topography and chemical composition of the compound. Consequently, it was discovered that the newly synthesized complex ferrites correspond to the formula $\text{CrNaFe}_2\text{O}_5$. The particles of the formed compounds have a large size (between 200 μm , 20.0 μm and 5 μm). The results of the element analysis show that the compound is compatible.

Keywords: Sol-gel method; ferrites; syngony; radiography; pycnometric density; elementary cell parameters.

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Introduction

Magnetic materials, particularly nano-sized ferrites, show a significant change in physical, electrical, and magnetic properties in contrast to their bulk counterparts due to their high surface-to-volume ratio of the grains. Ferrite nanoparticles have scientific and technological importance in recent years due to their magnetic properties and wide range of applications especially when the size of the particles approaches to nanometer scale [1]. Among all magnetic materials, ferrites are the most useful because in addition to magnetic properties, they are also good electrical insulators [2]. They have been used for high-frequency transformer cores, rod antennas, and radio-frequency coils [3-4]. Multiferroic materials that demonstrate a changed electric polarization by a magnetic field or a changed magnetization by an electric field have recently initiated enormous interest for their potential applications in the next-generation novel multifunction devices such as spintronics, data storage, sensors and so on [5-6].

Polycrystalline ferrites are very good dielectric materials which have numerous applications at microwave frequencies. The study of dielectric properties gives valuable information about the behavior of localized electric charge carriers and can explain the phenomenon of dielectric polarization and electrical conduction in the material. The experimental conditions used in the preparation of these materials show a strong impact on the properties of the resultant ferrite nanoparticles. For this reason, several methods have been used in the preparation of nanoparticles, like the co-precipitation method [7-8], sol-gel technique [9-11], hydrothermal method [12-13], microwave sintering method [14], spray-spin-heating-coating method [15] and auto combustion method [16]. Out of all these, sol-gel method is promising technique for the synthesis of nano ferrites in bulk scale due to the production of homogeneous particles. The sol-gel method is the most convenient technique to synthesize nanoparticles because of its simplicity, inexpensive precursors, short preparation time, better control over crystallite size and other properties of the materials [17]. The current effort has been focused on studying the composition and frequency-dependent dielectric properties of Cr^{+3} ferrites synthesized through sol-gel technique.

1. Experimental

The Sol-Gel method was used as an efficient way to synthesize a new composite ferrite with a complex mixed composition. In order to determine the composition of the new complex mixed ferrite obtained by the Sol-Gel method, an X-ray phase study was carried out, and a scanning electron microscope study was executed in order to run a quantitative and qualitative analysis.

X-ray analysis was carried out at the Kazakh National Women's Pedagogical University on the diffractometer Miniflex/600 (Rigaku). Analysis using a sika beam ($U=30$ KV, $J=10$ MA, rotation speed 1000 pulses per second, time constant $t=5$ sec., 2θ with an angle interval between 5 and 90) was carried out on the Miniflex 600 RIGAKU, filtered by a filter.

A study was carried out using a scanning electron microscope (SEM) (APPLICATION Note team, Brooker, Germany) to study the spectrum of distribution of the element, quantitative and qualitative analysis, and the percentage content of elements.

As a primary raw material, distilled water of the Chromium (III) oxide ("H. T.") brand, Iron (III) oxide ("H. T.") brand and sodium carbonate ("H. T.") brand were used. The use of citric acid and glycerin as clarifiers has a positive effect on the formation of a homogeneous phase of samples. Stoichiometric amounts of oxides were crushed and mixed in an agate kilt until a homogeneous mixture was obtained. Purified water, glycerin and citric acid were added to the resulting mixture. To obtain the gel, the mass was heated in an electric oven. After that, the formed gel was subjected to repeated annealing in a muffle furnace with an increase of 100°C per hour in the range of 600-1100°C temperature. The firing was divided into six stages. The first stage is 600°C, the second stage is 700°C, the third stage is 800°C, the fourth stage is 900°C, the fifth stage is 1000°C, the sixth stage is 1100°C, the total duration is 39 hours. After each stage of synthesis, intermediate grinding was carried out [18].

X – rays of synthesized polycrystalline powders are indicative by the method of Homology (homologue is a distorted structural type of perovskite). The pycnometric density of ferrites was determined by the method [19].

