

## The Effect of Calcination Temperature in NiFe<sub>2</sub>O<sub>4</sub> Nanoparticles Synthesis with Microwave Combustion Method

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Magnetic ferrites are a group of technologically important magnetic materials. Synthesis of nanocrystalline spinel ferrite has been investigated intensively in recent years due to their potential applications in high-density magnetic recording, microwave devices, and magnetic fluids. In this study, NiFe<sub>2</sub>O<sub>4</sub> nanoparticles were prepared with microwave combustion methods. In experiments, samples obtained by microwave method were calcined at various temperatures. The structural and morphological properties of NiFe<sub>2</sub>O<sub>4</sub> nanoparticles were determined by X-ray powder diffraction (XRD) and Scanning Electron microscopy (SEM). Results showed that increasing calcination temperature contributed to crystallinity of NiFe<sub>2</sub>O<sub>4</sub> nanoparticles. But also average particle size increased. As a result, average particle size calculated by using Debye-Scherrer Formula as approximately 30 nm. However, this results was confirmed with SEM analysis.

**Keywords:** Nanoparticles, Nickel ferrite, Microwave, Calcination.

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### 1. INTRODUCTION

Spinel ferrites, MFe<sub>2</sub>O<sub>4</sub>, are technologically important group of materials due to their enhanced optical, magnetic, and electrical properties. These properties make them very attractive for a variety of applications including but not limited to use as electrodes in energy storage devices, as catalysts, in magnetic storage devices, etc. [1-2-3].

Spinel ferrites have the general formula of AFe<sub>2</sub>O<sub>4</sub> (where A<sup>2+</sup>: Fe, Co, Ni, Zn, etc.) and unit cell contains 32 oxygen atoms in cubic close packing with 8 tetrahedral (Td) and 16 octahedral (Oh) occupied sites. By changing type of the divalent cation, it is possible to obtain significantly different physical and magnetic properties in these ferrites [3].

Magnetic ferrites are a group of technologically important magnetic materials. Synthesis of nanocrystalline spinel ferrite has been investigated intensively in recent years due to their potential applications in high-density magnetic recording, microwave devices, and magnetic fluids [4-5]. Nickel ferrite (NiFe<sub>2</sub>O<sub>4</sub>) is one of the most important spinel ferrites as well as a typical spin soft-magnetic ferrite. It has an inverse spinel structure showing ferrimagnetism that originates from magnetic moment of anti-parallel spins between Fe<sup>3+</sup> ions at tetrahedral sites and Ni<sup>2+</sup> ions at octahedral sites [6]. Recently, various methods have been developed to synthesize nanocrystalline NiFe<sub>2</sub>O<sub>4</sub> such as mechanical alloying [7], pulsed wire discharge [6], sol-gel method [8], microemulsion [9], hydrothermal-microwave [10] and hydrothermal processes [11]. Among these established methods, hydrothermal synthesis has attracted great interest because it offers many advantages, including the enhancement of solubility, diffusion, and crystallization as well as the control of the morphologies, sizes and phase transformation, etc. [12-13].

The purpose of this experimental study is to determine whether the effect of calcination in NiFe<sub>2</sub>O<sub>4</sub> nanoparticles synthesis by microwave combustion method. The effects of calcination on particle size and shape were investigated. Therefore, experiments were made at various calcination 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<sub>3</sub>)<sub>2</sub>·6H<sub>2</sub>O), ferric nitrate nonahydrate (Fe(NO<sub>3</sub>)<sub>3</sub>·9H<sub>2</sub>O) and urea (CO(NH<sub>2</sub>)<sub>2</sub>) were purchased from Merck. An appropriate ratio of nickel nitrate, ferric nitrate and urea—to serve as fuel, were dissolved in deionized water and poured into a crucible, which was then placed in a kitchen-type microwave oven at a maximum power. The solution initially boils then under goes dehydration followed by decomposition with the evolution of large amount of gas. After the solution reaches the point of spontaneous combustion, it begins to burn by releasing lots of heat, vaporizes all the solution instantly and becomes a solid.

