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SYNTHESIS AND CHARACTERIZATION OF $NiFe_2O_4$ SPINEL NANOPARTICLES

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Abstract. *Introduction.* Nickel ferrite ($NiFe_2O_4$) spinel nanoparticles have attracted great interest in recent years due to their unique physicochemical properties and wide range of applications in various fields. *Methodology.* In this work, the synthesis of $NiFe_2O_4$ nanoparticles by the sol-gel method was studied. The synthesized $NiFe_2O_4$ nanoparticles were characterized using methods such as X-ray diffraction analysis and energy-dispersive X-ray microanalysis. *Results and discussion.* The results of X-ray diffraction analysis showed that the main phase in the analyzed samples is iron-containing oxide of cubic structure $NiFe_2O_4$, and the position and relative intensity of the peaks correspond to the standard JCPDS No. 54–0964. Also, EDS analysis of the studied samples showed that the nanopowders consist of the elements Ni, Fe and O, which indicates the purity of the synthesized sample and the absence of any impurities. FTIR spectrum of $NiFe_2O_4$ nanoparticles showed two main peaks at 458 cm^{-1} and 548 cm^{-1} corresponding to the metal-oxygen band. *Conclusion.* The results of the study of the magnetic properties of the obtained $NiFe_2O_4$ nanoparticles show that the magnetic properties change depending on the alloy components and phase composition, i.e. FeNiO (77% Fe_3O_4 - magnetite 23% $Fe_{0.64}Ni_{0.36}$) is a particle of about 25 nm size, a multicomponent ferromagnetic powder with a predominant magnetite phase, low coercivity and relatively large saturation magnetization values. This means that they are a soft magnetic material with high magnetic properties.

Keywords: nickel ferrite, spinel, nanoparticles, sol-gel synthesis, magnetic properties, X-ray diffraction, EDS, FTIR, Hysteresis loops, Ferrimagnetic material

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1. Introduction

One of the most problems troubling the scientific community in recent years is environmental pollution. Organic pollutants, especially dyes from the textile, leather and petroleum industries, discharged into surface and groundwater, annually cause serious damage to the environment [1]. The high structural stability of these pollutants and their nonbiodegradability make the removal of these molecules difficult. Therefore, treatment methods such as adsorption, advanced oxidation processes, coagulation, biological and membrane treatment have recently been widely used for wastewater treatment [2]. Additionally, the most widely used method is catalytic reduction as it is predominantly efficient, fast, environmentally friendly and cheap, unlike other methods which are usually expensive, slow and difficult to use. Thus, it is important to find and develop a good, inexpensive, efficient, easily recoverable and recyclable catalyst. Among the many existing catalysts, magnetic materials are of particular interest because they have a large specific surface area, low cost, and ease of preparation and reuse [3]. Nickel ferrite ($NiFe_2O_4$) spinel nanoparticles have attracted great interest in recent years due to their unique physicochemical properties and wide range of applications in various fields [4]. Being nanomaterials, they exhibit unique behavior and functionality compared to their bulk counterparts, making them very attractive for advanced technological applications. $NiFe_2O_4$ is a ferrimagnetic material with high electrochemical stability, low conductivity, low saturation magnetization (Ms), low coercivity (Hc). Ni^{2+} as well as Fe^{3+} are capable of exchanging electrons in the octahedral site, this demonstrates excellent electrical and magnetic properties and distinguishes them as unique materials. Also, nickel ferrite nanoparticles, depending on their size, can exhibit both superparamagnetic and ferrimagnetic properties. $NiFe_2O_4$ nanoparticles smaller than 15 nm can act as superparamagnetic, and those larger than 15 nm can act as a ferrimagnetic material [5].

Understanding the synthesis, characterization and applications of $NiFe_2O_4$ spinels is essential to fully exploit their potential in various fields such as electronics, medicine, energy and environmental engineering. In recent years, significant research has been carried out to study the synthesis methods, structural characterization and functional properties of $NiFe_2O_4$ spinel nanoparticles [6]. Various synthesis methods, including sol-gel, co-precipitation, hydrothermal and combustion methods, have been explored to tailor the size, morphology and magnetic properties of nanoparticles. In this work, the structural properties and elemental analysis of nanoparticles synthesized by the sol-gel method were investigated.

