



## The Effects of Fuel Type Above Adsorptive Properties of the Nickel Ferrite Nanoparticles synthesized with Microwave Method

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In this study, we were able to develop a new and practical method for the synthesis of the NiFe<sub>2</sub>O<sub>4</sub> nanoparticles. The synthesis of nickel ferrite nanoparticles was used various fuel substances such as glycine, urea and citric acid. The synthesis mixture prepared in stoichiometric rates was put in to the kitchen type microwave oven. In the end of reaction time was obtained a brown-black solid. The obtained solid was characterized with X-Ray Powder Diffraction and Scanning Electron Microscopy. The results of this analysis showed that all of the obtained particles has got nano-size particle size distribution. Later, the nanoparticles were analyzed by using a surface area analyzer and their adsorptive properties were investigated such as surface area and average pore size. We observed that the nanoparticles prepared with urea has the highest surface area. However, fuel type used in synthesis is quite effective on the surface properties of NiFe<sub>2</sub>O<sub>4</sub> nanoparticles.

**Keywords:** Nickel ferrite, Nanoparticles, Surface area, Average pore width, Total pore volume.

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### 1. INTRODUCTION

Nanotechnology is area of intense scientific research, due to a wide variety of potential applications in biomedical, optical, and electronic fields. Nanoparticles are of great scientific interest as they are effectively a bridge between bulk materials and atomic or molecular structures.

The properties of materials change as their size approaches the nanoscale and as the percentage of atoms at the surface of a material becomes significant. For bulk materials larger than one micrometre the percentage of atoms at the surface is minuscule relative to the total number of atoms of the material.

Synthesis and applications of nanoparticles of spinel ferrite structured is one of the most important research subjects due to their remarkable chemical and physical possessions like magnetic properties in recent years. The nanoparticles have been used in many applications, including ferrofluids, catalysts, microwave devices, gas sensors, and magnetic materials. The type of nickel ferrite with spinel structure is an essential composite material for the production of electronic and magnetic components that are extensively used in advanced technological applications to some remarkable of them are magnetic ferrofluids, dense information storage system and high frequency systems [1, 2].

Various chemical and physical techniques have been developed for the synthesis of NiFe<sub>2</sub>O<sub>4</sub> nanoparticles such as sol-gel, hydrothermal, co-precipitation, aerosol, microwave, etc. that have the advantages for instance the low cost, low reaction time, large scale production. Common problems for many of these techniques are factors such as complex processes, expensive precursors and low production rates [3, 4].

Alike the techniques, the combustion method is a promising chemical method for preparation of various

nano-sized ferrites in laboratory and semi-pilot scale that is fast and safe furthermore enables the formation of nano-sized metal oxide particles and allows to produce the ferrites without need of sophisticated equipments. Organic compounds like glycine, urea, citric acid, alanine etc. are used as fuels in the combustion method, suitable that they can be mixed directly with the metal salts to enhance the efficiency of the synthesis. Among the organic compounds, urea seems to be the most commonly used compound that it appropriately is cheap, readily available, non toxic and safe. The aim of this study is to investigate the effect of fuel type above adsorptive properties of NiFe<sub>2</sub>O<sub>4</sub> nanoparticles synthesized with microwave assisted combustion method [5, 6].

### 2. EXPERIMENTAL

#### 2.1 Synthesis

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 fuel substances were purchased from Sigma- Aldrich. Nickel nitrate, ferric nitrate and fuel substances were mixed in stoichiometric ratios, instantly the mixture was formed due to hygroscopicity of metal nitrates which absorbs the moist from the air therefore this method does not require use of water or any solvent. The composition of the synthesis mixture was given in Table 1. Then, the mixture was placed in a laboratory-type microwave oven at a maximum power of 800 W for 10 min. When the solution reached the point of spontaneous combustion, it started burning by releasing a very dense gas and heat, than the sample instantaneously becomes a solid.