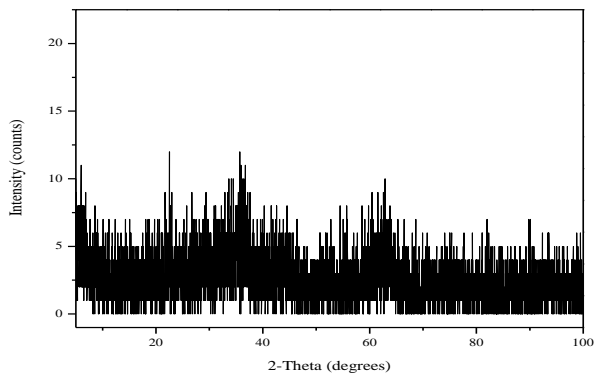
### 3. RESULTS

#### 3.1 X-ray Analysis

The XRD pattern for the solid obtained from experiment was presented in Fig. 1. The XRD patterns for the samples obtained from experiments performed at various calcination temperatures were presented in Fig. 2.

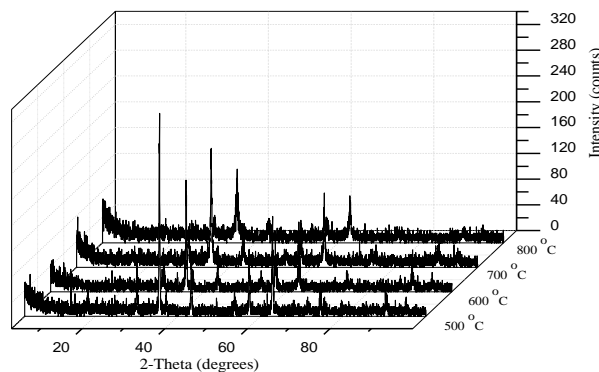
Fig. 1. shows the powder XRD pattern of this sample. According to this XRD pattern, nickel ferrite formations was not completed. Thus, the samples were calcined in high temperatures. Experiments were made

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**Fig. 1** – X-Ray diffraction pattern of NiFe<sub>2</sub>O<sub>4</sub> nanoparticles

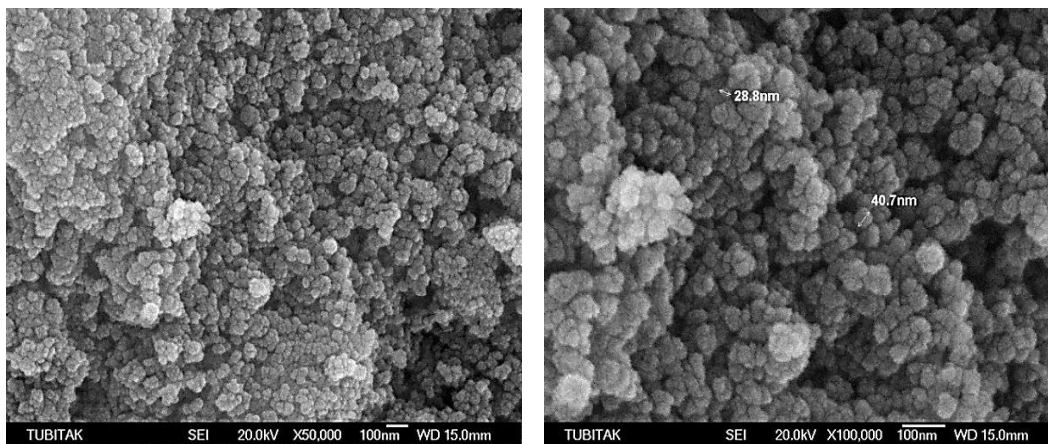
at 500, 600,700, 800 °C, respectively. In these experiments, the calcination time was fixed at 8h. In the samples obtained from these experiments, XRD analysis was performed. Fig. 2. shows the powder XRD patterns of the samples prepared at various temperatures. All the reflection peaks can be readily indexed to the Joint Committee on Powder Diffraction Standards (JCPDS) powder diffraction data for NiFe<sub>2</sub>O<sub>4</sub> (#10-0325). However, the reflection peaks become sharper and narrower along with increasing calcination temperature, indicating the improvement of crystallinity. In the end of this experiments, the nickel ferrite nanoparticles are obtained as the only product. No secondary phase was detected in XRD and electron microscopy analysis.



