STRUCTURE AND PROPERTIES OF $Ni_{0.7}Zn_{0.3}Fe_2O_4$ FERRITE NANOPARTICLES SYNTHESIZED BY SOLID-STATE REACTION METHOD

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Abstract: *The main aim of this study is to determine the structure and properties of the $Ni_{0.7}Zn_{0.3}Fe_2O_4$ ferrite nanoparticles (FNP) synthesized by solid-state reaction method under the laboratory conditions of the unit and compare them with those of world publication. Ferrite structure was determined by X-ray diffraction (XRD), ferrite crystallite size (D) was determined by scanning electron microscope (SEM) and Dybye-Sherer equation, chemical composition was determined by energy – dispersive X-ray spectroscopy (EDS), the magnetic properties of ferrite were determined by vibrating sample magnetometer (VSM). The results of $Ni_{0.7}Zn_{0.3}Fe_2O_4$ FNP properties have obtained: Saturation magnetization (M_s) 57 emu/g, coercivity (H_c) 114 Oe, the remanence (M_r) 10 emu/g and D of about 50 nm.*

Keywords: $Ni_{0.7}Zn_{0.3}Fe_2O_4$ ferrite nanoparticles; Magnetic properties; Crystallite size.

1. INTRODUCTION

Nanoscale magnetic ferrite materials in general and nickel-zinc FNP have $Ni_xZn_{1-x}Fe_2O_4$ general formula in a particular play, keep a very important role in many areas such as electronics, medicine, especially in the military, etc [1-9]. There are many different ways to synthesize nickel-zinc FNP depending on the purpose of use, the method of fabrication as well as the formula reasonable. $Ni_xZn_{1-x}Fe_2O_4$ NFP (where x = 0.1, 0.3) were synthesized using chemical co-precipitation method, and have their novel physical aspects like spin, supper magnetism and surface anisotropy at room temperature, so that wide ranging applications in high density information storage devices, transformer cores, magnetic fluids [5]. Nickel zinc FNP were synthesized by sol-gel technique that are used electronic devices such as EMS suppressor and electromagnetic wave absorber due to their low initial permeability [9]. $Ni_{0.7}Zn_{0.3}Fe_2O_4$ FNP were synthesized by solid state reaction that annealed at 30, 500, 800, 1000 and 1200 ^oC for 3 hours (h). It was found that the optimum temperature is 1000 ^oC with Saturation magnetization (Ms) 65 emu/g, coercivity (Hc) 77 Oe, the remanence (Mr) 3.4 emu/g and D of about 34 nm. This NFP has been used as radar absorbing materials in frequencies of 8 – 12 GHz (X-band) [8]. Based on specific laboratory equipment of the Institute for Tropicalizational Environment (VITTEP) that this investigation selects technological parameters: calcination temperature 900 ^0C for 4 h, heating speed 10 ^0C/minute and precursor concentration 0.1 M for synthesis $Ni_{0.7}Zn_{0.3}Fe_2O_4$ FNP.

2. EXPERIMENTAL

2.1. Synthesis of $Ni_{0.7}Zn_{0.3}Fe_2O_4$ FNP

The preparation process of $Ni_{0.7}Zn_{0.3}Fe_2O_4$ is shown in figure 1. The chemical reagents are high purity Nickel Chloride ($NiCl_2.6H_2O$ Merck, Japan), Ferric Chloride ($FeCl_3.6H_2O$ Merck, Germany), Zinc Chloride ($ZnCl_2$ Merck, Germany) and 25% Ammonium Hydroxide (NH_4OH Merck, Germany). Powders of $NiCl_2.6H_2O$, $FeCl_3.6H_2O$, $ZnCl_2$ were weighed based on the calculated formula and dissolved in double distilled water to the concentration 0.1 M with the corresponding volume ratio. Subsequently, these solutions were mixed, stirred and heated at 60 °C to that the powders were dissolved completely. Then the 25% NH_4OH is added to the above mixture drop by drop while stirring constantly. The addition of ammonia will stop when pH is maintained at 8.5.

The product obtained in the form of precipitates. The product was separated from water and washed several times in double distilled water to remove salt residues and other impurities. The product was dried at 110 °C for 12 h and it was further calcined at 900 °C for 4 h. Finally, the product was groud in millstone to obtain a fine powder.