In this work, the authors studied the synthesis of $NiFe_2O_4$ nanoparticles by the sol-gel method, and also carried out X-ray structural and energy-dispersive X-ray analyses, and investigated the magnetic properties of the obtained nanomaterials.

2. Conditions and methods of research

Materials: iron (III) nitrate nonahydrate, purity ≥99.95%, nickel (II) acetylacetonate, purity ≥95% were obtained from Sigma-Aldrich. All chemicals were of analytical grade and used without pretreatment.

Methods: X-ray diffraction analysis was carried out on an automated diffractometer DRON-3 with CuKa radiation, β-filter. Conditions for taking diffraction patterns: U=35 kV; I=20 mA; shooting θ-2θ; detector 2 deg/min. EDS analysis was carried out on a scanning electron microscope Zeiss Crossbeam 540 – FE – SEM. The Crossbeam 540 is a dual-beam FIB-SEM microscope for nanotomography and nanofabrication that enables EDS studies. The FTIR analysis of nanopowders was carried out on Nicolet iS10 FT-IR Spectrometer

Preparation of $NiFe_2O_4$ nanoparticles

The molar ratio of metal salts (8:4), iron (III) nitrate nonahydrate (3.308 g), nickel (II) acetylacetonate (0.996 g) was used as a precursor of metal powders. The original precursor salts were dissolved in ethanol in a three-neck flask equipped with a heated magnetic stirrer and a reflux condenser. 1 mL of acetic acid was used as a catalyst to increase the rate of hydrolysis. After 20 minutes, 2-methoxyethanol is added as a solvent, and H_2O, ethylene glycol and ethanol are added to form a gel while heating and stirring. The synthesized gel solutions were dried at a temperature of 100°C for approximately 24 hours in an oven. A well-dried sample was calcined at a temperature (600°C) for 8 hours in a muffle furnace.

3. Discussion of research results

X-ray diffraction patterns of the studied $NiFe_2O_4$ sample are presented in Figure 1. X-ray diffraction analysis of the synthesized $NiFe_2O_4$ sample showed all peaks corresponding to metallic ferrites. Also, the identified phases are consistent with the experimental results. From the X-ray diffraction analysis data it is clear that the studied $NiFe_2O_4$ sample has nano-sized polycrystalline structures, this is evidenced by the broadening and low intensity of the diffraction peaks. The main phase in the analyzed samples is iron-containing oxide for $NiFe_2O_4$ cubic structure. It can be noted that the position and relative intensity of the peaks are consistent with the standard $NiFe_2O_4$ according to JCPDS No. 54–0964, which confirms the structure [7].

