

# Preparation and Characterization of $\text{CuFe}_2\text{O}_4$ Nanoparticles by the Sol-Gel Method and Investigation of its Microwave Absorption Properties at Ku-band Frequency using Silicone Rubber

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Received: 22 April 2018; Accepted: 26 April 2018; Published: 14 May 2018

**Abstract:** Recently, using microwave devices emitting electromagnetic waves due to enhancing convenient life have been increased that can be harmful to the environment. In this study,  $\text{CuFe}_2\text{O}_4$  nanoparticles were prepared through the conventional sol-gel procedure and then were characterized by X-ray powder diffraction (XRD), vibrating sample magnetometer (VSM), field emission scanning electron microscopy (FE-SEM), Fourier transform infrared (FT-IR), and vector network analyzer (VNA) using S parameters. Results illustrated that pure crystal structure of magnetic nanoparticles has been synthesized by the sol-gel method with magnetic saturation ( $M_s$ ) of 22 emu/g. Finally,  $\text{CuFe}_2\text{O}_4$  nanoparticles were composited by silicone rubber to investigate of its microwave absorption properties. Results showed that  $\text{CuFe}_2\text{O}_4$ /silicone rubber nanocomposite absorbed more than 94.87% of microwave irradiation at ku-band frequency with 1.7 mm thickness and the maximum reflection loss was -60.38 dB at 16.1 GHz. Magnetic and dielectric properties of the  $\text{CuFe}_2\text{O}_4$  nanoparticles and silicone rubber polymeric matrix in the nanocomposite demonstrated desirable microwave absorption properties.

**Keywords:**  $\text{CuFe}_2\text{O}_4$ ; microwave absorption; sol-gel; nanoparticles; silicone rubber

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## 1. Introduction

The  $\text{CuFe}_2\text{O}_4$  nanoparticles have attracted substantial attention because of their various applications in photocatalysts, sensors, water treatment catalysts, air purification systems, and etc. [1,2]. Microemulsion, thermal decomposition, aerosol, co-precipitation, mechanochemical, and hydrothermal methods have been used to synthesis of spinel nanoparticles [3–7]. In the last decade, exploration in the microwave absorption capability of materials has been widely investigated due to various applications of microwave absorbing materials in the electric industries. The impedance matching, permittivity, and permeability are the most important factors affecting on the microwave attenuation given by transmission line theory [8–10]. To improve impedance matching, various chemical and physical methods have been employed to merge of magnetic and dielectric features of the components led to more microwave absorption [7,11,12]. The  $\text{CuFe}_2\text{O}_4$  spinel structure is the potential candidate for microwave devices because it has shown acceptable magnetic and dielectric properties at high frequency [13,14]. In this research  $\text{CuFe}_2\text{O}_4$  nanoparticles were prepared using sol-gel method. Subsequently, microwave absorption properties of the nanoparticles were investigated using silicone rubber medium.

## 2. Experimental

### 2.1. Materials and Instruments

$\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$ , iron (III) nitrate nonahydrate, citric acid, and ammonia solution were purchased from Merck. To prepare the microwave absorbing nanocomposite, silicone rubber was obtained from ELASTOSIL® M4503. Wacker RTV-2.

The chemical functional groups were revealed by Shimadzu 8400 S FT-IR. SEM images of the nanoparticles were obtained using Tescan Mira2. IRI Kashan VSM presents the magnetic properties of sample. The crystal structure of nanoparticles was explored by Philips X'Pert MPD instrument. Finally, Agilent technologies, E8364A exhibited microwave absorption properties.

### 2.2. Synthesis of $\text{CuFe}_2\text{O}_4$ Nanoparticles

To prepare of  $\text{CuFe}_2\text{O}_4$  nanoparticles by the sol-gel method, stoichiometric amount of nitrate salts and citric acid were dissolved in distilled water. Then, alkaline pH was adjusted by ammonia solution. After that, a wet gel was obtained by maintaining the solution at 90 °C and then the gel was calcined at 750 and 850 °C for 4 h to compare the results.

