

MICROEMULSION-BASED METHOD OF SYNTHESIS OF ZINC-NICKEL FERRITE AND THEIR MAGNETIC PROPERTIES

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Solid solutions of nickel-zinc ferrites $Zn_xNi_{1-x}Fe_2O_4$ were synthesized by two different methods: synthesis in microemulsions and by stepwise precipitation. The properties of the resulted nano-sized particles synthesized by two different methods were compared. It was found that the increase in zinc content leads to an increase in the lattice parameters. During the synthesis by the method of microemulsions, the temperature of a single-phase product formation is 400 °C, while by the method of precipitation, a single-phase product formation begins at 600 °C. It was shown that the materials synthesized by both methods have similar unit cell parameters. The average size of ferrite nanoparticles synthesized in microemulsions is smaller, lattice strain is higher compared to ferrites synthesized by stepwise precipitation. Also, lower treatment temperatures provide higher stoichiometry, and homogeneity of materials while magnetization difference is negligible. The particles of the obtained powders have high saturation magnetization $M_s = 45.6 \text{ Am}^2/\text{kg}$ for synthesis from microemulsions and $M_s = 44.8 \text{ Am}^2/\text{kg}$ for co-precipitated samples, the low coercive force $H_c = 1.3 \text{ kA/m}$ and $H_c = 3 \text{ kA/m}$, respectively. Changes made make synthesized particles more applicable in film deposition, and manufacturing of high-quality magnetic coatings.

Keywords: $NiFe_2O_4$, $ZnFe_2O_4$, magnetic properties, lattice constant, zinc substitution.

INTRODUCTION. Ferrites are a unique class of materials that combine high magnetic permeability with dielectric/insulator properties. Such combination allows their use in microwave electronics [1], optics [2], and power electronics as non-reciprocal components, active components of memory devices [3], and magnets. In addition, there are applications for ferrites in spintronics, self-controlled magne-

tic hyperthermia [4], and absorbent coatings. Moreover, the properties of different ferrites compositions can vary greatly, while most of these materials allow the properties to be adjusted using dopants [5].

Two types of ferrites are most often used for microwave applications in the case of small (from 0 to thousands Oe) magnetic fields: materials with the garnet and spinel structure.

Garnets are distinguished by low energy losses, but only in the monocrystalline form [6]. The properties of spinels differ.

Spinel based on Cu, Co, Mn, Mg, Ni, Zn and their solid solutions are used. For microwave applications, spinels based on Ni systems are the most interesting due to their highest electrical resistance and low energy loss compared to other materials. The crystalline structure of Ni ferrite is almost independent of the annealing temperature [7]. The addition of Co increases the coercive force of the material [8]. The addition of Mn and Cu increases the conductivity. Such changes are harmful in the microwave range due to increased energy loss. Ni-Zn solid solutions, on the other hand, make it possible to increase the saturation magnetization and reduce the coercive force. And energy losses at microwaves do not increase significantly.

Despite the long history of research, the synthesis of ferrites remains an urgent problem due to the constant development of communications and increasing requirements for materials. Ferrites are often used as ceramic samples for which the sintering temperature of ceramics exceeds 1200 °C [9]. Lowering the sintering temperature and increasing the homogeneity of ceramics can be achieved by using small ferrite particles with low agglomeration. Also, particles' size affects the possibility of their use in films, the coercive force of the product, and thus the energy absorption ability (or quality factor depending on the application).

Solid-state synthesis used in industry cannot ensure the chemical homogeneity of material particles and requires high temperatures. The essence of this method is the mechanical mixing of reagents, in the case of ferrites, these are metal oxides [10]. A product is formed that

is homogeneous at the macro level, but chemically heterogeneous. In addition, additional heat treatment at high temperatures is required. Due to this, additional grinding of the product is required and contamination with grinding bodies occurs.

Other methods used are thermal decomposition of precursors [11, 12], hydrothermal [13, 14] and sol-gel synthesis [15, 16], high-energy ball milling, and precipitation from solutions [17, 18], synthesis from microemulsions. The decomposition of precursors requires close decomposition temperatures of different precursors, which complicates the production of a single-phase product [19]. In the case of hydrothermal synthesis, the disadvantage is the use of very expensive equipment, which is not always available. The disadvantage of high-energy ball milling is the contamination of the product as a result of wear and tear, which occurs mainly from the balls and partially from the housing.