2. Results and Discussion

In the course of the chromite – ferrite X-ray method, it was observed from the X-ray taken after the first stage that the process of decomposition of carbonate in the primary components was not completed and the sample was in an amorphous state, the crystallization process was not started, and the sol-gel reaction did not occur. After the first X-ray taken, it showed the need for the completion of the carbonate decomposition process in the initial components and the passage of the sol-gel reaction and the extension of the synthesis time in order for the sample to go from the amorphous state, to the crystallization process. In the course of X-ray observations of mixed complex chromite-ferrites synthesized at the second and subsequent stages, in the range of 700-1100°C, it was noted that the samples had a decrease in the amorphous state, the crystallization process was in full swing, the kinetics of the sol-gel reaction was low. In addition, from the diffractogram shown below, it was proved that the samples changed from an amorphous state to a completely polycrystalline state, completely forming an independent phase.

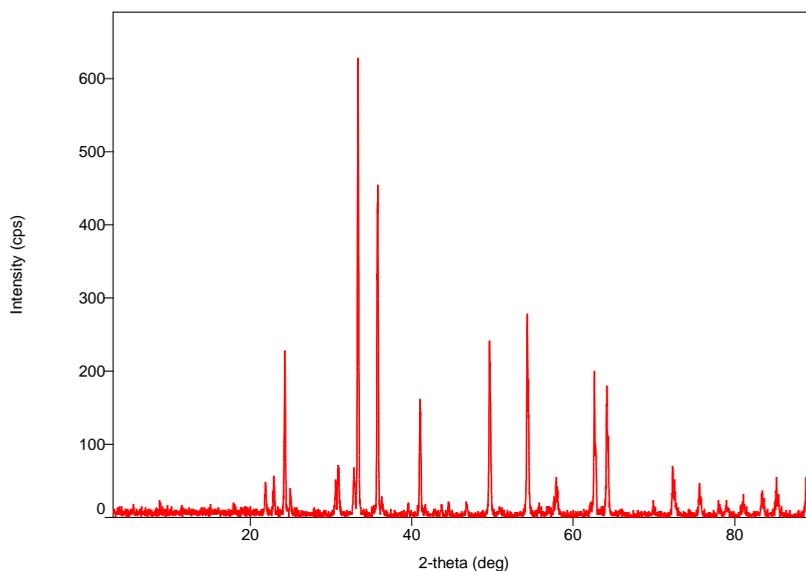


Figure 1 - X-ray diffractogram of the complex ferrite $\text{CrLiFe}_2\text{O}_5$. Insert: phase ratio diagram.

Below are the results of indexing the diffractogram of the complex ferrite studied by X-ray analysis using the Rietveld method.

Toluene and distilled water were used as neutral liquids. The density of the composite materials was measured 5 times and the average values were calculated.

Table 1 - Values of unit cell parameters for the sample under study.

Sample	Phase name	a(A)	b(A)	c(A)	α	β	γ	$V(\text{A}^3)$
1	$\text{CrNaFe}_2\text{O}_5$	5.0289	5.0289	13.6938	90	90	120	299.91

Table 2 - Lattice constants.

Sample	Phase name	Space group	Z	Calc. Density (g/cm^3)	Рyc. Density (g/cm^3)*
1	$\text{CrNaFe}_2\text{O}_5$	167 : R-3c,hexagonal	6	5.327	5.331

By the method of X-ray phase analysis, the temperature regime of the synthesis of complex mixed ferrite in $\text{CrNaFe}_2\text{O}_5$ was determined. With the radiographic method, the syngony type and unit cell parameters are distinguished. It was found that complex mixed ferrite is lysed in Crystal cubic syngony (Table 1 is given), and the correctness of the results of X-ray studies of Ferrite was

confirmed by the correspondence of the values of X-ray and pycnometric densities. The results of the ferrite X-ray indication are presented in Table 2.

A scanning electron microscope (SEM) is designed to obtain a magnified image of an object by scanning it with an electron beam directed at it and recording the signal generated by the interaction of electrons with a detector. The small diameter of the probe, even at low accelerating voltages and high currents, allows for elemental analysis of samples with dimensions of the analyzed area of several tens of nanometers. The beam current detector is located on the microscope column below the aperture of the objective lens, so that the beam current can be monitored at any time during the analysis.