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## 2.2 The Measurements of the Surface Area of Nanoparticles

A sample contained in an evacuated sample tube is cooled (typically) to cryogenic temperature, and then is exposed to an analysis gas at a series of precisely controlled pressures. With each incremental pressure increase, the number of gas molecules adsorbed on the surface increases. The equilibrated pressure ( $P$ ) is compared to the saturation pressure ( $P_0$ ) and their relative pressure ratio ( $P/P_0$ ) is recorded along with the quantity of gas adsorbed by the sample at each equilibrated pressure.

**Table 1** – The composition of the synthesis mixture

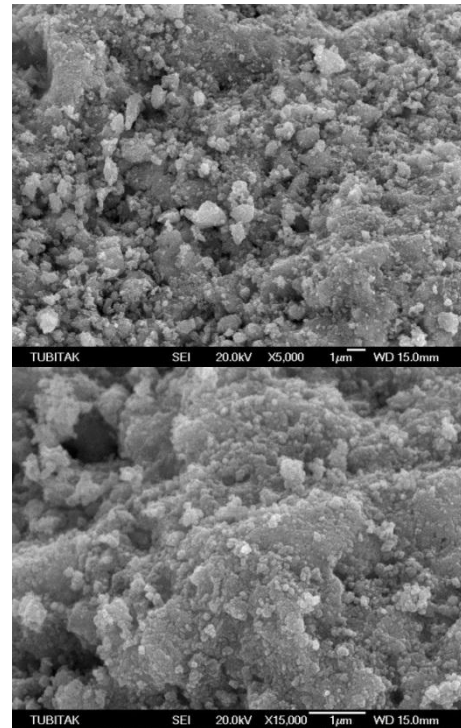
The Sample	Composition of the synthesis mixture
NFU	(Ni(NO <sub>3</sub> ) <sub>2</sub> .6H <sub>2</sub> O, Fe(NO <sub>3</sub> ) <sub>3</sub> .9H <sub>2</sub> O), Urea
NFC	(Ni(NO <sub>3</sub> ) <sub>2</sub> .6H <sub>2</sub> O, (Fe(NO <sub>3</sub> ) <sub>3</sub> .9H <sub>2</sub> O), Citric Acid
NFG	(Ni(NO <sub>3</sub> ) <sub>2</sub> .6H <sub>2</sub> O, (Fe(NO <sub>3</sub> ) <sub>3</sub> .9H <sub>2</sub> O), Glycine

As adsorption proceeds, the thickness of the adsorbed film increases. Any micropores in the surface are filled first, then the free surface becomes completely covered, and finally the larger pores are filled by capillary condensation. The process may continue to the point of bulk condensation of the analysis gas. Then, the desorption process may begin in which pressure systematically is reduced resulting in liberation of the adsorbed molecules. As with the adsorption process, the changing quantity of gas on the solid surface at each decreasing equilibrium pressure is quantified. These two sets of data describe the adsorption and desorption isotherms. Analysis of the shape of the isotherms yields information about the surface and internal pore characteristics of the material.

## 3. RESULTS

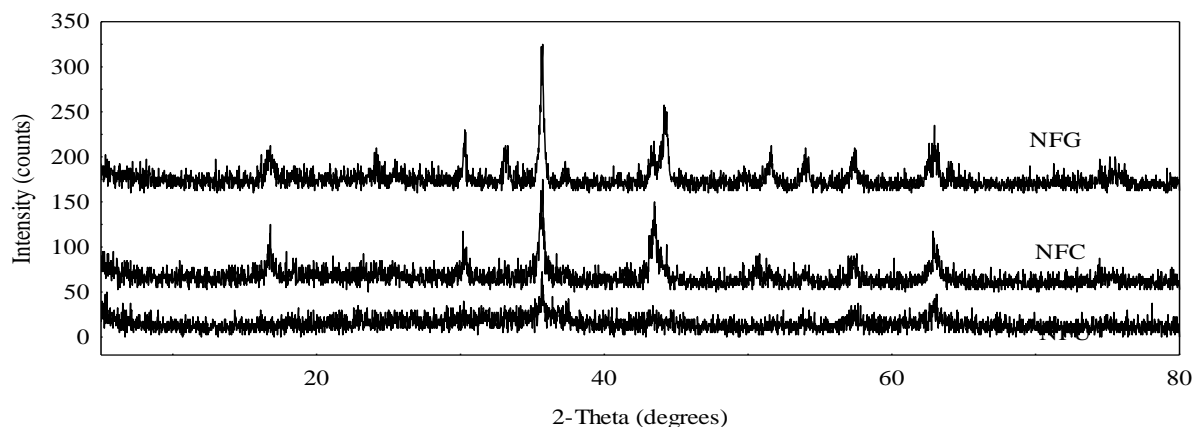
The NiFe<sub>2</sub>O<sub>4</sub> nanoparticles were prepared with microwave assisted combustion method. In this method, three different chemical substances were used as fuel. The