Precipitation from solutions is a widespread method of synthesis, which allows the obtaining of large quantities of the product. Synthesized materials are more chemically homogeneous compared to solid-phase synthesis, and lower heat treatment temperatures allow smaller particles to be obtained. Thus, this method is a candidate to replace the solid-state route in industry. But also the properties of the obtained material depend on a large number of factors (solubility of particles, the concentration of material and precipitant in the volume of the reactor, etc., which also depend on various environmental conditions). It is difficult to control the processes of nucleation and growth of crystals, which leads to the formation of an amorphous precipitate. The precipitate is poorly filtered and washed, and

after heat treatment, a strongly agglomerated product is formed, which requires mechanical grinding [20]. Nanoparticle growth and size distribution increase with increasing concentration. The choice of precipitator significantly affects the overall size, purity of the product, and the agglomeration process. Therefore, currently, when using synthesis by precipitation from aqueous solutions, it is difficult to control the processes of nucleation and growth of nanocrystallites, which often prevents the synthesis of nanoparticles with the necessary properties [21, 22].

On the other hand, the synthesis of nanoparticles using the microemulsion method is also of great interest. After the discovery of microemulsions, they become increasingly important for fundamental research and synthesis of materials of various natures [23, 24]. Due to its unique characteristics, namely ultra-low interfacial tension, high reaction surface area, thermodynamic stability, and ability to dissolve otherwise immiscible liquids. The use and applications of microemulsions are numerous in the chemical and biological fields. The microemulsion technique promises to be one of the versatile preparation methods that allow controlling particle properties such as particle size, geometry, morphology, homogeneity, and surface area.

This work aims to obtain small and weakly agglomerated zinc-nickel ferrite nanoparticles with the use of synthesis in microemulsions, to compare the temperature of nanoparticle formation during microemulsion synthesis and the classical synthesis of stepwise precipitation from aqueous solutions. Study the influence of the synthesis method on the magnetization properties.

EXPERIMENT AND DISCUSSION OF THE RESULTS. Nano-size spinel composites $\text{Zn}_x\text{Ni}_{1-x}\text{Fe}_2\text{O}_4$ with $x = 0-1$ were synthesized by using microemulsion of cetyltrimethylammonium bromide, cyclohexane, and n-pentanol. The obtained mixture of cetyltrimethylammonium bromide (1 g), cyclohexane (75 ml), and n-pentanol (2.5 ml) was stirred for 20 min. The aqueous solution of oxalic acid $\text{H}_2\text{C}_2\text{O}_4$ (1.2 M, 3.75 ml) was added to obtained mixture and was stirred for 1 h. After that aqueous solutions (1.25 ml) containing $\text{Zn}(\text{NO}_3)_2$ (0.4x M), $\text{Ni}(\text{NO}_3)_2$ (0.4-0.4x M), and $\text{Fe}(\text{NO}_3)_3$ (0.8 M) were added to the obtained microemulsion and stirred for 20 h at room temperature. Obtained zinc-nickel-iron oxalate was washed with methanol, dried, and milled in an agate mortar. After calcination at 400 °C for 4 h, spinel-type ferrite of $\text{Zn}_x\text{Ni}_{1-x}\text{Fe}_2\text{O}_4$ was formed.

For comparison, $\text{Zn}_x\text{Ni}_{1-x}\text{Fe}_2\text{O}_4$ materials were synthesized by co-precipitation from aqueous solutions. A route similar to that described in [25] was used. The solutions of $\text{Ni}(\text{NO}_3)_2$, $\text{Zn}(\text{NO}_3)_2$, and $\text{Fe}(\text{NO}_3)_3$ were used as initial reagents. The precipitant was an aqueous solution of NaOH.

The precipitation was carried out in several stages. At each stage determined constant pH values had been met. Initially, $\text{Fe}(\text{NO}_3)_3$ salt solutions and precipitant solutions were added to the reactor dropwise at the pH value of $4 \div 4.5$ with continuous stirring. After precipitation of $\text{Fe}(\text{OH})_3$, the pH of the mother liquor was adjusted to $7.0 \div 7.2$ and a solution of $\text{Zn}(\text{NO}_3)_2$ salt and a precipitant were added. After precipitation of $\text{Zn}(\text{OH})_2$, the pH of the mother liquor was raised to $8.5 \div 8.7$, and a solution of $\text{Ni}(\text{NO}_3)_2$ salt and a precipitant were added dropwise with continuous stirring.

After precipitation of all the components, the suspension was heated to 80 °C for 1 hour. The precipitate obtained was filtered off from the mother liquor and washed on the filter with double-distilled water. The resulting product was dried in an oven in a cuvette at the temperature of 110–120 °C. The final product was obtained after heat treatment of the precipitate in a muffle furnace with an automatic program control unit in an air atmosphere at temperatures of 600 °C, 700 °C, 800 °C, and 850 °C for two hours.

X-ray powder diffraction was performed using a DRON-4-07 diffractometer (Cu K α radiation, 40 kV, 20 mA) for the analysis of phases formed during the synthesis. The unit cell parameters of the samples were calculated using the Le Bail procedure and the FullProf software [26]. Anisotropic distribution of powder particles was ensured by sieving through a sieve onto the surface of a quartz substrate with an applied organic binder. The average stress coefficient was calculated by the Williamson-Hall method.

