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## **Effect of surfactant type on the magnetic and morphological properties of NiFeO<sub>4</sub>/ reduced graphene oxide composites**

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A systematic study of the effect of various types of surfactants (polyethylene glycol, Trilon X-100 and cetyltrimethylammonium bromide) and subsequent annealing on the magnetic ordering and morphology of the nickel ferrite / reduced graphene oxide composite materials has been carried out. Hydrazine hydrate has been used both as an agent to initiate precipitation and as a reducing agent to remove oxygen functionality from the surface of graphene oxide particles. To characterize the material, Mössbauer spectroscopy and nitrogen absorption porosimetry have been used. The obtained result suggests a new way for one-pot synthesis of NiFe<sub>2</sub>O<sub>4</sub>/rGO composite materials with controlled magnetic ordering and porous structure.

**Keywords:** graphene oxide, nanocomposite, nickel ferrite, Mössbauer spectroscopy.

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### **Introduction**

Scientific and technological progress in nanotechnology requires new functional nanomaterials with predetermined physical and chemical properties. Composite carbon-containing materials have attracted a lot of attention due to their potential applications in energy storage devices, electromagnetic wave absorption, biotechnology, etc. [1]. Great efforts have been made for composites based on functionalized graphene materials, especially reduced graphene oxide (rGO). rGO obtained from graphene oxide (GO) is actively studied, in particular, due to the relatively low technological cost and the possibility of changing the surface properties from hydrophilic to hydrophobic with an increase in the reduction degree with a simultaneous change in the electronic structure [2]. Composites based on transition metal oxides (including those with a spinel structure) and rGO are a fundamentally multifunctional material with tailoring properties and a wide application potential. The key point in many cases is a controllable balance between the morphological and magnetic characteristics of

composite materials for the absorption of microwave radiation or hyperthermic therapy of cancerous tumors [3]. Progress in this area requires the improvement of synthesis methods and the development of algorithms for controlling their properties. The aim of this work is to establish the effect of various types of surfactants on the magnetic and morphological properties of nickel spinel / reduced graphene oxide (NiFe<sub>2</sub>O<sub>4</sub> / rGO) composite materials.

### **I. Experimental details**

Colloidal GO was synthesized by the modified Hummers method [4]. Graphite powder (Aldrich, # 282863) with a particle size of 20 μm or less was used as a starting material. The synthesis protocol included the following steps. Graphite powder (15 g) was slowly mixed with 350 ml of sulfuric acid at 0°C and KMnO<sub>4</sub> (45 g) was added with continuous stirring. The temperature of the mixture was slowly raised to 40-50°C with the gradual addition of water (300 ml), resulting in an exothermic

reaction. Hydrogen peroxide (30% aqueous solution, 100 ml) was added with continuous stirring and a yellow-brown colloidal solution was collected. After several stages of ultrasonication and centrifugation, a highly stable colloidal solution was obtained with a final GO concentration of 2.7 mg / mL.

Metal nitrate solutions were prepared by dissolving Ni(NO<sub>3</sub>)<sub>2</sub>·6H<sub>2</sub>O and Fe(NO<sub>3</sub>)<sub>3</sub>·9H<sub>2</sub>O in distilled water. The resulting solutions (50 ml each) were mixed at a temperature of 30-40°C for 1 hour at a Ni/Fe molar ratio of 1:2. The composition of the precursors was controlled using an Expert 3L XRF analyzer (accuracy 0.1%). The final pH of the mixture was 2.3. Separately, surfactant solutions were prepared: polyethylene glycol (CH<sub>2</sub>n+2O<sub>n+1</sub>, PEG-6000) in an amount of 10 g/L, cetyltrimethylammonium bromide ([C<sub>16</sub>H<sub>33</sub>N(CH<sub>3</sub>)<sub>3</sub>]Br, CTAB) in an amount of 2.5 g/L and TritonX-100 (C<sub>14</sub>H<sub>22</sub>O(C<sub>2</sub>H<sub>4</sub>O)<sub>10</sub>, TrX-100) in an amount of 8 g/L at 40-50°C. The resulting solutions were slowly added dropwise to a mixture of nickel and iron nitrates with continuous stirring for 1.5-2.0 hours. At the next stage, a colloidal GO presonicated (20 kHz, 150 W) (150 ml) and preheated to 40°C was added. In all cases, an increase in the density of the solution and the formation of fine agglomerates were observed after the addition of GO. All solutions become homogeneous brownish-green in color after stirring at 75-80°C for 1 hour. A 10% aqueous solution of hydrazine hydrate N<sub>2</sub>H<sub>4</sub>·H<sub>2</sub>O (total 100 mL) was slowly added dropwise to the mixtures. For all graphene-containing materials, a gradual color change from green-brown to dark brown and then to black was observed. The color of the mixture without GO changed from green-yellow to light brown. After sedimentation, washing and drying at 90°C, the following systems were obtained: X0 – without surfactants, without GO; X1 – without surfactants, the presence of GO; X2 – with the addition of PEG-6000 and GO; X3 – with the addition of CTAB and GO; X4 – with the addition of Triton-X and GO. Annealing in an argon atmosphere was carried out for all as-synthesized samples