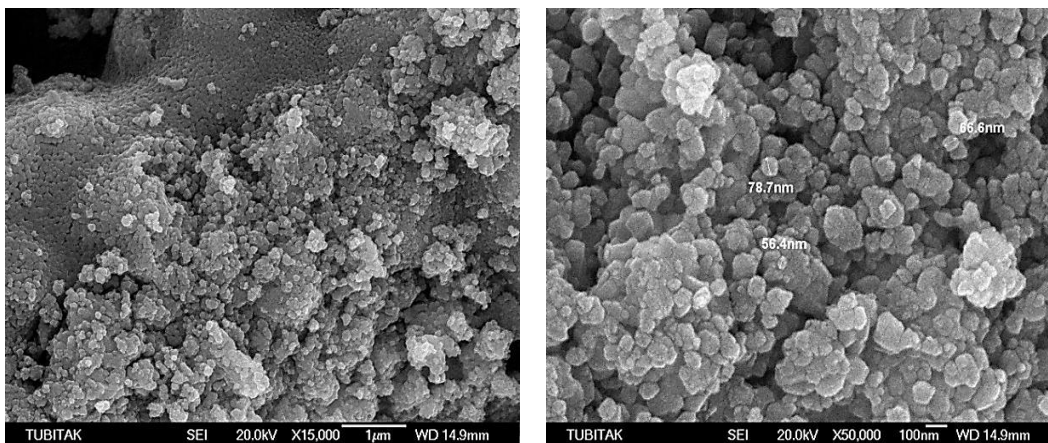
**Fig. 2** – Powder X-ray diffraction (XRD) patterns of various NiFe<sub>2</sub>O<sub>4</sub> nanoparticles synthesized under different temperature

**3.2 SEM Analysis**

The morphologies, microstructures and particle sizes of the as-prepared samples were determined by SEM. Fig. 3-6 shows the SEM images of samples. This figure reveals remarkable changes in the microstructure, regarding grain size, porosity and the particle distribution of the as-prepared solids by changing the heat treatment conditions depending . Individual particle size increases to about 50 nm was attesting a better crystallinity of the spinel phase. It is recognized that the extremely fine powders show a strong tendency to formation of aggregates and/or agglomerates.