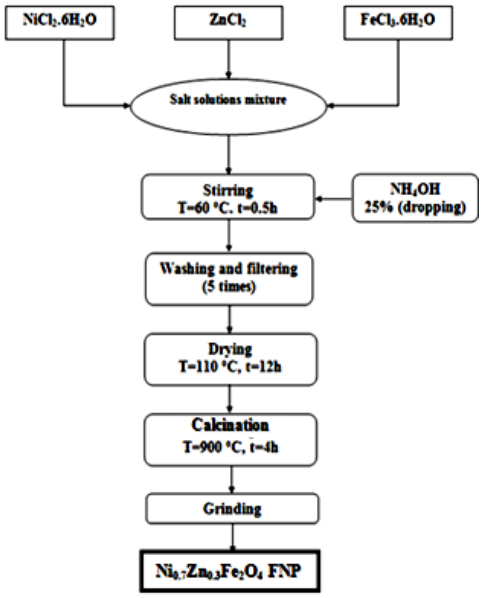


Figure 1. *The preparation process of* $Ni_{0.7}Zn_{0.3}Fe_2O_4$.

2.2. The methods for analyzing the structure and properties of FNP

The structure and phase of ferrite nano were analyzed by X-ray diffraction (XRD). The crystallite size and its distribution were calculated from the SEM image and Debye–Scherer equation:

$$D = 0.94 \times \lambda / \beta \times \cos\theta \quad (1)$$

D is the crystalline size in nm, λ is the Cu-Kα wavelength (0.154 nm), θ is the Bragg angle of diffraction at the peak with maxima intensity, and β is the full width at half of the diffraction peak with maxima intensity (FWHM) measured in radians**.**

The chemical component of FNP was analyzed by energy-dispersive X-ray spectroscopy (EDS). The magnetic properties were measured by a vibrating sample magnetometer (VSM).

3. RESULTS AND DISCUSSION

3.1. Crystal structure and chemical component

The XRD pattern (figure 2) was obtained with the 2θ range of 25–65° and the scanning speed of 0.05°/s. It shows that the peaks of six structure planes (220), (311), (400), (422), (511) and (400) which were shown in the detail in table 1.

Table 1. *The relationship of Miller index and interplanar spacing.*

Plane	Miller index			Interplanar spacing
	h	k	l	
(220)	2	2	0	2.95
(311)	3	1	1	2.51
(400)	4	0	0	2.08
(422)	4	2	2	1.70
(511)	5	1	1	1.60
(400)	4	4	0	1.47

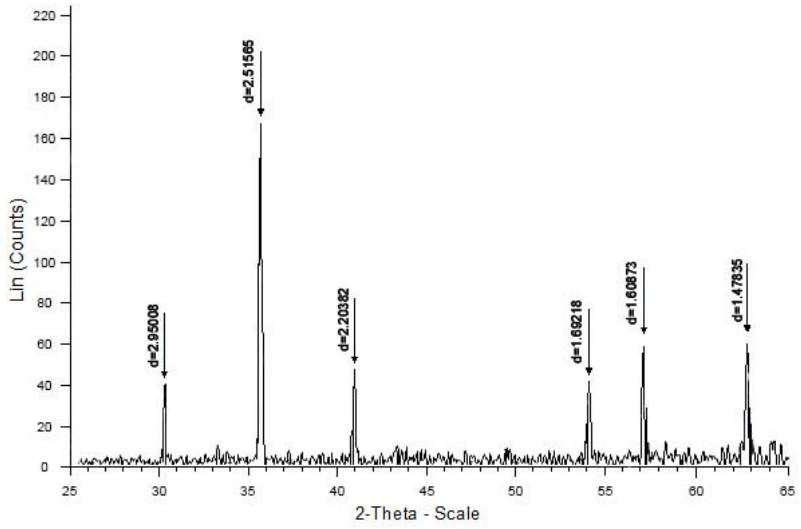


Figure 2. *XRD pattern of $Ni_{0.7}Zn_{0.3}Fe_2O_4$ FNP.*

The crystal structure of Ni-Zn FNP is a face-centered cubic, therefore, the interplanar spacing is calculated using the following equation:

$$d = \frac{a}{\sqrt{h^2 + k^2 + l^2}} \quad (2)$$

d is the interplanar spacing, a is the lattice parameter, and h, k, l are the Miller indexes.