The size and morphology of nanoparticles were studied by transmission electron microscopy (TEM) using Scanning Transmission Microscope JEM-2100F. To study nanoparticles by the TEM method, powder particles were deposited on the Agartscintific copper mesh. Using the imageJ [27] calculations by the method of the equivalent circle diameter were performed [28]. Sampling was performed in three different regions based on at least 50 grains in each region.

The infrared absorption spectra of the sample were recorded in the wave number range 390–1800 cm⁻¹ by using an FTIR-650 spectrometer (using the technique of pressing samples from KBr where $m_{\text{sample}}/m_{\text{KBr}} = 1/50$).

The saturation magnetization (M_s), coercivity (H_c), remanence (M_r), and squareness (M_r/M_s) at room temperature were performed using a commercial VSM-250 vibrating sample magnetometer in the magnetizing field (H) range from -10 to 10 kOe.

Mössbauer spectra were obtained with an MS1101E spectrometer. Its resolution is characterized by a half-width of 0.24 mm/s for sodium nitroprusside as an absorber. The non-linearity of the velocity scan was smaller than 0.45 % at the conditions used in measurements. ⁵⁷Co in chromium with an activity of 25 mC served as a source of gamma quanta. The spectra were fitted with the Lorentz lineshape, the linewidth being considered as an iteration parameter.

The XRD patterns of the Zn–Ni ferrioxalate precursor and Zn_xNi_{1-x}Fe₂O₄ materials, calcined for 2 h at different temperatures are shown in **Fig. 1**. As the air temperature increases, metal oxalates decompose with the formation of metal oxides and carbon dioxide. It was shown that the single-phase product Zn_xNi_{1-x}Fe₂O₄ is formed after 400 °C (**Fig. 1a**). The obtained XRD diffraction patterns for each sample after 400 °C heat treatment contain the diffraction peaks of Zn_{0.4}Ni_{0.6}Fe₂O₄ with the spinel structure. No other diffraction lines were observed. The sharp peaks indicate well-crystallized grains after sintering at 600 °C. Also, for comparison, XRD data for materials synthesized by precipitation from aqueous solutions are shown (**Fig. 1b**), a single-phase product formation begins at 600 °C. Thus, microemulsion synthesis of Zn_xNi_{1-x}Fe₂O₄ particles provided a decrease in temperatures of treatment (200 °C compared with the temperature for co-precipitated ferrites).

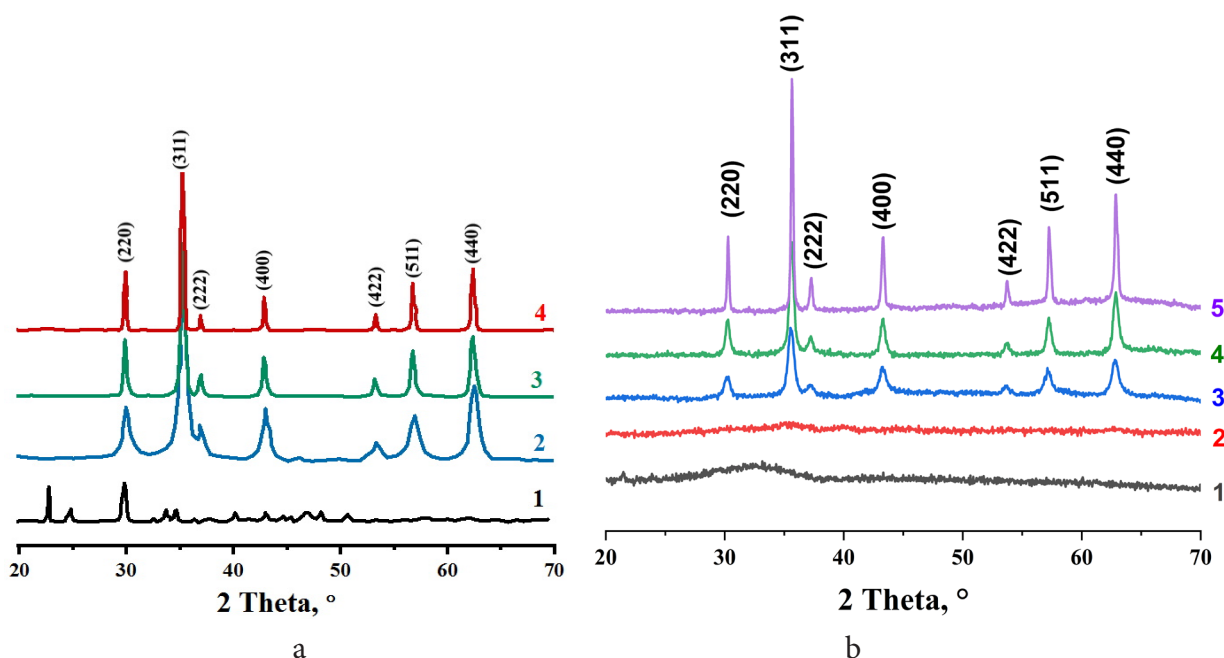


Fig. 1. a – XRD patterns of the Ni–Zn ferrioxalate precursor at $x=0.4$ (1) and $\text{Zn}_{0.4}\text{Ni}_{0.6}\text{Fe}_2\text{O}_4$ material calcined at 400 °C (2), 600 °C (3), and 800 °C (4) synthesized by the method of microemulsions;