**Fig. 3** – SEM images of sample calcined at 500 °C for 8 h



**Fig. 4** – SEM images of sample calcined at 600 °C for 8 h

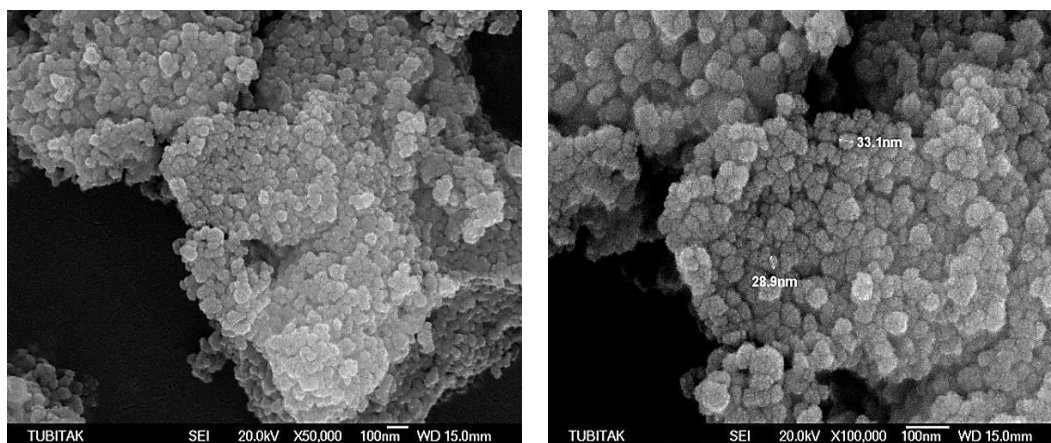


Fig. 5 – SEM images of sample calcined at 700 °C for 8 h

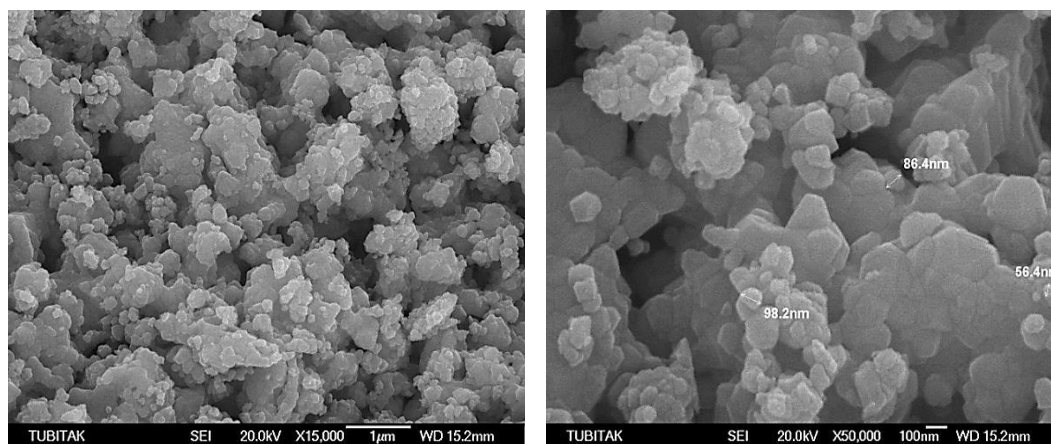


Fig. 6 – SEM images of sample calcined at 800 °C for 8 h

In SEM examinations of these samples was observed that the melt in samples with increasing temperature. As a result, the average particle size of the particles is an increasing trend. Therefore, temperature of calcination must be well controlled for an optimal particle size distribution and to obtain a regular crystal structure. A secondary phase in SEM images of this samples wasn't observed. It is understood that the form is completed the conversion of the reaction. It is understood that reaction has been completed and full conversion was achieved. Fig. 3-6 shows SEM photographs of this samples. It is seen from this figure that the particles are spherical in shape with weak agglomeration. Moreover, there is homogeneous and uniform distribution of these particles in the powder samples.

#### 4. CONCLUSION

In this experimental study, in synthesis of NiFe<sub>2</sub>O<sub>4</sub> nano-particles by microwave method on average particle size and crystal structure was investigated to effects of calcination process and calcination tempera-

tures. According to XRD pattern given in Fig. 1, nickel ferrite formations was not completed in microwave process. Thus, samples were heat treated at various temperatures for 8 h to enhance their crystallinity and remove the residual charred organic materials. Results showed that increasing calcination temperature contributed to crystallinity of NiFe<sub>2</sub>O<sub>4</sub> nanoparticles. But also average particle size increased. In addition, in samples were observed agglomeration at high temperatures. However, conditions of experiment at high temperatures. However, conditions of experiment made at 700 °C value was accepted as optimum conditions for synthesis of NiFe<sub>2</sub>O<sub>4</sub> nanoparticles by microwave method. Under these conditions, the average particle size and crystal structure was confirmed by XRD and SEM-analysis.

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