The peaks of these planes are consistent with that of the $Ni_{0.7}Zn_{0.3}Fe_2O_4$ standard XRD pattern [8, 10]. Thus, it's planes of $Ni_{0.7}Zn_{0.3}Fe_2O_4$.

The element contents of ferrite nano were analyzed by EDS. The calculated results are in good agreement with the theoretical contents (figure 3 and table 2).

Table 2. *Element contents of FNP from EDS pattern and theory calculation.*

Element	Measured contentsby EDS, %	Theoretical contents, %
Ni K	17.05	17.44
Zn L	8.25	8.23
Fe K	47.26	47.30
O K	27.44	27.03

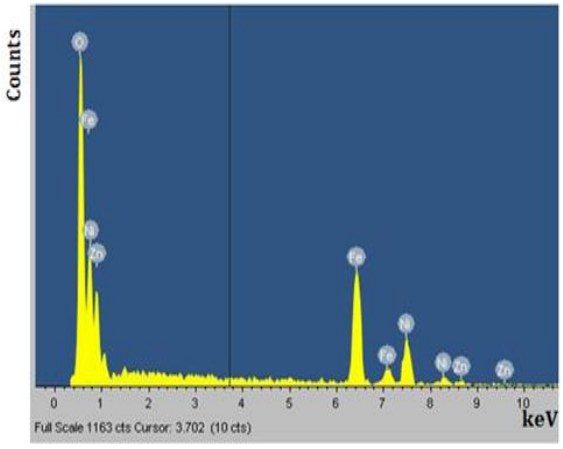


Figure 3. *EDS pattern of $Ni_{0.7}Zn_{0.3}Fe_2O_4$ FNP.*

From Figure 2, it can be seen that no other phase spectra appear, so the $Ni_{0.7}Zn_{0.3}Fe_2O_4$ ferrite is a single crystal.

3.2. Morphology and crystalline size

Figure 4 showed the SEM image of $Ni_{0.7}Zn_{0.3}Fe_2O_4$ FNP, which were spherical and the D of about 50 to 60 nm.

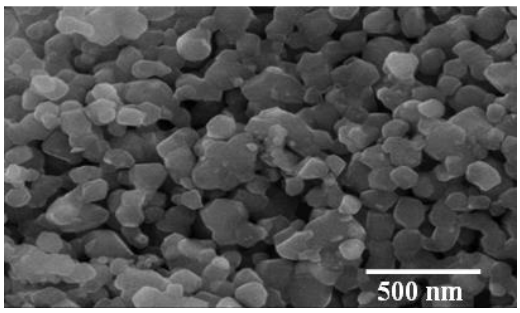


Figure.4. *SEM image of $Ni_{0.7}Zn_{0.3}Fe_2O_4$ FNP.*

The crystallite size was also calculated from the XRD pattern (figure 5) using (1).

The (311) plane has maxima intensity with d = 2.51 and 2θ = 35.64°, while β = 0.157° = 0.003 radian, λ = 0.154 nm. From (1), we have D = (0.94 x 0.154)/(0.003 x 0.952) = 50 nm. This result of D is consistent with the analyzing result from SEM image.

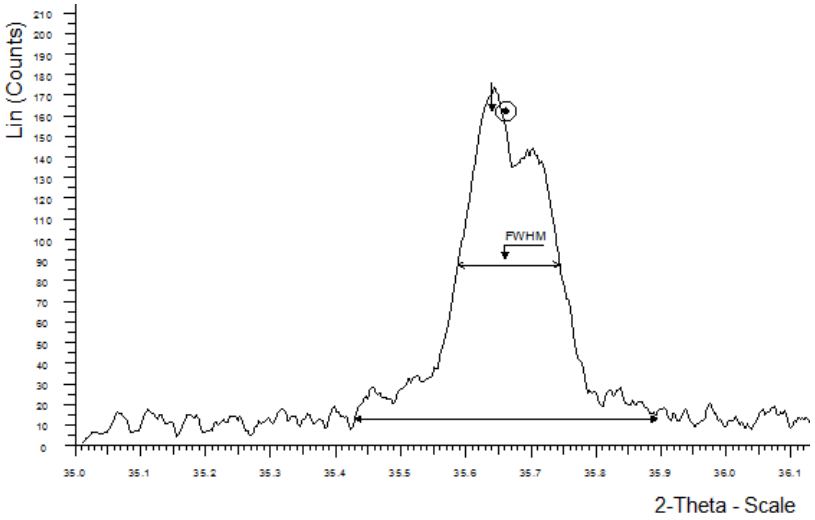


Figure.5. *The WFHM of $Ni_{0.7}Zn_{0.3}Fe_2O_4$ FNP.*

From Fig. 4, that the crystals are uniformly distributed and spherical shape. These ferrite nanoparticles are smaller than ferrite nanoparticles of the study [8], because of more long annealing time.