b – XRD patterns of the $\text{Zn}_{0.4}\text{Ni}_{0.6}\text{Fe}_2\text{O}_4$ ferrite calcined at 120 °C (1), 400 °C (2), 600 °C (3), 800 °C (4), and 850 °C (5) synthesized by the precipitation from aqueous solutions.

Le Bail method was used to determine lattice parameters of ferrite particles (**Fig. 2**). It can be seen, that crystal lattice parameters in materials synthesized by the method in microemulsions increase with Zn content (**Table 1**). For instance, spinel ferrites have the general formula of AFe_2O_4 (where A: Fe, Co, Ni, Zn). The unit cell contains 32 O^{2-} ions in cubic close packing with 8 tetrahedral and 16 octahedral occupied sites. Zinc–nickel ferrite is a mixed spinel in which tetrahedral sites are occupied by Zn^{2+} and Fe^{3+} ions and the octahedral sites are occupied by Ni^{2+} and Fe^{3+} in the cubic spinel lattice. In inverse spinel NiFe_2O_4 half of the Fe^{3+} ions preferentially fill the tetrahedral sites A sites and the rest occupy the octahedral B sites. Zinc ferrite ZnFe_2O_4 is a normal spinel-type where Fe^{3+} ions occupy B sites, which

allow antiferromagnetic interactions between B–B ions to dominate in the magnetic properties of zinc ferrite. And the reason for an increase in the lattice parameter is the large Zn^{2+} ions size, larger than the radius of Ni^{2+} and O^{2-} . Therefore, we can conclude that Zn^{2+} ions enter the lattice and solid solutions of nickel-zinc ferrites are formed.

Particle size and particle morphology were estimated by transmission electron microscopy (TEM). $\text{Zn}_{0.4}\text{Ni}_{0.6}\text{Fe}_2\text{O}_4$ samples synthesized at 600 °C for the microemulsions synthesis method and 800 °C for the stepwise precipitation method with well-crystallized grains were selected for TEM studies and to calculate the lattice parameters (**Table 1**). The TEM images of the samples synthesized by both methods (**Fig. 3**) show that the Ni–Zn ferrite nanoparticles are of

uniform size of nano-dimensions and are highly crystalline. Samples exhibited good dispersion, spherical morphology and grain boundaries were well-developed. The average size of the crystallites changes slightly with the increase in zinc concentration. The grain size changes in the range of 29–42 nm (for $\text{Ni}_{1-x}\text{Zn}_x\text{Fe}_2\text{O}_4$

synthesized in microemulsions at 600 °C), 48–60 nm (at 800 °C) and 52–66 nm (for $\text{Ni}_{1-x}\text{Zn}_x\text{Fe}_2\text{O}_4$ synthesized by precipitation from aqueous solutions at 800°C) (Table 1). As can be seen (Fig. 3a, b), an increase in synthesis temperature leads to an increase in particles size.