Mössbauer studies were carried out on an MS-1104 Em spectrometer, the linewidth of metallic  $\alpha$ -Fe was 0.29 mm/s; the isomer shift was calibrated relative to  $\alpha$ -Fe. The Mössbauer spectra were analyzed using the UnivemMS 7.01 software.

N<sub>2</sub> sorption study at 77 K of the morphology of materials was carried out on a Quantachrome Nova 2200e. The specific surface area (S<sub>BET</sub>, m<sup>2</sup>/g) was calculated by the multipoint BET method. The calculation of the pore size distribution was carried out by the NLDFT method in the approximation of slit-like pores [5].

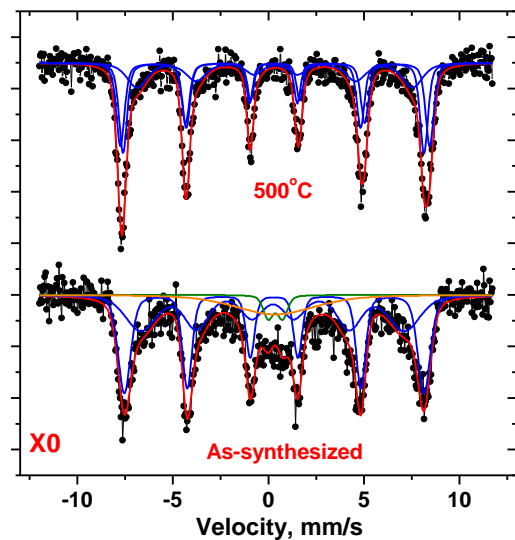
## II. Results and discussion

A decrease in the size of ultrafine ferrite particles causes an increase in its surface energy, and when this value becomes comparable with the specific magnetic energy of the particle, the single-domain uniformly magnetized state becomes thermodynamically favorable. Spinel ferrites, in particular nickel, due to the symmetry of the arrangement of magnetic cations in the crystal lattice, have uniaxial magnetic anisotropy with several (at least two) paired directions of the possible orientation of the

magnetic moment of the particles. The states with the opposite orientation of the magnetic moment are separated by a potential barrier  $KV$  ( $K$  is the constant of the uniaxial magnetic anisotropy of the material,  $V$  is the particle volume) [6]. Thermally induced oscillations of the magnetic moment of a particle between easy magnetization axes lead to the observation of a ferromagnetic-paramagnetic transition at  $kT \geq KV$ . The Néel relaxation time ( $\tau_N$ ) of the magnetic moment localization

is calculated as follows  $\tau_N = \tau_0 \exp\left[\frac{KV}{k_0T}\right]$ , where  $\tau_0$  is

determined by the crystal structure, for spinel ferrites  $\tau_0$  is about  $10^{-10}$  s. When  $\tau_N$  becomes less than the observation time, the particle will be observed as paramagnetic. The observation time for Mössbauer spectroscopy is the lifetime  $\tau_m$  of the excited state of the <sup>57</sup>Fe nucleus –  $\tau_m = 1.42 \cdot 10^{-7}$  s. The Mössbauer spectra of X0 samples before and after annealing at 500°C (labeled as X0-500) are shown in Fig. 1.