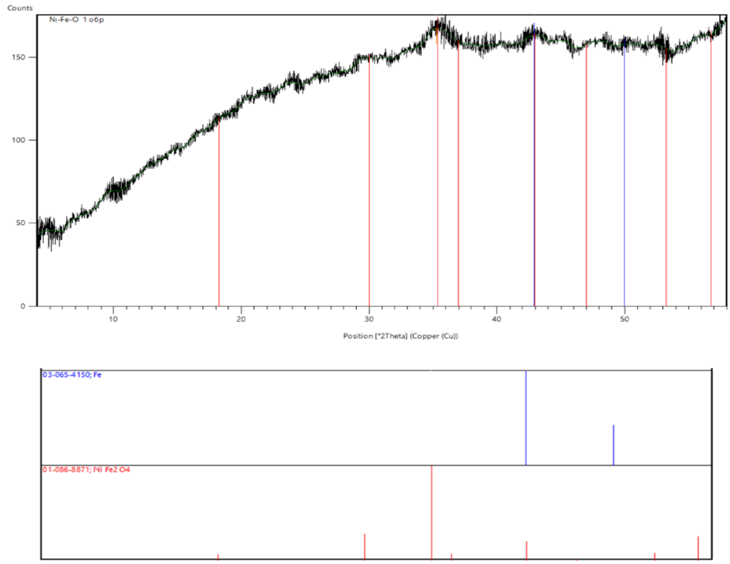

Figure 1 - X-ray diffraction analysis of $NiFe_2O_4$ nanoparticles

Figure 2 shows the results of EDS analysis of the synthesized $NiFe_2O_4$ nanopowders. According to the results of EDS analysis, the nanopowder samples consist of the elements Ni, Fe and O. In addition, signals corresponding to N and C were not detected in the EDS spectrum, which indicates the purity of the synthesized sample and does not contain any impurities [8].

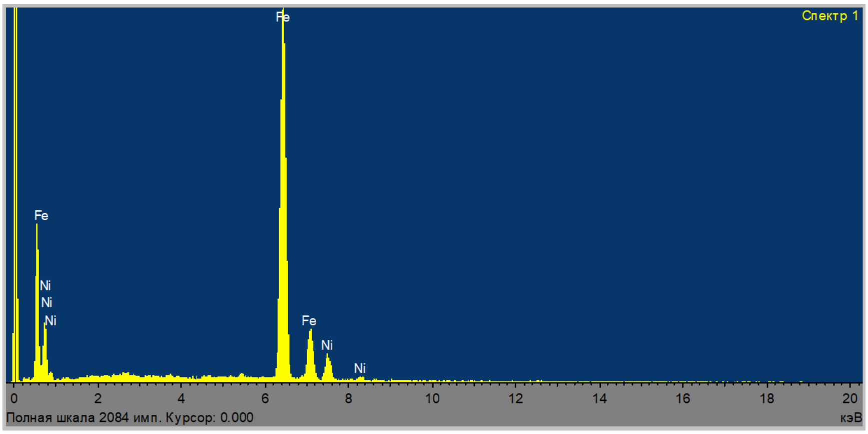

Figure 2 - EDS spectrum of $NiFe_2O_4$ nanoparticles

The formation of nickel ferrite nanoparticles was further identified by FTIR spectrum. Figure 3 shows the FTIR spectrum of $NiFe_2O_4$ nanoparticles is in the wavenumber range of 400–1250 cm^{-1}. Two main peaks at 458 cm^{-1} and 548 cm^{-1}

correspond to the metal-oxygen band. The band appearing at 458 cm^{-1} is attributed to the octahedral stretching vibrations of the metal (Fe^{3+}— O and Ni^{2+}— O), and the strong absorption band at 548 cm^{-1} corresponds to the stretching vibrations of the metal in the tetrahedral position of Fe ↔ O.

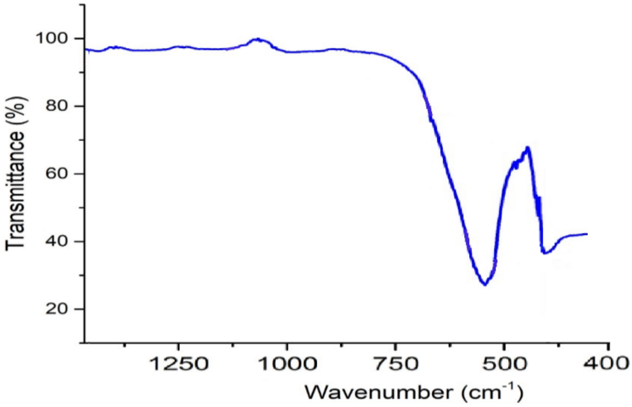

Figure 3 - FTIR spectrum of $NiFe_2O_4$ nanoparticles

Figure 4 shows the results of measuring the magnetic characteristics of the samples under study using vibration magnetometry. The measurements were carried out by the induction method, by measuring the induced electromotive force of induction in signal coils by a magnetized sample oscillating at a certain frequency, while recording the temperature and external magnetic field at the time of measurement. This method ensures an error in the magnetization measurement result of no worse than 1.5% when the measured magnetization value is not lower than ±1·10-6 A·m^2 (±1·10-3 A·m^2).