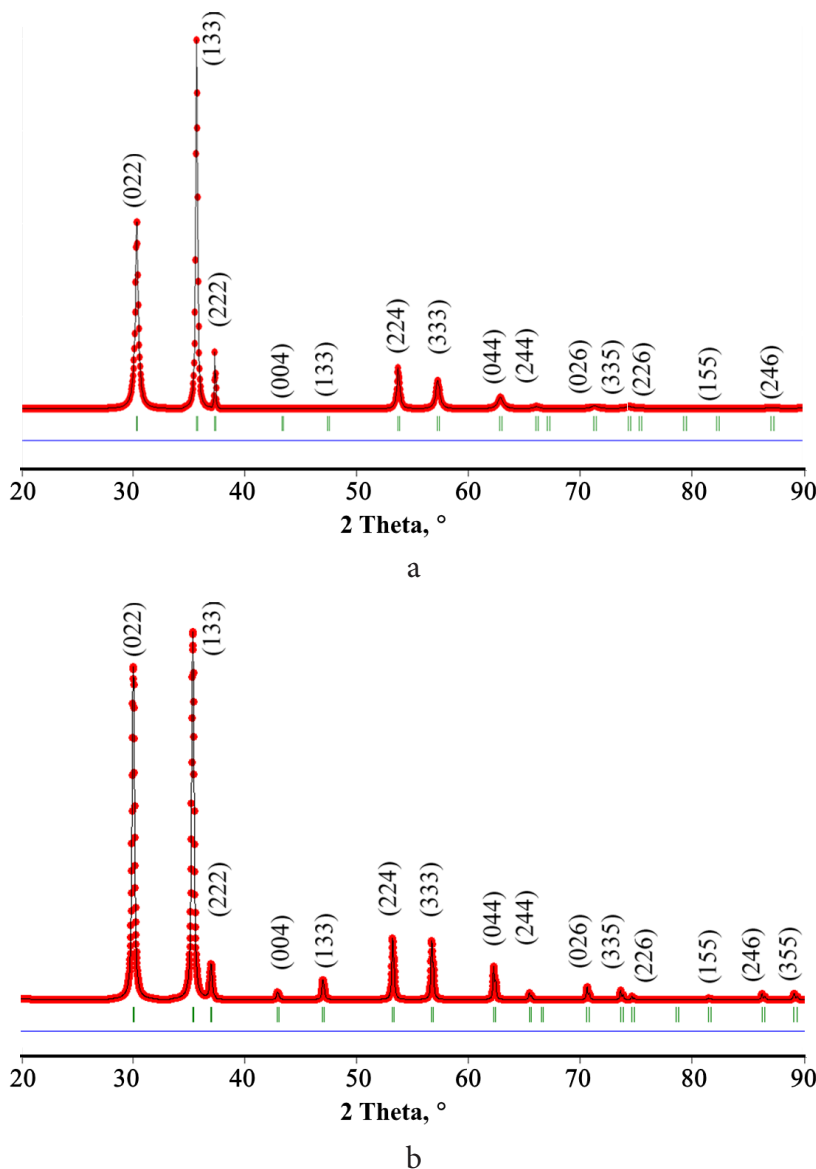


Fig. 2. Le Bail refinement of X-ray data for $\text{Zn}_{0.4}\text{Ni}_{0.6}\text{Fe}_2\text{O}_4$ ferrite synthesized by the method in microemulsions (a) and synthesized by the precipitation from aqueous solutions (b).

There was no significant change in the lattice parameter for $\text{Zn}_x\text{Ni}_{1-x}\text{Fe}_2\text{O}_4$ ferrite synthesized by both methods, as shown in **Table 1**. Variation in the lattice parameter with increasing the calcination temperature suggests a change in the cations distribution between tetrahedral A and octahedral B sites due to thermal fluctuation [29]. A change in the cations distribution causes a change in the mean ionic radii of A-site (r_A) and B-site (r_B) [30]. Lattice strain for particles synthesized by the microemulsion method is higher compared to ferrites synthesized by stepwise precipitation.

Table 1.

Lattice parameters (a), the average size of crystallites D_{TEM} of $\text{Ni}_{1-x}\text{Zn}_x\text{Fe}_2\text{O}_4$ particles synthesized by the method in microemulsions at 600 °C and synthesized by the precipitation from aqueous solutions 800 °C. R_b is the Bragg coefficient, R_f is the form conformity factor and the lattice strain η .

$\text{Ni}_{1-x}\text{Zn}_x\text{Fe}_2\text{O}_4$ synthesized by the method in microemulsions at 600 °C											
x	0.00	0.10	0.20	0.30	0.40	0.50	0.60	0.70	0.80	0.90	1.00
$a, \text{\AA}$	8.331	8.341	8.351	8.362	8.375	8.391	8.410	8.418	8.431	8.444	8.452
D_{TEM}, nm	32	37	29	36	31	35	30	36	39	42	37
$\eta, \times 10^{-3}$	0.90	1.05	1.78	1.99	2.37	2.58	3.05	3.16	3.06	2.89	2.74
$R_f, \%$	6.5	4.4	5.2	4.7	5.3	5.1	5.1	4.7	4.7	4.5	5.3
$R_{Bragg}, \%$	3.7	3.8	3.5	3.1	3.2	2.9	4.2	8.7	4.1	3.7	4.2
$\text{Ni}_{1-x}\text{Zn}_x\text{Fe}_2\text{O}_4$ synthesized by the precipitation from aqueous solutions at 800 °C											
x	0.00	0.10	0.20	0.30	0.40	0.50	0.60	0.70	0.80	0.90	1.00
$a, \text{\AA}$	8.387	8.393	8.399	8.407	8.426	8.430	8.433	8.447	8.454	8.472	8.483
D_{TEM}, nm	52	54	58	63	55	52	62	58	64	66	60
$\eta, \times 10^{-3}$	0.64	0.68	0.71	0.83	0.85	0.87	0.90	0.94	1.14	0.98	0.97
R_f	5.8	5.4	5.4	4.5	5.7	4.6	5.6	4.4	5.3	5.2	4.8
R_{Bragg}	4.2	4.7	4.2	3.6	4.2	3.5	4.6	3.7	4.5	4.4	3.7

