

## SYNTHESIS, MICROSTRUCTURE AND MAGNETIC PROPERTIES OF BINARY Ni–Zn BASED FERRITES

*Plutenko M.O.*<sup>1</sup>, *Fedorchuk O.P.*<sup>2</sup>, *Plutenko T.O.*<sup>2</sup>, *Torchyniuk P.V.*<sup>2</sup>

<sup>1</sup>Taras Shevchenko National University of Kyiv, Faculty of Chemistry, Kyiv, Ukraine

<sup>2</sup>V.I. Vernadsky Institute of General and Inorganic Chemistry, Kyiv, Ukraine

alex1377c9@gmail.com

The use of nanosized Ni-Zn spinel ferrites opens up new perspectives in electronics, information technology, media, biomedicine, and ferrofluids. Nanoscale ferrites exhibit properties such as superparamagnetism, single-domain structure, and chemical stability. To obtain nanosized spinel materials, various methods are used, such as sol-gel [1], coprecipitation [2], hydrothermal [3], inverse micelle [4], and microemulsion [5]. The physical properties of these nanomaterials largely depend on the synthesis method. The sol-gel combustion synthesis includes nitrates and is commonly used as the method for producing ferrite nanoparticles. It is easy, cheap, and provides an uniform size distribution, small particle size. Glycine, tartaric acid, L-ascorbic acid, sucrose, dextrose, acid, acetic acid, ethylene glycol, and urea have been described as fuels [6-10]. The size and shape of particles affect the magnetic properties of nanoferrites. In addition, magnetic and electrical properties of spinel ferrites primarily depend on magnetic interactions between cations, magnetic moments at tetrahedral A sites and octahedral B. Zn-Ni ferrites have high electrical resistivity, moderate saturation magnetization, low dielectric loss, good chemical stability has attracted considerable interest of researchers in the fields of miniaturization of electronic devices, hyperthermia, non-reciprocal components development.

The aim of the presented work was the synthesis of nanocrystalline Ni-Zn ferrite spinel by using urea as a precursor and to study the effect of nonmagnetic zinc ions on microstructure and magnetic properties.

Zn-Ni spinel ferrite nanoparticles of the general chemical formula  $\text{Ni}_{1-x}\text{Zn}_x\text{Fe}_2\text{O}_4$  with  $0 \leq x \leq 0.9$  were synthesized by the sol-gel method. Nickel, zinc, and iron nitrates were used as precursors. The ratio of metal nitrates to urea was 1 : 3. Weighted metal nitrates were dissolved separately in distilled water and mixed. Urea was also dissolved in distilled water and then added to a mixed solution of metal nitrates. The mixed solution was stirred at 100 °C for 4 hours. With the formation of a viscous gel, the temperature was then increased to 140 °C. Auto combustion of the gel was carried out and the powder was formed. The prepared fine powder was heat-treated at a temperature of 700 °C for 6 hours.

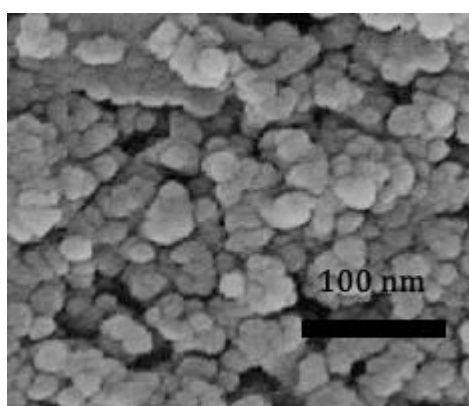
To study the formation of the spinel ferrite phase, the TGA-DTA method in a nitrogen atmosphere was used. It was found that at temperatures above 450 °C organic components are removed completely. In addition, the TGA curve shows no significant weight loss after a temperature of 620 °C, which can be attributed to the ferritic phase formation.