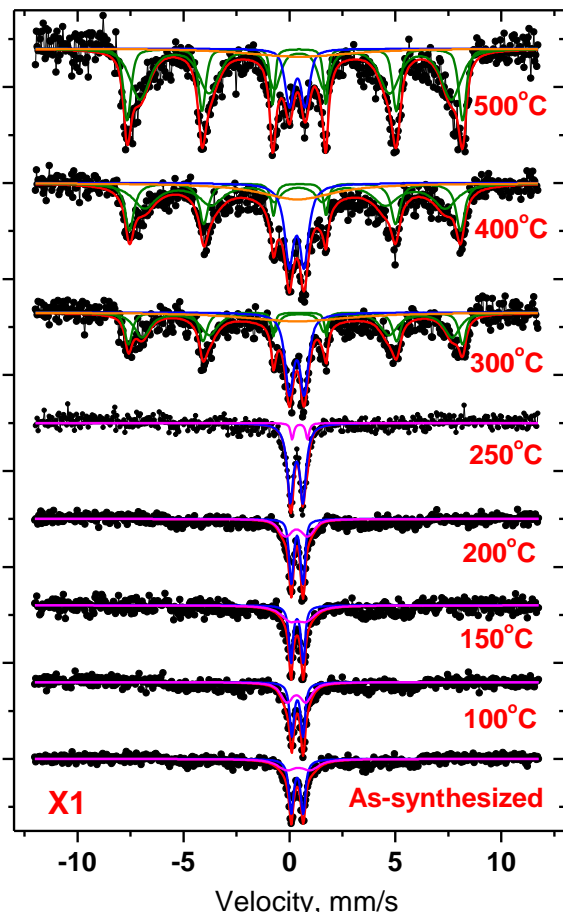
**Fig.1.** Mössbauer spectra of X0 material: (a) as-synthesized and (b) annealed at 500°C.

The optimal approximation of the spectrum of the as-synthesized material was made by two sextet components (corresponding to the resonant absorption of  $\gamma$ -quanta by iron nuclei in tetrahedral and octahedral crystallographic sites of a spinel structure). The spectra contain two doublet components. The first one corresponds to Fe<sup>3+</sup> ions in the paramagnetic state due to the transition of ultrafine nickel ferrite particles to the superparamagnetic state. The second one is the result of the presence of ferrite particles with a balance of sizes and magnetic anisotropy, which determine the intermediate state of magnetic ordering at room temperature. The presence of a paramagnetic component in the Mössbauer spectra of nanoparticles of ferromagnets is a consequence of superparamagnetic relaxation. The value of the magnetic anisotropy constant of nickel-ferrite spinel at 300 K is about  $10^5$  J/m<sup>3</sup> [7]. The Néel relaxation time was calculated depending on the particle diameter for  $K = 1 \cdot 10^5$  J/m<sup>3</sup>. The ferromagnetic-superparamagnetic transition at room temperature can be expected for nickel ferrite particles smaller than 7.3 nm.

The Mössbauer spectra of the as-synthesized X1

material (graphene-containing composite obtained without surfactants) indicate only the paramagnetic state of the material.

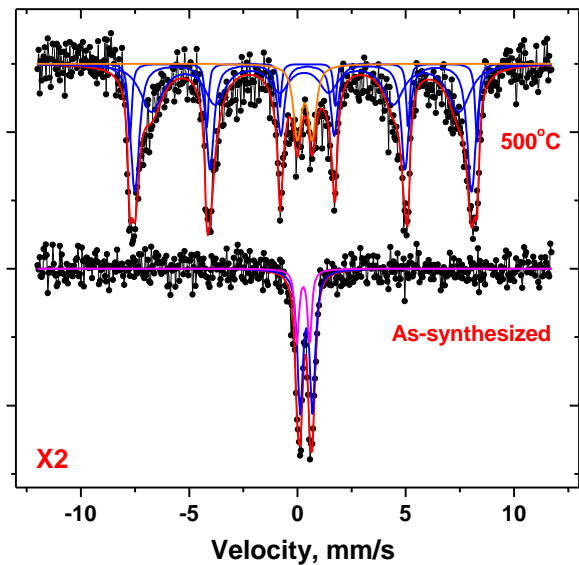
The spectra were optimally fitted by two doublet components associated with high-spin  $\text{Fe}^{3+}$  in different crystal coordination. An increase in the annealing temperature causes the growth of individual particles, but only in the temperature range of 250-300°C these sizes exceed the transition threshold to the superparamagnetic state, which is reflected by the appearance of sextet components corresponding to iron ions in hematite and nickel spinel particles larger than about 8 nm. The phases were identified by the values of the quadrupole splitting  $\Delta$  ( $-0.21 \pm 0.02$  and  $0 \pm 0.05$  mm/s for  $\alpha\text{-Fe}_2\text{O}_3$  and  $\text{NiFe}_2\text{O}_4$ , respectively). An increase in the annealing temperature and a corresponding increase in the particle size lead to a decrease in the content of the doublet component (the amount of  $\text{Fe}^{3+}$  excluded from superexchange interaction decreases), but even after annealing at 500°C, the fraction of particles (up to 10%) retains superparamagnetic properties. The Mössbauer spectra of X1 materials annealed at a temperature of 300-500°C contain broad components corresponding to the intermediate ferromagnetic-superparamagnetic phase.