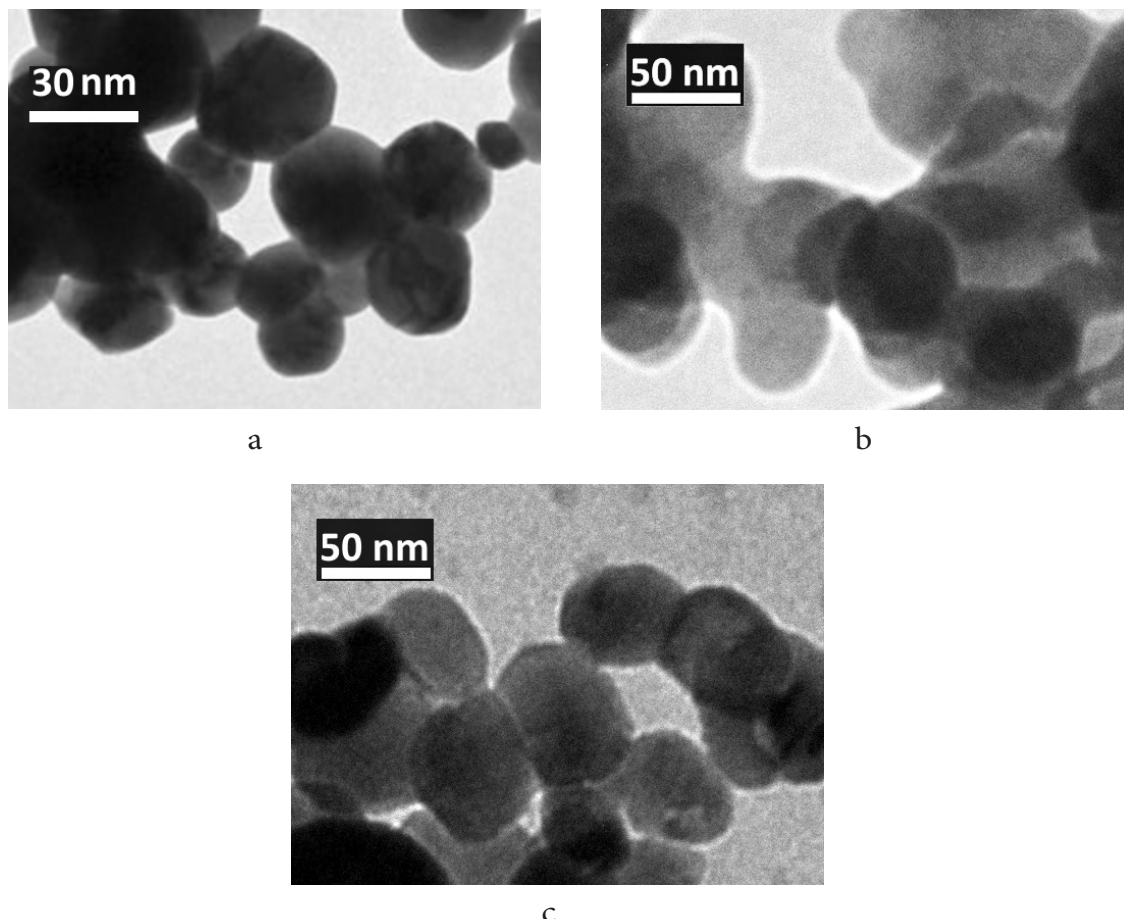


Fig. 3. TEM image of $\text{Zn}_{0.4}\text{Ni}_{0.6}\text{Fe}_2\text{O}_4$ ferrite synthesized from microemulsions after heat treatment at 600 °C (a), at 800 °C (b) and by co-precipitation after heat treatment at 800 °C (c).

The $\text{Zn}_{0.4}\text{Ni}_{0.6}\text{Fe}_2\text{O}_4$ materials' characterization was also carried out by FTIR within the wavelength range of 400–2000 cm^{-1} (**Fig. 4**). Strong vibration in curve 1 at 1632 cm^{-1} is assigned to asymmetric $\nu_a(\text{CO})$ vibration and the strong vibration at 1310 cm^{-1} is assigned as symmetric $\nu_s(\text{CO})$ vibration of the oxalate group [31–33]. The observed spectral region at 1357 cm^{-1} is attributed to C–C symmetrical stretching in zinc-nickel ferrioxalate (**Fig. 4**, curve 1). These regions are absent in $\text{Zn}_{0.4}\text{Ni}_{0.6}\text{Fe}_2\text{O}_4$ (**Fig. 4**, curve 2). The peak at 823 cm^{-1} was assigned to the C–C bending vi-

bration absorption in curve 1 and disappeared in curve 2 indicating that oxalate was decomposed in heat treatment, which can be verified by XRD data. The other low energy vibration at 740 cm^{-1} in curve 1 can be related to mixed vibrations arising from stretching and bending mode including water in the lattice. Bending vibrations of Zn–O, Ni–O, and the vibration of Fe–O in the tetrahedron may be related to the two absorption peaks in the 550–670 cm^{-1} in curve 1. After calcination these peaks are absent but a single peak of metal oxides at 583 cm^{-1} in curve 2 has appeared.