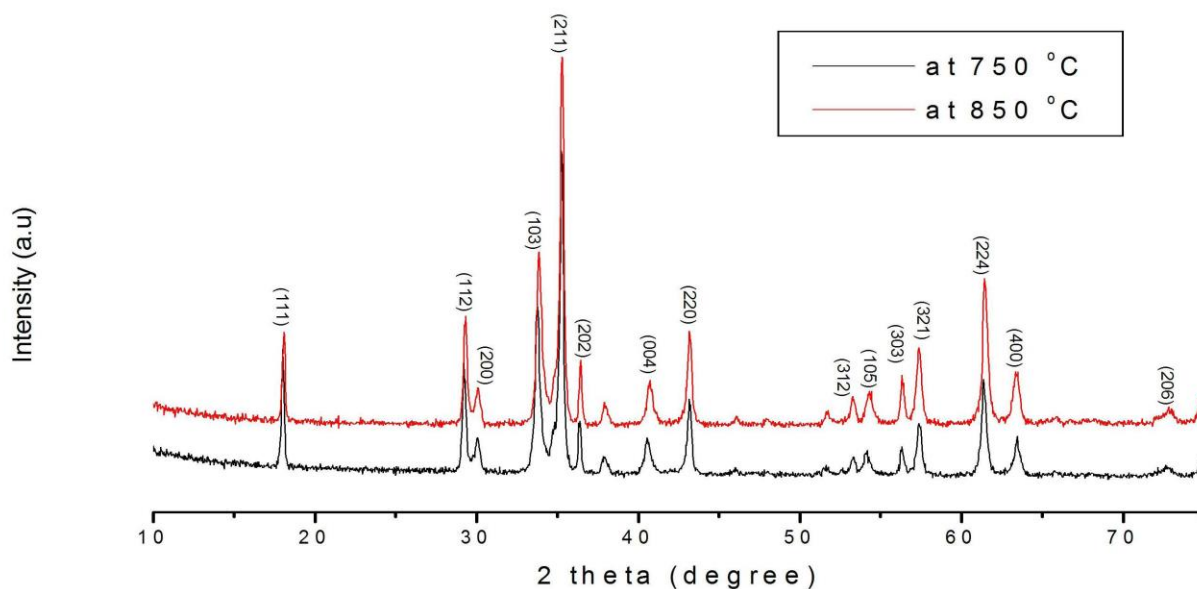
### 2.3. Preparation of Microwave Absorbing Composite

The  $\text{CuFe}_2\text{O}_4$  nanoparticles were added to silicone resin and then was molded to investigate microwave absorption properties using hardener with 20 Wt.%.