Phase identification and analysis of samples were performed by X-ray studies. It was shown that a single-phase cubic structure of spinel formed. The lattice parameter increases with increasing Zn content and obeys Vegard's law. The increase in the lattice parameter with increasing Zn content can be explained on the basis of the ionic radius of Zn.

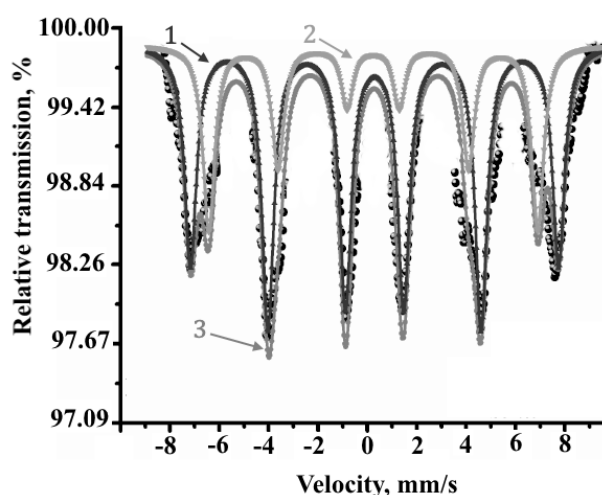
The primary analysis of the FESEM image in Fig. 1 shows the formation of spherical geometry grains. The fine grain size can be easily observed in FESEM images.

Agglomerations were also observed, which can be explained by high surface energy due to the nanocrystalline nature of the obtained spinel ferrite nanoparticles and magnetic interactions between them. Analysis of FESEM images will allow noticing the smallest grain size is 35 nm, while the largest size is 64 nm.

The Mossbauer spectra at room temperature for  $\text{Ni}_{1-x}\text{Zn}_x\text{Fe}_2\text{O}_4$  are shown in Fig. 2. The obtained Mossbauer spectra were adjusted to take into account two ultrathin magnetic sextets. The width of the line of tetrahedral sections is wider than that of octahedral sections. The expansion of the line width can occur due to the distribution of ultrafine fields caused by the cationic distribution of  $\text{Zn}^{2+}$ ,  $\text{Ni}^{2+}$ , and  $\text{Fe}^{3+}$  ions in the available tetrahedral A and octahedral B sites. In addition, it is observed that the isomeric shift for octahedral regions is greater than for tetrahedral regions, and decreases with zinc substitution. In cubic spinel structures the bond length of  $\text{Fe}^{3+} - \text{O}^{2-}$  is greater in octahedral sites than in tetrahedral sites.



**Fig. 1.** SEM image of  $\text{Ni}_{1-x}\text{Zn}_x\text{Fe}_2\text{O}_4$  ( $x=0.5$ ).



**Fig. 2.** Mossbauer spectra for sample of  $\text{Ni}_{1-x}\text{Zn}_x\text{Fe}_2\text{O}_4$  ( $x=0.5$ ). 1 - first sextet, 2 – second sextet, 3 – fitted curve.

Since the observed values of an isomeric shift in this study are less than 0.5 mm/s the possibility of the presence of  $\text{Fe}^{2+}$  ions is excluded. Quadrupole values of cleavage are also insignificantly small. This is due to the preserved cubic symmetry between  $\text{Fe}^{3+}$  and the surrounding ions with substituted  $\text{Zn}^{2+}$  ions.

Substituted zinc-nickel spinel ferrite nanoparticles have been successfully obtained using urea for the synthesis of sol-gel combustion. The X-Ray spectra revealed a single-phase cube spinel structure and the substitution of Zn ions increased the lattice parameter. The spherical shape of Ni-Zn spinel ferrites particles was detected by SEM and TEM analysis. The saturation magnetization increases to  $x = 0.5$  and decreases at other values of  $x$ . The distribution of ultrafine interactions caused an expansion of the line width due to the distribution of  $\text{Zn}^{2+}$ ,  $\text{Ni}^{2+}$  and  $\text{Fe}^{3+}$  cations with tetrahedral A and octahedral B regions available.

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