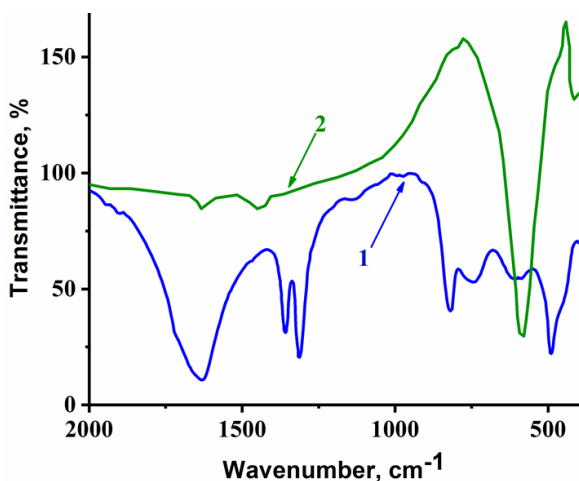


Fig. 4. Infrared spectra of zinc-nickel ferrioxalate (1) and $\text{Zn}_{0.4}\text{Ni}_{0.6}\text{Fe}_2\text{O}_4$ (2) obtained by calcining at 600 °C for 2 h.

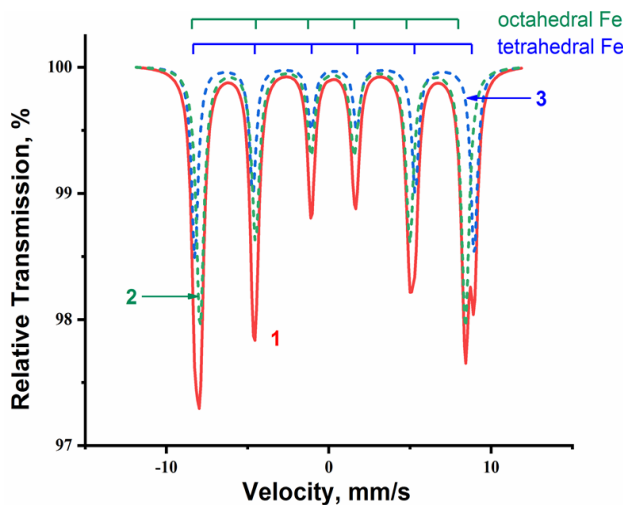


Fig. 5. ^{57}Fe Mössbauer spectra of $\text{Zn}_{0.4}\text{Ni}_{0.6}\text{Fe}_2\text{O}_4$ spinel nanoparticles at room temperature. The experimental data are represented as a bold red line (1), while two components corresponding to the octahedral and tetrahedral sites in the spinel structure are represented as green (2) and blue (3) lines, respectively.

In Fig. 5 the Mössbauer spectrum of the $\text{Zn}_{0.4}\text{Ni}_{0.6}\text{Fe}_2\text{O}_4$ ferrite at room temperature

is shown. Fe^{3+} ions occupy lattice sites in two sub-lattices of spinel, thus two sextets are observed in the spectrum. Sextet lines broaden with increasing zinc content, which occupies tetrahedral positions in the structure.

It was found that the cations in the sub-lattice are distributed randomly. The Fe^{3+} ions in the tetrahedral position give a less ultrafine field and isomeric shift compared to the Fe^{3+} ions in the octahedral field of ligands. Fe^{3+} ion has a high spin compared with Zn^{2+} , as the Zn concentration increases, the ultrafine values decrease due to the presence of Zn as the nearest neighbour of Fe.

Magnetic characteristics of powders of all compositions, synthesized at 800 °C (and at 600 °C for the synthesis from microemulsions), were determined using a vibrating magnetometer. Samples demonstrate the magnetically soft behaviour (Fig. 6).

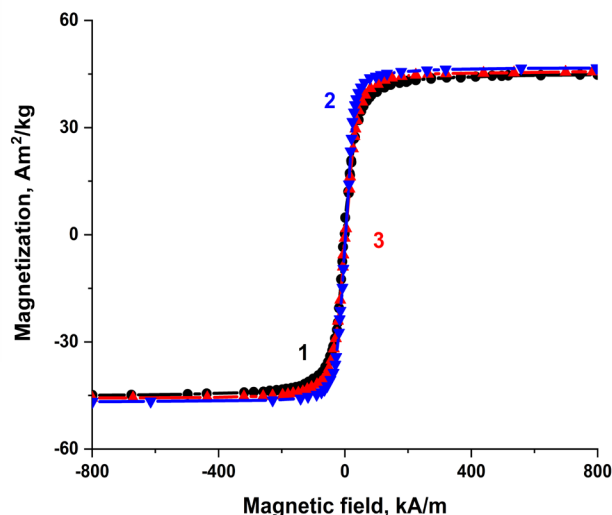


Fig. 6. Hysteresis loops at room temperature of $\text{Zn}_{0.4}\text{Ni}_{0.6}\text{Fe}_2\text{O}_4$ ferrites synthesized from microemulsions after heat treatment at 600 °C (1), at 800 °C (2) and by co-precipitation after heat treatment at 800 °C (3).