**Fig.2.** Mössbauer spectra of X1 material: as-synthesized and annealed in the temperature range of 100-500°C.

The Mössbauer spectra of X2 material (graphene-containing composites obtained by PEG-6000 assisted route), both as-synthesized and annealed, are close in properties to X1 system (Fig.3). All X2 materials contain two oxide phases,  $\text{NiFe}_2\text{O}_4$  and  $\alpha\text{-Fe}_2\text{O}_3$ , which change

from superparamagnetic to ferromagnetic arrangement, and for X2-500 material, about 10 % of oxide particles remain monodomain.

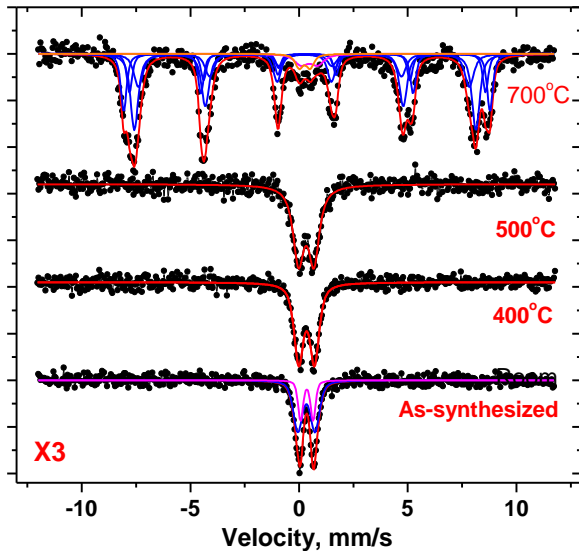


**Fig.3.** Mössbauer spectra of X2 materials: as-synthesized and annealed at 500°C.

The spectra of the as-synthesized X3 material (graphene-containing composites obtained using the CTAB assisted route) also contain two doublet components (relative intensities of 70% and 30%) with close values of isomer shift, but different quadrupole splitting values (0.79 and 0.56 mm/s, respectively). The presence of two components can be explained by the difference in the magnetic surrounding for  $\text{Fe}^{3+}$  ions, which form the inner (respectively, more ordered) and outer (contacted with the graphene component) parts of ferrite particles. The paramagnetic state of the material does not change after annealing at 400 and 500°C, but the composition of the spectrum changes – only one doublet component (linewidth values of 0.51 and 0.60 mm/s, respectively). The stability of the quadrupole splitting values for samples X3-400 and X3-500 (0.72 and 0.71 mm/s) indicates the stability of the close surrounding of  $\text{Fe}^{3+}$  ions in the crystal lattice. The growth of particles after annealing at 700°C causes the transformation of the magnetic microstructure of the composite.

In the spectra of X3-700 material, magnetic splitting predominates, but there is also a superparamagnetic component (Fig. 4). These spectra have a complex composition; it was optimally fitted with 4 sextet and 2 doublet components, which correspond to differences in the  $\text{Fe}^{3+}$  surrounding that cause changes in the effective magnetic fields at the nuclei and quadrupole splitting values. The association of  $\text{Fe}^{3+}$  ions with tetrahedral and octahedral sites of the crystal structure was made using the values of the isomeric shift ( $\delta$ ) of each sextet component, since  $\delta$  for tetrahedrally coordinated iron ions is lower due to the correspondingly greater strength of Fe-O covalent bonds [8]. An analysis of the integral intensities of the sextet components makes it possible to calculate the distribution of  $\text{Fe}^{3+}$  ions between the tetra (A)- and octa (B)-sublattices:  $(\text{Ni}_{0.07}\text{Fe}_{0.95})_{\text{A}}[\text{Ni}_{0.93}\text{Fe}_{1.05}]_{\text{B}}\text{O}_4$ . Nickel ferrite is an inverse spinel structure in which all  $\text{Ni}^{2+}$

cations are in B-sites, while Fe<sup>3+</sup> cations occupy both A- and B-sites. The formation of a mixed structure causes a decrease in the particle size and a nonequilibrium state of the composite material [9]. The relative content of Fe<sup>3+</sup> ions in superparamagnetic particles is about 8% due to the relatively high annealing temperature (700°C), which indicates the high stability of oxide nanoparticles in the NiFe<sub>2</sub>O<sub>4</sub>/rGO composite.