## 3. Results and Discussions

### 3.1. Phase Identification Analysis

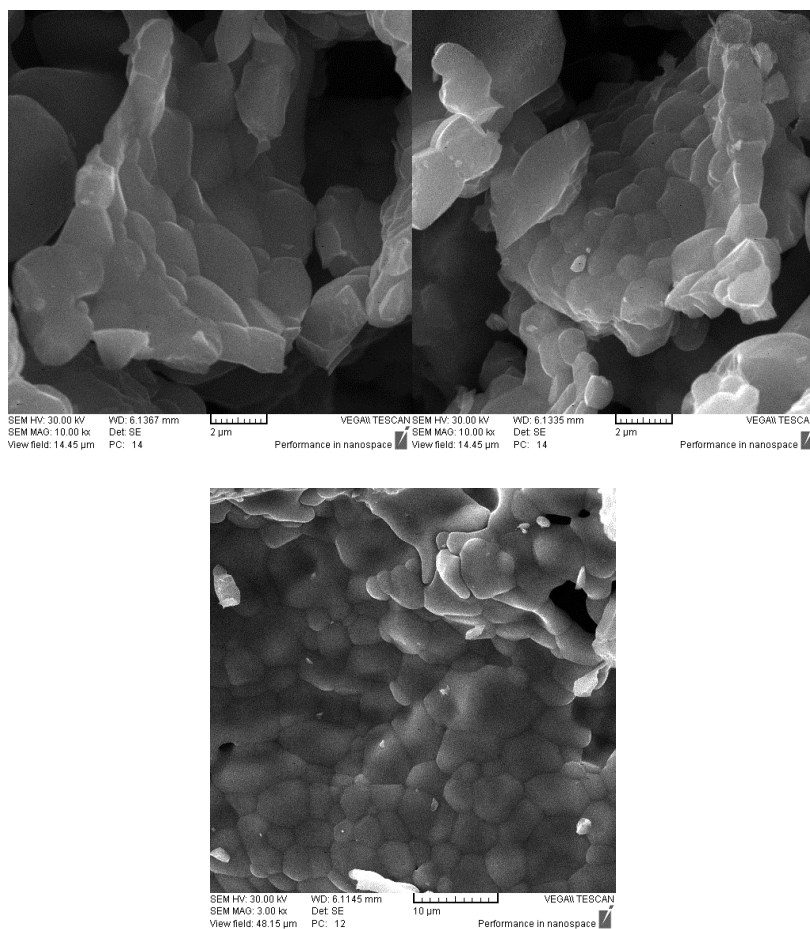
The XRD patterns of  $\text{CuFe}_2\text{O}_4$  nanoparticles calcined at 750 °C and 850 °C have been shown in Figure 1. As shown in the patterns, all angels of the crystal planes are corresponded to the [034-0425] standard cart with any impurities of crystal phase. The crystal size of nanoparticles calculated 15.6 nm based on the Scherrer equation.



**Figure 1.** XRD patterns of  $\text{CuFe}_2\text{O}_4$  nanoparticles calcined at 750 °C and 850 °C.

### 3.2. Morphology

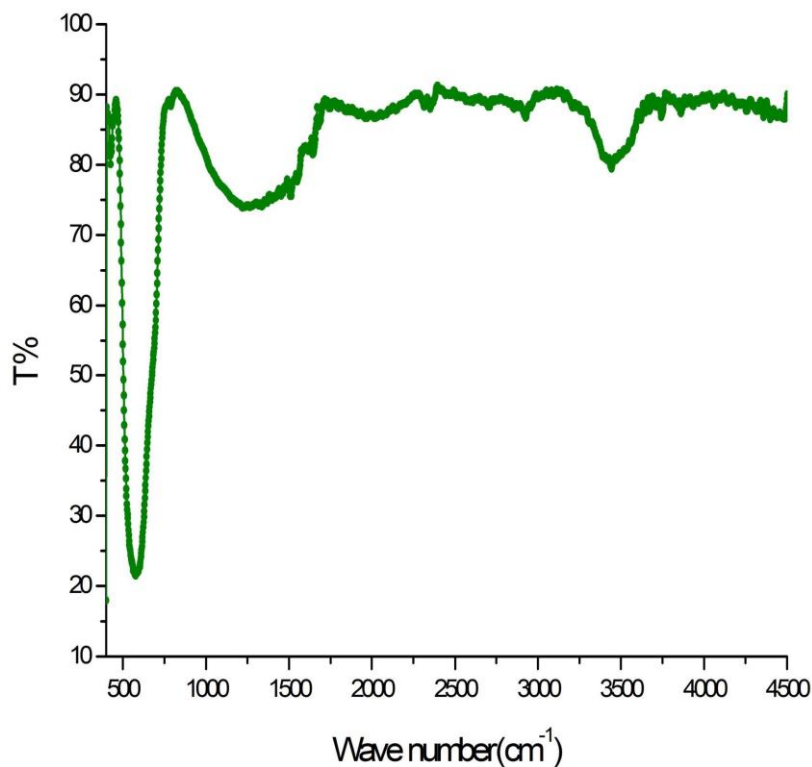
The morphology of  $\text{CuFe}_2\text{O}_4$  nanostructures prepared at  $850\text{ }^\circ\text{C}$  was investigated by SEM analysis. SEM micrographs confirmed that the structure, size, and shape of  $\text{CuFe}_2\text{O}_4$  nanoparticles were same (Figure 2).