Magnetic properties change with increasing Zn concentration. The highest value of saturation magnetization was observed for $\text{Zn}_{0.4}\text{Ni}_{0.6}\text{Fe}_2\text{O}_4$ ferrite synthesized by the method of microemulsions after the heat treatment at 800 °C. The particles of the obtained powders have high saturation magnetization $M_s = 45.6 \text{ Am}^2/\text{kg}$ for synthesis from microemulsions and $M_s = 44.8 \text{ Am}^2/\text{kg}$ for co-precipitated samples, the low coercive force $H_c = 1.3 \text{ kA/m}$ and $H_c = 3 \text{ kA/m}$, respectively. Although the characteristics of particles synthesized from microemulsions appear to be slightly better, the difference for magnetization is within the margin of error.

CONCLUSIONS. The formation temperatures of single-phase spinels synthesized from microemulsions and precipitation from aqueous solutions were determined by X-ray analysis. It was found that the materials synthesized by the microemulsion method have lower particle formation temperatures (the difference is 200 °C). The parameters of the elementary cell and the average size of nanoparticles were calculated by the method of full profile analysis. It was found that with an increase in zinc, the parameter of the unit cell increases uniformly by Vegard's law. The average size of nanoparticles also increases with increasing zinc. The formation of the spinel structure was proved by the method of IR spectroscopy. Mössbauer spectroscopy showed that the cations in the sub-lattice are distributed randomly. The study of magnetic properties confirmed that the materials are magnetically soft and have a high saturation magnetization. The magnetization of materials synthesized from emulsions and precipitation from aqueous solutions differ little while coercive force differs near 3 times (1.3 and 3 kA/m) and is lower for the parti-

cles synthesized from microemulsions. At the same time, lower treatment temperatures allow the formation of smaller particles, higher stoichiometry, and homogeneity of the resulting product, making it more applicable in films and high-quality coating deposition.



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МІКРОЕМУЛЬСІЙНИЙ МЕТОД СИНТЕЗУ ЦИНК-НІКЕЛЕВИХ ФЕРИТІВ ТА ЇХНІ МАГНІТНІ ВЛАСТІВОСТІ

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Ферити – магнітні діелектрики, вирізняються серед інших матеріалів поєднанням високих магнітних характеристик та низькою електричною провідністю. Тому їх широко використовують у виробництві електронних пристроїв, різних типів не-взаємних надвисокочастотних компонентів, магнітних рідин, поглиначів, в оптиці, а також у самоконтрольованій магнітній гіпертермії. Незважаючи на давню історію досліджень, триває удосконалення методів отримання феритів та їхніх характеристик, вивчення окремо об'ємних матеріалів та нанорозмірних часток.

У роботі синтезовано тверді розчини нікель-цинкових феритів $Zn_xNi_{1-x}Fe_2O_4$ з мікроемульсій та осадженням із водних розчинів. Проведено РФА-дослідження часток, ІЧ-спектроскопію, мессбаурівську спектроскопію, дослідження магнітних властивостей часток із використанням вібраційного магнітометра. Було порівняно властивості отриманих нанорозмірних частинок феритів, синтезованих двома методами.

Використання синтезу з мікроемульсій дозволило знизити температуру формування однофазного продукту на 200 °С порівняно з осадженням із розчинів. Це також дає можливість отримання часток меншого розміру, забезпечити більшу однорідність матеріалу, краще витримати стехіометрію складу у об'ємі.

Встановлено, що збільшення вмісту цинку призводить до збільшення параметрів елементарної комірки. ІЧ-спектроскопія підтвердила повне розкладання й виведення оксалату при синтезі феритів із мікроемульсій. Мессбаурівські спектри матеріалів підтвердили присутність йонів заліза в

обох ґратках фериту й формування твердого розчину. Намагніченість частинок, синтезованих різними методами, відрізняється несуттєво, тоді як коерцитивна сила відрізняється в рази (1,3 і 3 кА/м) і є меншою для зразків, синтезованих із мікроемульсій.

Вказані параметри дозволяють стверджувати, що отримані частинки можна використовувати при створенні магнітних плівок, добротних магнітних покриттів та компонентів на їхній основі.

Ключові слова: $NiFe_2O_4$, $ZnFe_2O_4$, магнітні властивості, постійна ґратки, заміщення цинку.

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