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Investigation of the Effects of Reaction Temperature in $NiFe_2O_4$ Nanoparticles Synthesis by Hydrothermal Method

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In this experimental study was investigated the effect of reaction temperature in $NiFe_2O_4$ nanoparticles synthesis with hydrothermal method. An appropriate ratio of solutions nickel nitrate and ferric nitrate were dissolved in deionized water and poured into a crucible. Later, polyethylene glycol 600 (PEG 600) was added to this mixture. Samples were adjusted to pH 11 values using NaOH solution. Accordingly, experiments were made at 180, 200 and 250 °C, respectively. The other parameters, were fixed as reaction time 24 h and pH value 11. The structural and morphological properties of $NiFe_2O_4$ nanoparticles were determined by X-ray powder diffraction (XRD) and Scanning Electron microscopy (SEM). Results showed that increasing calcination temperature contributed to cyristallinity of $NiFe_2O_4$ nano particles. But also average particle size increased. As a result, average particle size was calculated by using Debye-Scherrer Formula as approximately 30 nm. However, this results was confirmed with SEM and TEM analysis.

Keywords: Nanoparticles, Nickel ferrite, Hydrothermal.

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1. INTRODUCTION

In the last few decades, much attention has been paid on nanomaterials due to their fundamental and technological applications [1,2]. The physical and chemical properties of nanomaterials have been enhanced because of their surface-to-volume ratio [3]. Nickel ferrite ($NiFe_2O_4$) nanocrystalline is one of the most important ferrites among other ferrites because of most promising applications in storage devices, microwave devices, gas sensors, ferrofluids and catalysts [4–5]. Recently, considerable attention has been paid on $NiFe_2O_4$ with different morphology and their shape and size dependent properties as well corresponding applications were investigated [6,7].

Both the physical and chemical methods have been developed for synthesis of the $NiFe_2O_4$ nanostructure with various morphology. The chemical methods have advantages over the physical methods such as low cost, reaction taking place at room temperature and large scale production possibility. The $NiFe_2O_4$ nanocrystalline has been synthesised by various methods such as co-precipitation, sonochemical process, polymeric precursor techniques, mechanical alloying, sol–gel, pulsed wire discharge, shock wave, reverse micelles, hydrothermal and ultrasonically assisted hydrothermal processes [8–9].

$NiFe_2O_4$ exhibits unusual physical and chemical properties when its size is reduced into the nanoregion. The solid state reaction method has been conventionally used for the synthesis of nickel ferrites. There are some disadvantages of this technique, such as higher operating temperatures, inhomogeneity of the product, poor stoichiometry, and larger crystallite size. All of the listed qualities have a strong influence on its magnetic properties. To overcome these problems, wet chemical routes, such as sol-gel, combustion, and polyol synthesis are investigated for the synthesis of nanocrystalline oxide powders. Owing to the extremely small dimensions of nano-structured materials, a major portion of the atoms lie at the grain boundaries, which in turn is responsible for superior magnetic, dielectric, and mechanical properties in these materials compared to their conventional coarse grained counterparts [10].

Among the methods that have been used to prepare ferrite nanoparticles, [11−12] the hydrothermal method is one of the abundantly used method because it is economical and has a high degree of compositional control [13]. In addition, the hydrothermal synthesis route does not require extremely high processing temperatures.

The purpose of this experimental study is to determine whether the effect of reaction temperature in NiFe2O4 nanoparticles synthesis by hydrothermal method. The effects of reaction temperature on particle size and shape was investigated. Therefore, experiments were made at various reaction temperatures. Later, in samples obtained from this experiments were characterized by using X-Ray powder diffraction and Electron microscopies.

2. EXPERIMENTAL

Analytical grade nickel nitrate hexahydrate ($Ni(NO_3)_2.6H_2O$), ferric nitrate nonahydrate ($Fe(NO_3)_3.9H_2O$) and Poly ethylene glycol 600 (PEG 600) were purchased from Sigma- Aldrich and used without further purification. $NiFe_2O_4$ composite powders were prepared according to the hydrothermal method.