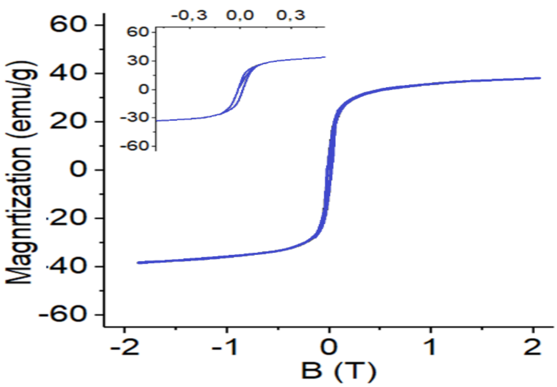

Figure 4 - Hysteresis loops of the $NiFe_2O_4$ nanoparticles

Based on the presented graph of field dependencies, the main magnetic characteristics were determined (Hc - coercivity, Mr - remanent magnetization, Ms - saturation magnetization, Mr / Ms - hysteresis loop squareness coefficient), which are given in Table 1.

Table 1 - Main magnetic characteristics

Compound	$H_{c.}$ Oe	$M_{r.}$ emu/g	$M_{s.}$ emu/g	M_r / M_r
FeNiO	15.60	7.20	38.30	0.19

When analyzing the course of the hysteresis loop, a change in the magnetic properties is obvious, depending on the components of the alloys and the phase composition of the samples under consideration. The FeNiO sample (77% Fe_3O_4 – Magnetite 23% $Fe_{0.64}Ni_{0.36}$) is a multicomponent ferromagnetic powder with a predominance of the magnetite phase with a particle size of about 25 nm and with low coercivity and relatively large saturation magnetization values, corresponding to the characteristics of magnetite particles with similar sizes [9]. The magnetic contribution of FeNi particles, which is a soft magnetic material, is significant [10].

4. Conclusion

$NiFe_2O_4$ nanoparticles were successfully synthesized by sol-gel method. The synthesized $NiFe_2O_4$ nanoparticles were characterized using techniques such as X-ray diffraction, energy-dispersive X-ray microanalysis. The results of X-ray diffraction analysis showed that the main phase in the analyzed samples is the iron-containing oxide of cubic structure $NiFe_2O_4$. Also, EDS analysis of the studied samples showed that nanopowders consist of the elements Ni, Fe and O, which indicates the purity of the synthesized sample and the absence of any impurities. FTIR spectrum of $NiFe_2O_4$ nanoparticles showed two main peaks at 458 cm−1 and 548 cm−1 corresponding to the metal-oxygen band.The results of the study of the magnetic properties of the obtained $NiFe_2O_4$ nanoparticles show that the magnetic properties change depending on the alloy components and phase composition, i.e. FeNiO (77% Fe_3O_4 - magnetite 23% $Fe_{0.64}Ni_{0.36}$) is a particle of about 25 nm size, a multicomponent ferromagnetic powder with a predominant magnetite phase, low coercivity and relatively large saturation magnetization values. This means that they are a soft magnetic material with high magnetic properties.

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Conflict of Interests: No conflict of interest.