In order to study the morphology of the surface layer of the new complex mixed ferrite samples synthesized by the sol-gel method, a study was carried out using an electron microscope scanning the microstructures of the electric diffraction image. Electron monographs of the compound taken in an imaging electron microscope are given in Figures 2 a), b), c).

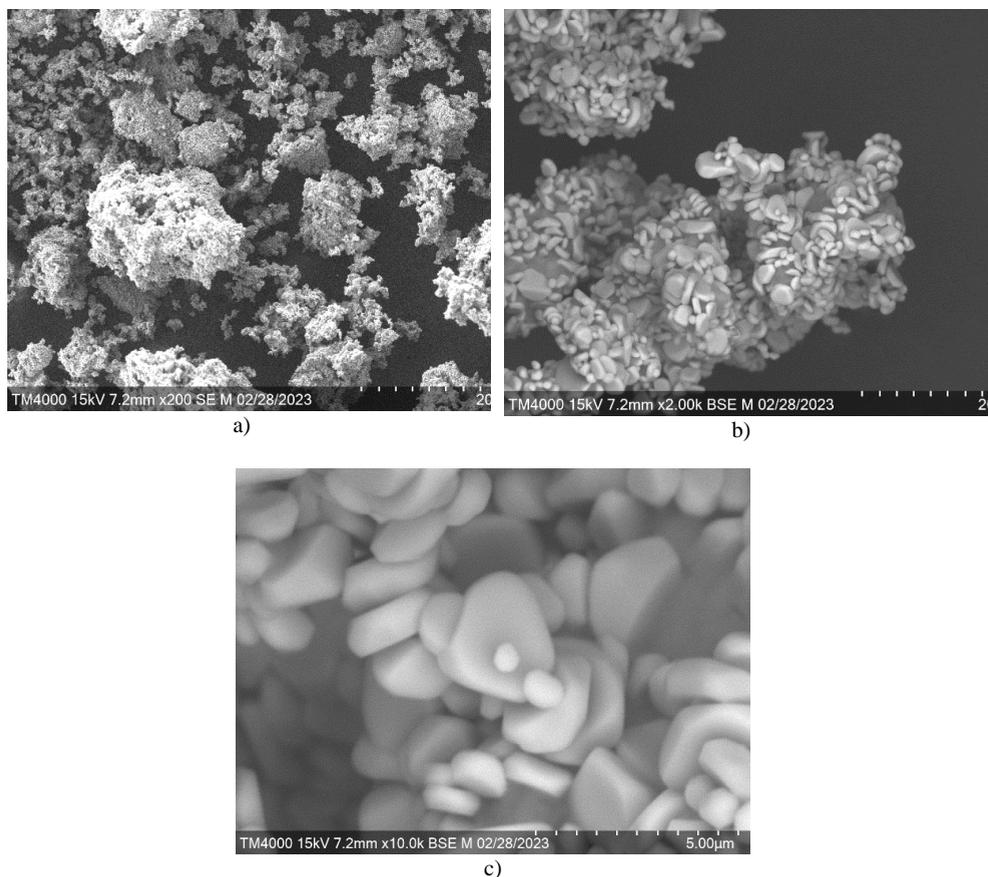


Figure 2 - Three different micrometer measurements of the new mixed complex ferrite $\text{CrNaFe}_2\text{O}_5$.

The above images show the results of micrographs taken at magnifications of 200µm, 20.0µm and 5 µm, and also show the general appearance of the complex ferrite surface layer. As a result, the compound consists of one phase, the clarity of its structure was determined by the topography and chemical composition of the compound.