Experiments were made to determine the effect of temperature on reaction. Accordingly, experiments were made at 180, 200 and 250 °C, respectively. The other parameters, were fixed as reaction time 24 h and pH value 11. A mixed solution of nickel nitrate hexahydrate and ferric nitrate nonahydrate were prepared in deinozed water with vigorous stirring at room temperature. A specific volume of polyethylene glycol 600 (PEG 600) was added to the above mixed solution. After, the

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solution of NaOH was added until pH values reached 11. Reaction temperature is one of the main factors on which the final composition of the product depends, which can be varied to get the desired final product. The solution was vigorously stirred for 2 h to ensure the proper mixing of the components and the subsequently transferred into the Teflon lined stainless steel autoclave. The temperature of the autoclave was raised slowly up to desired temperature and kept the whole system at this temperature for 24 h. Once the reaction time was completed, the autoclave was cooled to room temperature and the resulting brown precipitate had been washed several times with ethanol and deionized water. The obtained precipitate was dried in a vacuum oven at 105 °C for 8 h. In the samples obtained from experiments was made X-ray powder diffraction analysis (XRD), Scanning Electron Microscopy (SEM) and Transmision Electron Microscopy analysis.

3. RESULTS

3.1 X-ray Analysis

The sample was characterized by X-ray diffraction (XRD) and scanning electron microscopy (SEM). Average particle size was calculated by using Debye-Scherrer Formula as approximately 30 nm. The XRD patterns for the samples obtained from experiments performed at various reaction temperatures were presented in Figs. 1-3. All the reflection peaks can be readily indexed to the Joint Committee on Powder Difraction Standards (JCPDS) powder diffraction data for $NiFe_2O_4$ (#10-0325).

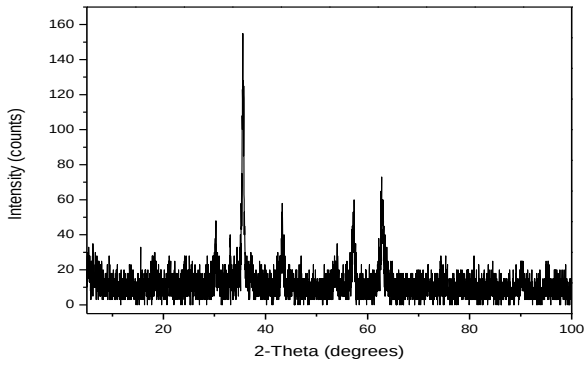

Fig. 1 – X-Ray Powder Diffraction pattern of sample prepared at 180 °C

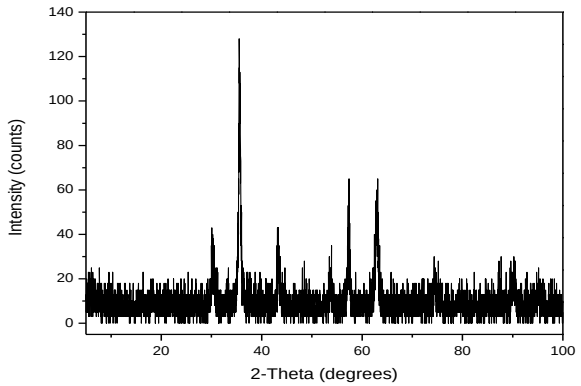

Fig. 2 – X-Ray Powder Diffraction pattern of sample prepared at 200 °C

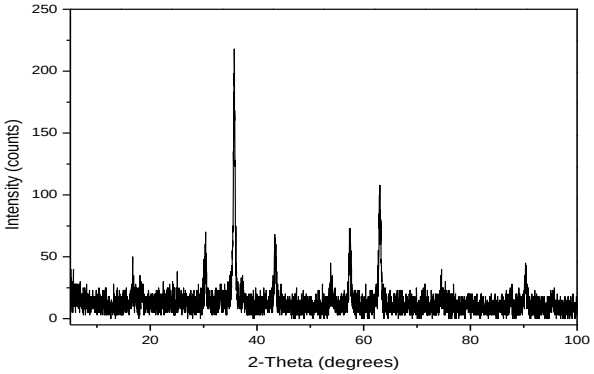

Fig. 3 – X-Ray Powder Diffraction pattern of sample prepared at 250 °C

3.2 SEM Analysis

SEM analysis in nanoparticles is made to determine to morphological properties and particle size of materials. Solid phase obtained from the first experiment were examined by Scanning Electron Microscopy (SEM) under appropriate conditions. SEM images of this example are shown in Figs. 4-5. Result of this analysis showed that examples was not washing well by ethanol and distilled water. Because, the around of particles was coated with a layer of hydrocarbon.