**Figure 2.** FE-SEM images of  $\text{CuFe}_2\text{O}_4$  nanoparticles calcined at  $850\text{ }^\circ\text{C}$ .

### 3.3. FT-IR Spectroscopy

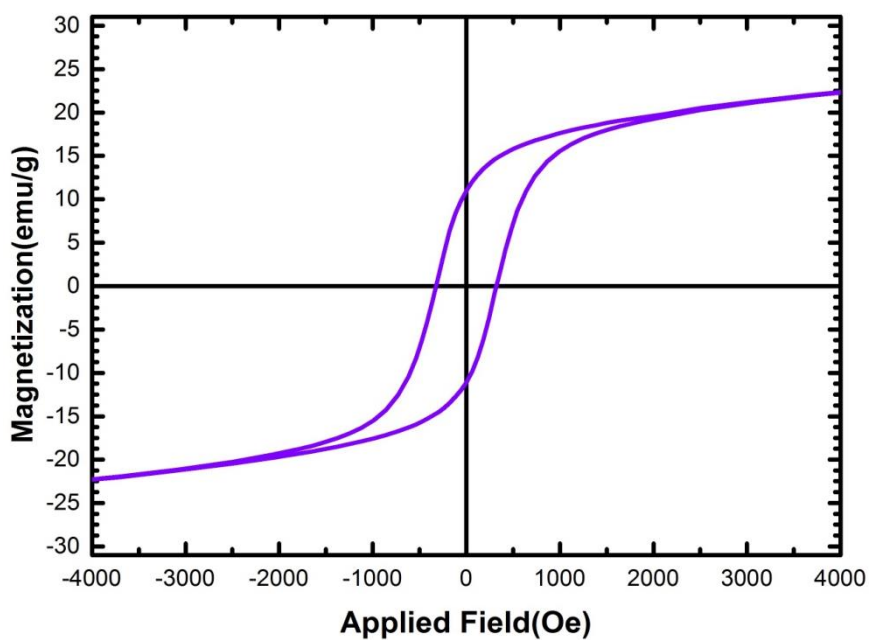
FT-IR was used to determine the structure and investigation of the chemical species. According to the result showed in Figure 3, broad band peaks at  $417.74$ ,  $577.38$ , and  $1200\text{cm}^{-1}$  are related to metal-oxide stretching vibrations of the octahedral, tetrahedral, and hexagonal sites in crystalline structures, respectively. The peak at  $1639.12\text{ cm}^{-1}$  and broad band peak at  $3440.80\text{ cm}^{-1}$  are assigned to the bending and stretching vibration of O–H related to the adsorbed water.