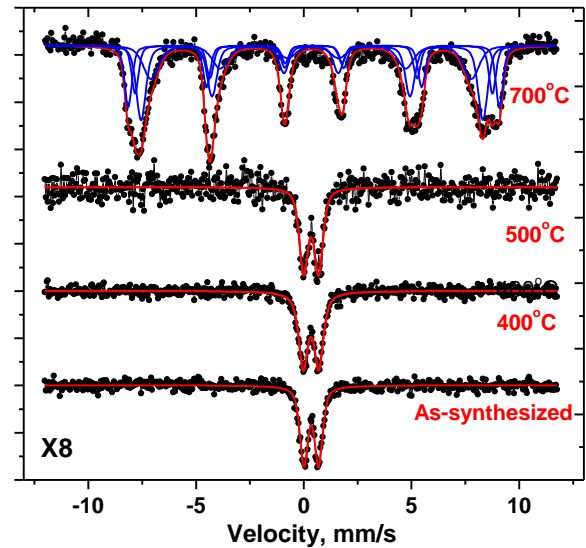


**Fig.4.** Mössbauer spectra of X3 materials: as-synthesized and annealed at 400, 500 and 700°C.

The Mössbauer spectrum of the as-synthesized X4 material (graphene-containing composites obtained by TRX-100 assisted route) consists of only one doublet component (linewidth of 0.44 mm/s,  $\Delta=0.68$  mm/s), which corresponds to a correspondingly lower concentration of surface defects compared to the as-synthesized X3 composite materials. Another indication of its relatively higher structural order is thermally induced changes in the linewidth. The linewidth of single doublet spectra of X4-400 sample is 0.51 mm/s with a slight increase in the quadrupole splitting (up to 0.72 mm/s), but a further increase in the annealing temperature to 500°C leads to a decrease in the linewidth (to 0.49 mm/s), which correlates with a decrease in the quadrupole splitting (to 0.69 mm/s). The magnetic ordering was completely restored after annealing at 700°C (X4-700 material), as follows from the Mössbauer data. Optimal fitting results were also achieved for 4 magnetic sextets.

The quadrupole splitting values of the sextet components are very close to the parameters of the components of the spectra of the X3-700 sample. The calculated distribution of cations for the X4-700 sample is  $(Ni_{0.03}Fe_{0.97})_A[Ni_{0.97}Fe_{1.03}]_B O_4$ , which is close to expected. However, correspondingly high linewidth values of the spectral component indicate the presence of structural disorder.

An analysis of the N<sub>2</sub> adsorption-desorption isotherm makes it possible to obtain independent information on the next-level structural properties of the NiFe<sub>2</sub>O<sub>4</sub>/rGO composites.