3.3. Magnetic properties

The magnetization properties of FNP were obtained at room temperature and showed in figure 6 and table 3: Ms = 57 emu/g; Mr = 10 emu/g; Hc = 114 Oe.

Table 3. *The magnetic properties of ferrite nano.*

Magnetic properties	Units	Values
Ms	emu/g	57
Hc	Oe	114
Mr	emu/g	10

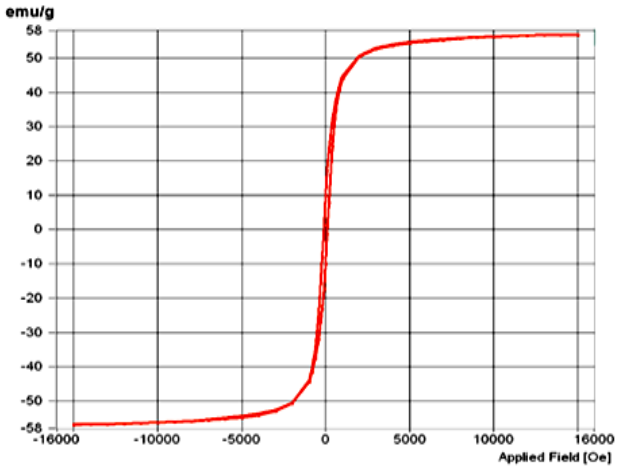


Figure.6. *Magnetization curve of $Ni_{0.7}Zn_{0.3}Fe_2O_4$ FNP.*

From Fig. 5-6, it looks that saturation magnetization (Ms) values is smaller than the study [8], because lower annealing temperature and non-standard annealing equipment, but it can use radar absorbing materials in frequencies of 8 – 12 GHz (X-band).

4. CONCLUSIONS

In this study, $Ni_{0.7}Zn_{0.3}Fe_2O_4$ FNP was synthesized successfully by solid-state reaction method. The properties of this material are equivalent to those in the study. The XRD pattern of $Ni_{0.7}Zn_{0.3}Fe_2O_4$ FNP was high crystal structure, SEM image and Debye Scherer equation were the D of ferrite nano that was 50 nm, the VSM measurements of magnetic properties have obtained Ms = 57 emu/g, Mr = 10 emu/g and Hc = 114 Oe. It can use radar absorbing materials in frequencies of 8 – 12 GHz (X-band).

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TÓM TẮT

CẤU TRÚC VÀ TÍNH CHẤT NANO FERIT $Ni_{0.7}Zn_{0.3}Fe_2O_4$ ĐƯỢC TỔNG HỢP BẰNG PHƯƠNG PHÁP PHẢN ỨNG PHA RẮN

Mục đích của nghiên cứu này là xác định cấu trúc và tính chất của nano ferit $Ni_{0.7}Zn_{0.3}Fe_2O_4$ (FNP) được tổng hợp bằng phương pháp phản ứng pha rắn trong điều kiện phòng thí nghiệm của đơn vị và so sánh chúng với các công bố thế giới. Cấu trúc ferrite được xác định bởi kỹ thuật nhiễu xạ tia X (XRD), kích thước hạt tinh thể (D) được xác định bằng kính hiển vi điện tử quét (SEM) và phương trình Dybye-Sherer, thành phần hóa học được xác định bằng phương pháp quang phổ tán xạ năng lượng tia X (EDS) các tính chất tính từ của ferite được xác định bằng từ kế mẫu rung (VSM). Kết quả đạt được của nghiên cứu FNP $Ni_{0.7}Zn_{0.3}Fe_2O_4$ là: Độ từ hóa bão hòa (M_s) 57 emu/g, lực kháng từ(H_c) 114 Oe, độ từ dư (M_r) 10 emu/g và kích thước hạt tinh thể 50 nm.

Từ khóa: Hạt nano ferit $Ni_{0.7}Zn_{0.3}Fe_2O_4$; Tính chất từ; Kích thước hạt tinh thể.

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