used fuels are urea, glycine and citric acid. The prepared nanoparticles were characterized with SEM and XRD analysis. X-Ray diffraction patterns of the obtained particles were given in Fig 1. The SEM images of the NiFe<sub>2</sub>O<sub>4</sub> synthesized by using urea as fuel were given in Fig. 1.

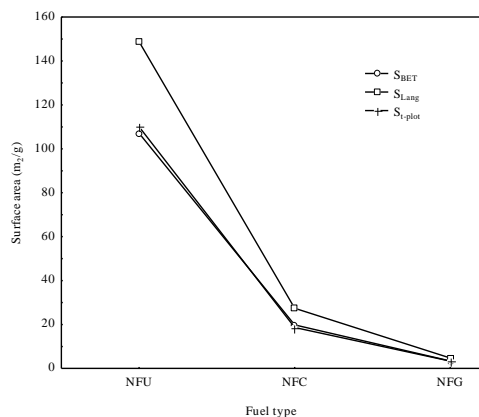


**Fig. 1** – The SEM images of the NiFe<sub>2</sub>O<sub>4</sub> synthesized by using urea as fuel

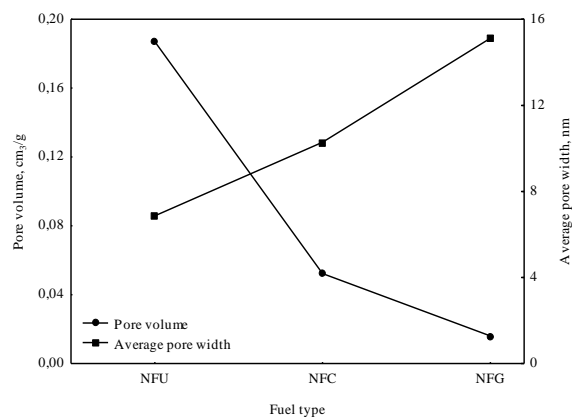
To determine the surface properties of the nanoparticles, the all of the obtained nanoparticles were analyzed by using a Micromeritics Gemini VI surface area analyzer. The various surface properties of the nanoparticles such as, average pore with, BET surface area and total pore volume were given in Fig. 1. and Fig. 2.



**Fig. 2** – X-Ray diffraction patterns of the nanoparticles



**Fig. 3** – The effect of fuel type on the surface area of the nanoparticles



**Fig. 4** – The effect of fuel type on total por volume and average pore width of the nanoparticles

#### 4. CONCLUSION

NiFe<sub>2</sub>O<sub>4</sub> nanoparticles can synthesis with various methods such as combustion, sol gel, hydrothermal et. In this study, NiFe<sub>2</sub>O<sub>4</sub> nanoparticles were synthesized with microwave assisted combustion method. In this method; the some substances have to be used as fuel. The aim of this work was to investigate the effect of fuel type above surface properties of NiFe<sub>2</sub>O<sub>4</sub> nanoparticles synthesized with microwave

method. We observed that fuel type is quite effective on surface properties of the particles. The nanoparticles synthesized by using urea as fuel have got the highest surface area. But they have quite poor cyristallinity. On the other hand, the nanoparticles synthesized by using glycine and citric acid have got more than better cyristallinty. But their surface areas are smaller than the particles produced with urea.

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