**Figure 3.** FT-IR spectrum of  $\text{CuFe}_2\text{O}_4$  nanoparticles calcined at  $850\text{ }^\circ\text{C}$ .

#### 3.4. Magnetic Properties

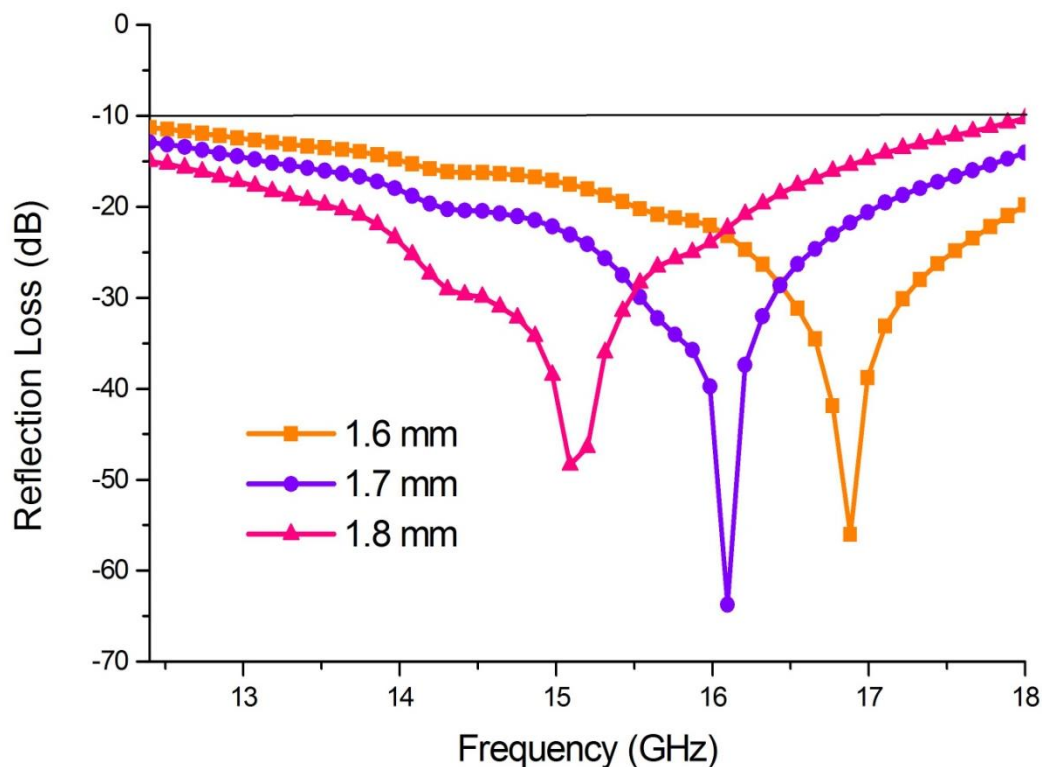
The magnetic properties obtained in room temperature using VSM instrument operating with frequency of 25 Hz and  $-4 < \text{kOe} < 4$  applied field. Figure 4 shows hysteresis loop of  $\text{CuFe}_2\text{O}_4$  nanoparticles calcined at  $850\text{ }^\circ\text{C}$ . According to the result, saturation magnetization, remanent magnetization, and coercivity were 22 emu/g, 10 emu/g, and 325 Oe, respectively.



**Figure 4.** The hysteresis loop of  $\text{CuFe}_2\text{O}_4$  nanoparticles calcined at  $850\text{ }^\circ\text{C}$ .

### 3.5. Microwave Characteristics

The Microwave absorption properties of  $\text{CuFe}_2\text{O}_4$  nanoparticles were revealed using silicone rubber medium. Figure 5 presents reflection losses of  $\text{CuFe}_2\text{O}_4$ /silicone rubber nanocomposite at different thicknesses. Results showed that  $\text{CuFe}_2\text{O}_4$ /silicone rubber nanocomposite absorbed more than 94.87% of microwave irradiation along the ku-band frequency with 1.7 mm thickness while the maximum reflection loss was  $-60.38$  dB at 16.1 GHz originated from suitable impedance matching, eddy current loss, magnetic resonance, conductive loss, electron migrating, as well as interfacial polarization.



**Figure 5.** The microwave absorption properties of  $\text{CuFe}_2\text{O}_4$ /silicone rubber nanocomposite at different thicknesses.

## 4. Conclusions

It can be concluded that pure  $\text{CuFe}_2\text{O}_4$  nanoparticles have been prepared using conventional and simple sol-gel method. XRD patterns confirmed that pure crystal structure of  $\text{CuFe}_2\text{O}_4$  nanoparticles were synthesized having a size of 15.6 nm. The uniform morphology of  $\text{CuFe}_2\text{O}_4$  nanostructures were confirmed by SEM images. FT-IR result showed that nanoparticles were completely calcined after the heat treatments. According to the results, the  $\text{CuFe}_2\text{O}_4$ /silicone rubber nanocomposite demonstrated considerable microwave absorption properties due to proper magnetic, dielectric, and impedance matching characteristics.

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