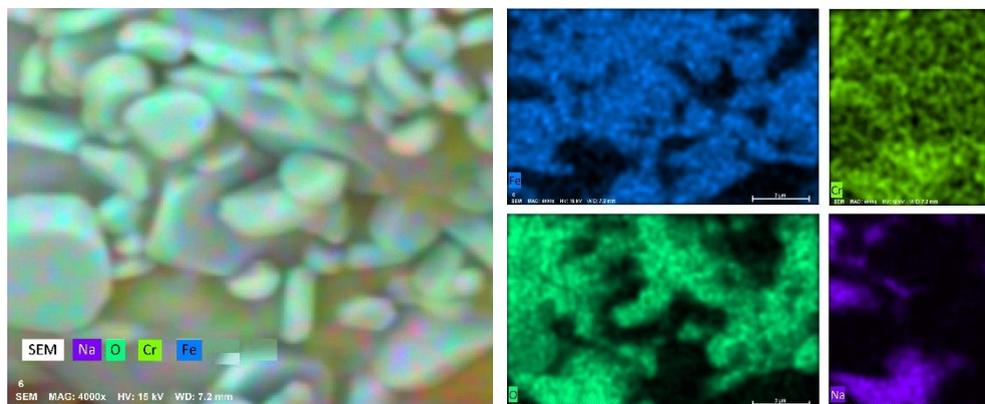


Figure 3 - MAP of the distribution of elements in the composition of the new mixed complex ferrite CrNaFe₂O₅ (order of the elements Cr, Fe, Na, O and color on the MAP).

Based on the distribution map of elements, on the basis of solving the nature of crystallization, the chemical composition with microstructure and distribution zones of chromium, iron, lithium, and oxygen atoms were studied. As a result of the numerical elemental composition study, it can be concluded that iron, chromium, sodium metals, oxygen, carbon atoms are distributed in the 3 µm regions (Figure 3). In an imaging electron microscope, it is possible to obtain nanoscale measurements of solids in powder form.

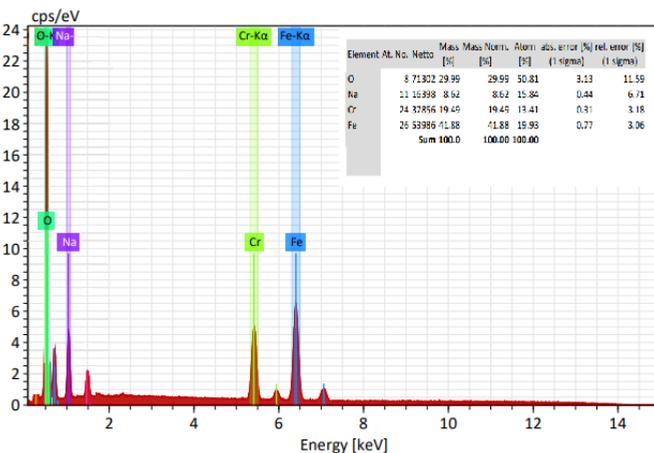


Figure 4 - Spectrum samples of the CrNaFe₂O₅ compound. The results of the element analysis are built-in.

To study the spectrum of distribution of the element, quantitative and qualitative analysis, and the percentage content of elements, a study was carried out using a scanning electron microscope. The spectrum samples of the synthesized new mixed complex Ferrite and the results of elemental analysis are shown in Figure 4.

3. Conclusions

Summarizing the results of the research, a new mixed complex ferrite containing $\text{CrNaFe}_2\text{O}_5$ was synthesized for the first time by Sol-Gel method. In order to determine the composition of the obtained new complex mixed ferrite, an X-ray phase study was carried out, and a scanning electron microscope study was carried out in order to conduct a quantitative and qualitative analysis.

For the first time, syngony types and parameters of the elementary cells of complex mixed ferrite synthesized by X-ray phase analysis were determined. $\text{CrNaFe}_2\text{O}_5$ (cubic, $a=5.0289$, $b=5.0289$, $c=13.6938 \text{ \AA}$, $Z=2$, $\rho_{\text{X-ray}}=5.327 \text{ g/cm}^3$, $\rho_{\text{печн.}}=5.331 \text{ g/cm}^3$); The results of X-ray examination showed that the synthesized compound is polycrystalline. The accuracy of the crystallochemical data is confirmed by the satisfactory accordance of X-ray and pycnometric densities.

Using a scanning electron microscope, microsamples were taken from different parts of $\text{CrNaFe}_2\text{O}_5$ type crystallites, the elemental composition of crystals was analyzed, and the general type of surface layer of complex ferrite was shown. As a result, the compound consists of one phase, the clarity of its structure was determined by the topography and chemical composition of the compound. As a result, it was determined that newly synthesized complex ferrites correspond to $\text{CrNaFe}_2\text{O}_5$ formula. The particles of the formed compounds have a large size (between $200 \mu\text{m}$, $20.0 \mu\text{m}$ and $5 \mu\text{m}$).