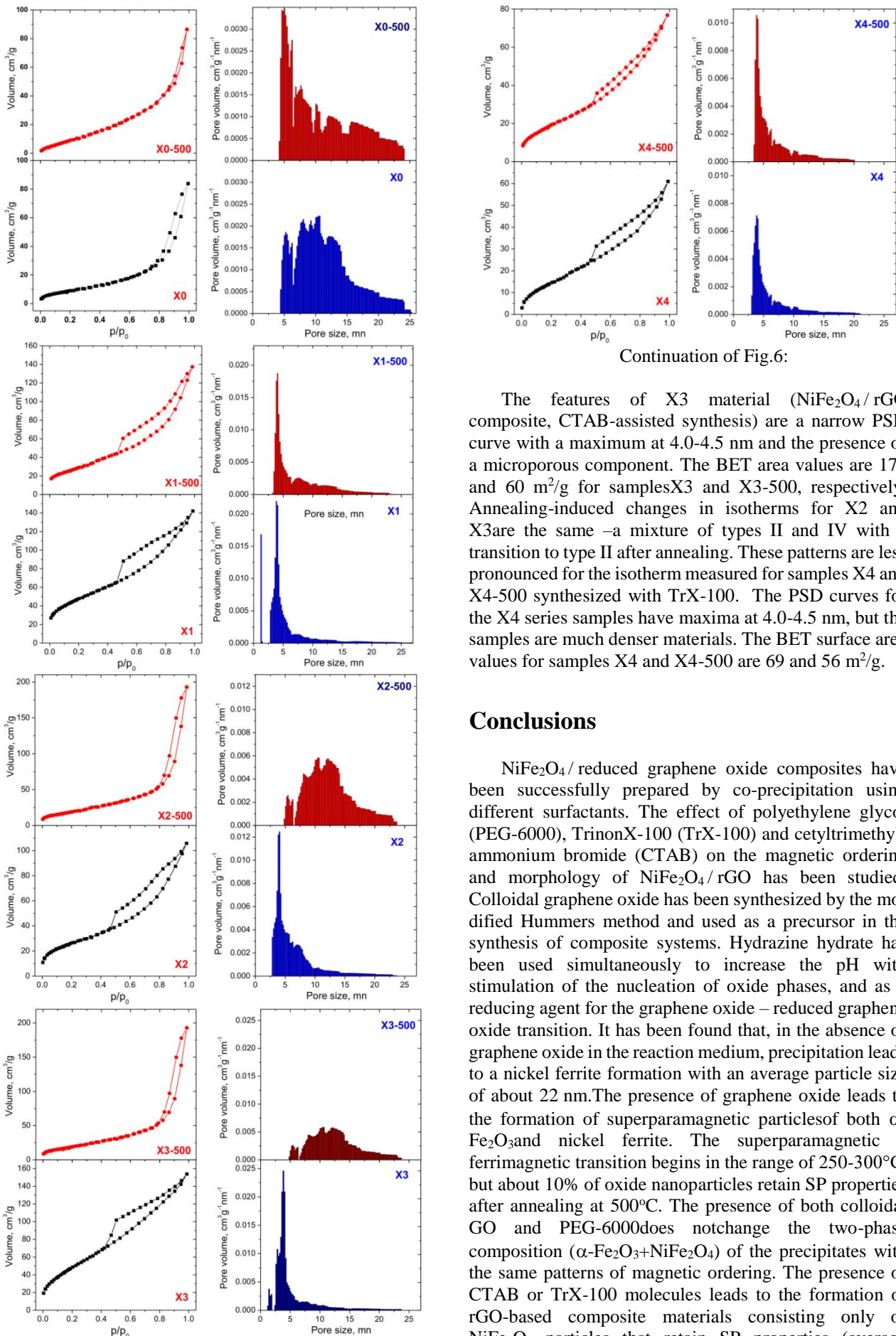


**Fig.5.** Mössbauer spectra of X3 materials: as-synthesized and annealed at 400, 500 and 700°C.

According to the IUPAC classification, the X0 sample, which demonstrates mixed isotherms of types II and IV, indicates the presence of both macro- and mesopores in accordance with the unsaturated character at a partial pressure close to  $p/p_0=1$ , and the presence of a hysteresis loop of type H3 [10]. The calculated pore size distribution (PSD) shows a wide range of pore diameters with maxima around 5 nm followed by a slight decrease in differential pore volume to a pore size of around 27 nm. Annealing at 500°C causes an increase in the content of mesopores with sizes in the range of 5-10 nm. Brunauer-Emmett-Teller (BET) specific surface area values are 32 and 25 m<sup>2</sup>/g for samples X0 and X0-500 (pure nickel ferrite).

The isotherms obtained for as-synthesized X1 samples also correspond to a mixture of types II and IV, but in this case the features of the hysteresis loop involve reversible polymolecular adsorption by the mesoporous structure. The DFT pore size distribution curve has a maximum at 4.5 nm, but there is also a micropore component with a narrow distribution around 2.0 nm. These pores can be attributed to the interstices between rGO particles, when the fraction of mesopores corresponds to the volume between agglomerates. The isotherm and pore size distribution curve measured for rGO-containing samples as-synthesized and sintered at 500°C (surfactant-free synthesis route) are close, but annealing causes the disappearance of micropores. The calculated BET surface area for the X1 sample is 172 m<sup>2</sup>/g, and for the X1-500 sample there is a decrease to 102 m<sup>2</sup>/g.