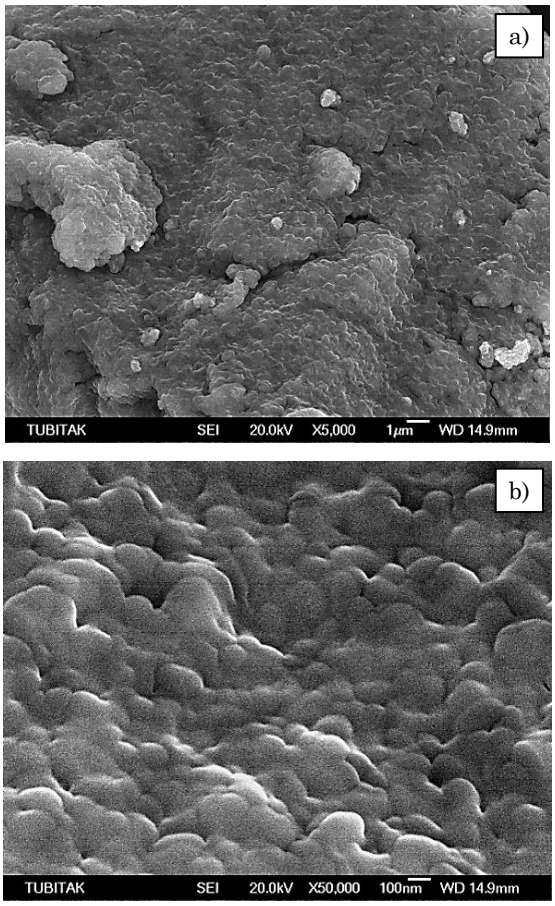

Fig. 4 – SEM imges of sample prepared at 200 °C

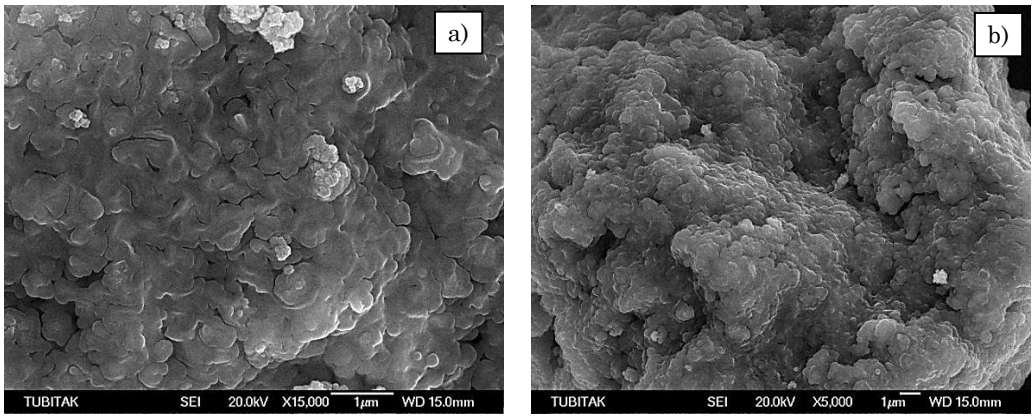

Fig. 5 – SEM images of sample prepared at 250 °C

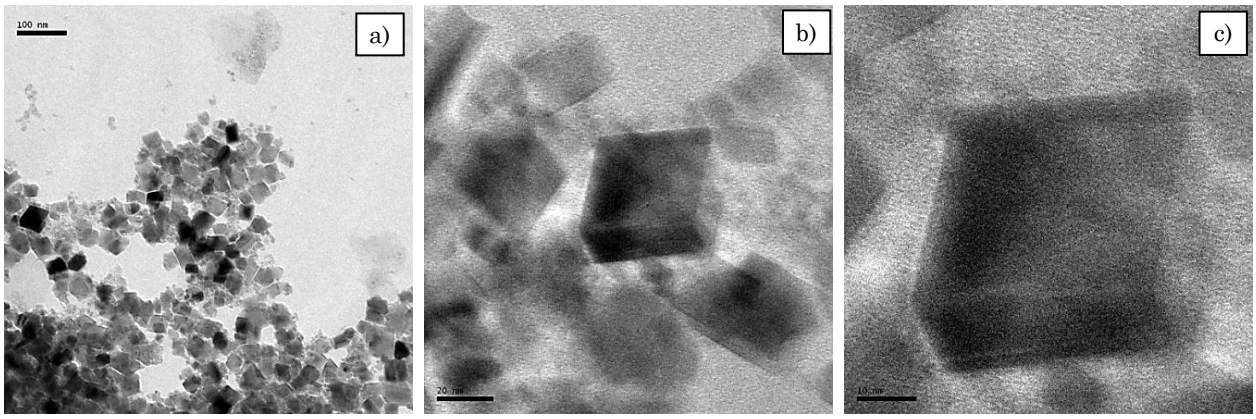

Fig. 6 – TEM imges of sample prepared at 200 °C

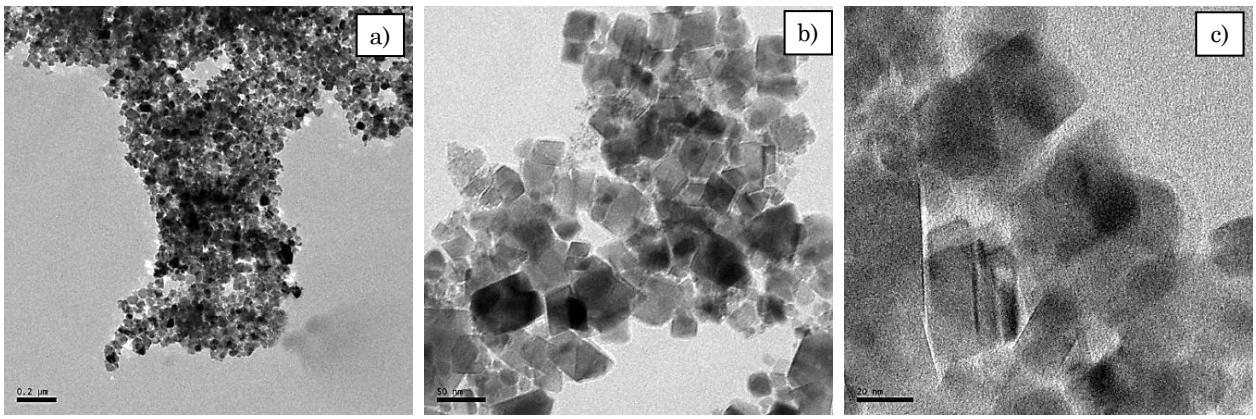

Fig. 7 – TEM images of sample prepared at 250 °C

3.3 TEM Analysis

In result wasn't calculated to average particle size from SEM images of the samples. Because around of the particles covered completely with a layer of hydrocarbon. Thus, TEM analysis was performed to verified the particle size calculated by the Scherrer formula. Accordingly, TEM images of samples of the particles obtained from this experiments were shown in Fig. 6 for 200 °C and in Fig. 7 for 250 °C. According to TEM images, the sizes of the particles is nanoscale. However, these particles have a quite regular crystalline structure.

4. CONCLUSION

As result, Reaction temperature is not effective in nano size $NiFe_2O_4$ particles synthesis by hydrothermal method. In result wasn't calculated to average particle size from SEM images of the samples. Because arounds of the particles covered completely with a layer of hydrocarbon. Accordingly, Examples obtained from this experiments should be washed with or organic solvents or this organic phase should be removed by burning at high temperature. TEM analysis was performed to verified the particle size calculated by the Scherrer formula. TEM images showed that samples obtained from all experiments is nanoscale. However, average particles size values of this particles are very close together. The results confirm the value calculated by the Scherrer formula.

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