$NiFe_2O_4$ ШПИНЕЛЬДІК НАНОБӨЛШЕКТЕРДІҢ СИНТЕЗІ ЖӘНЕ СИПАТТАМАСЫ

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Түйіндеме. *Кіріспе.* Никель ферриті ($NiFe_2O_4$) шпинельді нанобөлшектері соңғы жылдары бірегей физика-химиялық қасиеттеріне және әртүрлі салаларда қолданудың кең ауқымына байланысты үлкен қызығушылық тудыруда. Бұл жұмыста $NiFe_2O_4$ нанобөлшектерінің золь-гель әдісімен синтезі зерттелді. Синтезделген $NiFe_2O_4$ нанобөлшектері рентгендік дифракциялық және энергия-дисперсиялық рентгендік талдау әдістері бойынша сипатталды. *Нәтижелер және талқылау.* Рентгендік дифракциялық талдау нәтижелері талданатын үлгілердегі негізгі фаза $NiFe_2O_4$ текше құрылымды темірі бар оксид болып табылатынын, ал шыңдардың орналасуы мен салыстырмалы қарқындылығы JCPDS No 54–0964 стандартына сәйкес келетінін көрсетті. Сондай-ақ, зерттелген үлгілердің энергия-дисперсиялық рентгендік талдау нәтижелері наноұнтақтардың Ni, Fe және O элементтерінен тұратынын көрсетті, бұл синтезделген үлгінің тазалығын және ешқандай қоспалардың жоқтығын көрсетеді. $NiFe_2O_4$ нанобөлшектерінің ИҚ-Фурье спектрі металл-оттегі жолағына сәйкес екі негізгі 458 $см^{-1}$ және 548 $см^{-1}$ шыңдарын көрсетті. *Қорытынды.* Алынған $NiFe_2O_4$ нанобөлшектерінің магниттік қасиеттерін зерттеу нәтижелері қорытпа компоненттеріне және фазалық құрамына байланысты магниттік қасиеттердің өзгеруін, яғни FeNiO (77% Fe_3O_4 – Магнетит 23% $Fe_{0.64}Ni_{0.36}$) – бөлшектерінің өлшемі шамамен 25 нм болатынын, басым магнетит фазасы бар көпкомпонентті ферромагниттік ұнтақ және төмен коэрцивтілігі және салыстырмалы түрде үлкен қаныңу магниттелу мәндерін ие болатындығын көрсетеді. Бұл олардың магниттік қасиеттері жоғары жұмсақ магниттік материал екенін айқындайды.

Түйін сөздер: никель ферриті, шпинель, нанобөлшектер, золь-гель синтезі, магниттік қасиеттері, рентгендік дифракция, EDS, FTIR, гистерезис ілмектері, ферримагниттік материал

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СИНТЕЗ И ХАРАКТЕРИСТИКА НАНОЧАСТИЦ ШПИНЕЛИ $NiFe_2O_4$

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Резюме. *Введение.* Наночастицы шпинели феррита никеля ($NiFe_2O_4$) в последние годы привлекают большой интерес благодаря своим уникальным физико-химическим свойствам и широкому спектру применения в различных областях. В данной работе исследован синтез наночастиц $NiFe_2O_4$ золь-гель методом. Синтезированные наночастицы $NiFe_2O_4$ были охарактеризованы с использованием таких методов, как рентгеноструктурный и энергодисперсионный рентгеновский анализы. *Результаты и обсуждение.* Результаты рентгеноструктурного анализа показали, что основной фазой в анализируемых образцах является железосодержащий оксид кубической структуры $NiFe_2O_4$, а положение и относительная интенсивность пиков соответствуют стандарту JCPDS № 54–0964. Также нергодисперсионный рентгеновский анализ исследованных образцов показал, что нанопорошки состоят из элементов Ni, Fe и O, что свидетельствует о чистоте синтезированного образца и отсутствии каких-либо примесей. Спектр ИК-Фурье наночастиц $NiFe_2O_4$ показал два основных пика при 458 $см^{-1}$ и 548 $см^{-1}$, соответствующих полосе металлкислород. *Заключение.* Результаты исследования магнитных свойств полученных наночастиц $NiFe_2O_4$ показывают, что магнитные свойства изменяются в зависимости от компонентов сплава и фазового состава, то есть FeNiO (77% Fe_3O_4 - магнетит 23% $Fe_{0,64}Ni_{0,36}$) - частица Размер около 25 нм, многокомпонентный ферромагнитный порошок с преобладающей фазой магнетита, низкой коэрцитивной силой и относительно большими значениями намагниченности насыщения. Это означает, что они представляют собой магнитомягкий материал с высокими магнитными свойствами.

Ключевые слова: феррит никеля, шпинель, наночастицы, золь-гель синтез._магнитные свойства, дифракция рентгеновских лучей, EDS, FTIR, петли гистерезиса, ферримагнитный материал

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