A similar isotherm and, accordingly, a narrow mesopore distribution in the range of 5-10 nm are observed for the X2 sample synthesized by PEG-assisted route, but annealing at 500°C leads to a sharp change in the morphology with a decrease in the BET surface area from 93 to 61 m<sup>2</sup>/g when compacting the material (Fig.6, c).



Continuation of Fig.6:

The features of X3 material ( $\text{NiFe}_2\text{O}_4/\text{rGO}$  composite, CTAB-assisted synthesis) are a narrow PSD curve with a maximum at 4.0-4.5 nm and the presence of a microporous component. The BET area values are 178 and 60  $\text{m}^2/\text{g}$  for samples X3 and X3-500, respectively. Annealing-induced changes in isotherms for X2 and X3 are the same – a mixture of types II and IV with a transition to type II after annealing. These patterns are less pronounced for the isotherm measured for samples X4 and X4-500 synthesized with TrX-100. The PSD curves for the X4 series samples have maxima at 4.0-4.5 nm, but the samples are much denser materials. The BET surface area values for samples X4 and X4-500 are 69 and 56  $\text{m}^2/\text{g}$ .

## Conclusions

$\text{NiFe}_2\text{O}_4/\text{reduced graphene oxide}$  composites have been successfully prepared by co-precipitation using different surfactants. The effect of polyethylene glycol (PEG-6000), TrilonX-100 (TrX-100) and cetyltrimethylammonium bromide (CTAB) on the magnetic ordering and morphology of  $\text{NiFe}_2\text{O}_4/\text{rGO}$  has been studied. Colloidal graphene oxide has been synthesized by the modified Hummers method and used as a precursor in the synthesis of composite systems. Hydrazine hydrate has been used simultaneously to increase the pH with stimulation of the nucleation of oxide phases, and as a reducing agent for the graphene oxide – reduced graphene oxide transition. It has been found that, in the absence of graphene oxide in the reaction medium, precipitation leads to a nickel ferrite formation with an average particle size of about 22 nm. The presence of graphene oxide leads to the formation of superparamagnetic particles of both  $\alpha\text{-Fe}_2\text{O}_3$  and nickel ferrite. The superparamagnetic – ferrimagnetic transition begins in the range of 250-300°C, but about 10% of oxide nanoparticles retain SP properties after annealing at 500°C. The presence of both colloidal GO and PEG-6000 does not change the two-phase composition ( $\alpha\text{-Fe}_2\text{O}_3 + \text{NiFe}_2\text{O}_4$ ) of the precipitates with the same patterns of magnetic ordering. The presence of CTAB or TrX-100 molecules leads to the formation of rGO-based composite materials consisting only of  $\text{NiFe}_2\text{O}_4$  particles that retain SP properties (average particle size of nickel ferrite is less than 7-8 nm) after

**Fig.6.** Adsorption/desorption isotherms and pore size distribution for sample series X1, X2, X3, and X4.

annealing at 500°C. Restoration of magnetic ordering is observed after annealing at 700°C. The presence of only colloidal GO or both GO and TrX-100 causes the formation of a temperature-stable (at least up to 500°C) narrow distribution of mesopores with sizes in the range of 4-10 nm. In other cases, annealing leads to morphological transformations with the disappearance of small mesopores and pore-size distribution widening.

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## **Вплив поверхнево-активних речовин на магнітні та морфологічні властивості композитів NiFeO<sub>4</sub> / відновлений оксид графену**

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В роботі здійснено систематичне вивчення впливу різних типів поверхнево-активних речовин (поліетиленгліколю, Trilon X-1006 цетилтриметиламмонію броміду) та подальшого відпалу на магнітне впорядкування та морфологію композитних матеріалів на основі нікелевого фериту та відновленого оксиду графену. Гідразин гідрат використовувався водночас як агент для ініціювання осадження, так і як відновник для видалення кисневмісних функціональних груп з поверхні частинок оксиду графену. Застосовувалися месбауерівська спектроскопія та низькотемпературна адсорбційна порометрія. Отриманий результат пропонує новий спосіб однокрокового синтезу композиційних матеріалів NiFe<sub>2</sub>O<sub>4</sub> / відновлений оксид графену з контрольованими магнітним упорядкуванням і пористою структурою.

**Ключові слова:** оксид графену, нанокompозит, нікелевий ферит, месбауерівська спектроскопія.