$\text{CrNaFe}_2\text{O}_5$ КҮРДЕЛІ ФЕРРИТІН СИНТЕЗДЕУ ЖӘНЕ ФИЗИКА – ХИМИЯЛЫҚ СИПАТТАМАЛАРЫ

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Түйіндемe. Мақалада алғаш рет жоғары температуралы Золь – Гель әдісі арқылы синтезделініп алынған $\text{CrNaFe}_2\text{O}_5$ синтездеу, рентгенографиялық және электронды микроскопиялық зерттеу қарастырылған. Алғаш рет $\text{CrNaFe}_2\text{O}_5$ құрамды ферритінің құрылысын рентгендік фазалық талдау және сканерлеуші электронды микроскоп әдістерімен зерттелді, сингония типі, элементар ұяшық параметрлері, рентгенографиялық және пикнометрлік тығыздықтары, элементтік талдаулары анықталды: $\text{CrNaFe}_2\text{O}_5$ - $a=5.0289$, $b=5.0289$, $c=13.6938 \text{ \AA}$, $\rho_{\text{рент.}}=5.327 \text{ г/см}^3$, $\rho_{\text{пикн.}}=5.331 \text{ г/см}^3$. Бастапқы заттардың кристалдық ұяшық параметрлері мен алынған күрделі ферриттердің кристалдық ұяшық параметрлері арасындағы байланысына салыстырмалы талдау жүргізілді. Сканерлеуші электронды микроскоп арқылы $\text{CrNaFe}_2\text{O}_5$ типті кристаллиттің әр түрлі бөліктерінен микросынамалар алынып, кристалдардың элементтік құрамына талдау жасалынды, күрделі ферриттің беттік қабатының жалпы түрі көрсетілді. Нәтижесінде косылыстың бір фазадан тұратындығы, құрылысының айқындылығы топография мен косылыстың химиялық құрамымен

анықталды. Нәтижесінде жаңа синтезделініп алынған күрделі ферриттер $\text{CrNaFe}_2\text{O}_5$ формуласына сәйкес келетіні анықталды. Түзілген қосылыстардың бөлшектері үлкен өлшемге ие (200 мкм, 20.0 мкм және 5 мкм, аралығында). Элементтік талдау нәтижелері қосылыстың сәйкес келетіндігін көрсетеді

Түйін сөздер: Золь – Гель әдісі; ферриттер; сингония; рентгенография; пикнометрлік тығыздық; элементарлы ұяшықтың параметрлері.

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СИНТЕЗ И ФИЗИКО – ХИМИЧЕСКИЕ ХАРАКТЕРИСТИКИ СЛОЖНОГО ФЕРРИТА $\text{CrNaFe}_2\text{O}_5$

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Резюме. В статье рассматривается синтез сложного феррита $\text{CrNaFe}_2\text{O}_5$, рентгенографическое и электронно-микроскопическое исследование. Методом высокотемпературного золь – гелевого синтеза была синтезирована фаза сложных ферритов. Впервые строение феррита, содержащего $\text{CrNaFe}_2\text{O}_5$, исследовали методами рентгенофазного анализа и сканирующего электронного микроскопа, выявили сингонический тип, параметры элементарных ячеек, рентгенографические и пикнометрические плотности, элементный анализ: $\text{CrNaFe}_2\text{O}_5$ - $a=5.0289$, $b=5.0289$, $c=13.6938$ Å, $\rho_{\text{рент.}}=5.327$ г/см³, $\rho_{\text{плкн.}}=5.331$ г/см³, проведен сравнительный анализ взаимосвязи исходных веществ между параметрами кристаллической ячейки и параметрами кристаллической ячейки полученных сложных ферритов. С помощью сканирующего электронного микроскопа из различных частей кристаллита типа $\text{CrNaFe}_2\text{O}_5$ были получены микрососуды, проведен анализ элементного состава кристаллов, показан общий вид поверхностного слоя сложного феррита. В результате определялась однофазность соединения, четкость строения, топография и химический состав соединения. В результате было обнаружено, что новые синтезированные сложные ферриты соответствуют Формуле $\text{CrNaFe}_2\text{O}_5$. Частицы образующихся соединений имеют большие размеры (от 200 мкм, 20.0 мкм до 5 мкм). Результаты элементного анализа представлены в виде таблицы.

Ключевые слова: ферриты; сингония; рентгенография; пикнометрическая плотность; параметры элементарной ячейки

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