

## Microwave-Assisted Synthesis of $\text{CuFe}_2\text{O}_4$ Nanoparticles and Starch-Based Magnetic Nanocomposites

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### Abstract

Magnetic  $\text{CuFe}_2\text{O}_4$  nanoparticles were synthesized by a facile microwave-assisted reaction between  $\text{Cu}(\text{NO}_3)_2$  and  $\text{Fe}(\text{NO}_3)_3$ . The magnetic nanoparticles were added to starch to make magnetic polymeric nanocomposite. The nanoparticles and nanocomposites were characterized using X-ray diffraction and scanning electron microscopy. The magnetic properties of the samples were investigated using an alternating gradient force magnetometer (AGFM). The copper ferrite nanoparticles exhibited ferromagnetic behavior at room temperature, with a saturation magnetization of 29emu/g and a coercivity of 136 Oe. The distribution of the  $\text{CuFe}_2\text{O}_4$  nanoparticles into the polymeric matrixes decreases the coercivity (136 Oe to 66 Oe). The maximum coercivity of 82 Oe was found for 15% of  $\text{CuFe}_2\text{O}_4$  distributed to the starch matrix.

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## 1. Introduction

Magnetic materials are widely used as components in various applications of industrial and medical equipments. Ferrites have emerged as novel materials with vast technological and scientific interest considering their brilliant physical properties such as reliable magnetization, high coercive force, large magnetocrystalline anisotropy as well as remarkable chemical stability and low cost. Since their discovery in the 1950s the degree of interest in them has grown enormously, and is still growing today. The ferrite materials may be classified into

three different classes; spinel ferrites, garnet ferrites and hexagonal ferrites. The ferrites used for magnetic recording, data storage materials, radar absorbing materials due to their strong magnetic losses at the range of GHz frequency, magnetoelectric applications. These materials have a potential application at high frequency range due to their very low electrical conductivity, fairly large magneto-crystalline anisotropy, relatively large saturation magnetization, mechanical hardness, excellent chemical stability and low production costs [1-10].

In the last two decades polymer matrix nanocomposites have also been extensively investigated, since just a small amount of nanoparticles as an additive leads to production of novel high-performance materials with excellent physicochemical properties [11-13]. In this work, we report synthesis of  $\text{CuFe}_2\text{O}_4$  using a microwave-assisted reaction. The  $\text{CuFe}_2\text{O}_4$  nanoparticles were then incorporated into starch polymer. The magnetic properties of magnetic nanoparticles and starch/ $\text{CuFe}_2\text{O}_4$  magnetic nanocomposites were compared.

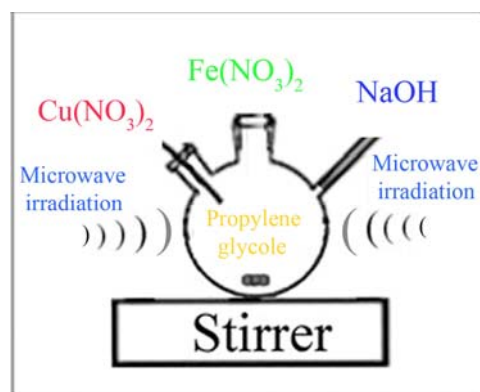
## 2. Experimental

### 2.1. Materials and characterization

$\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ ,  $\text{Cu}(\text{NO}_3)_2$ , cetyl trimethyl ammonium bromide and NaOH were purchased from Merck Company. All the chemicals were used as received without further purifications. X-ray diffraction (XRD) patterns were recorded by a Philips X-ray diffractometer using Ni-filtered  $\text{CuK}\alpha$  radiation. A multiwave ultrasonic generator (Bandeline MS 73) equipped with a converter/transducer and titanium oscillator operating at 20 kHz with a maximum power output of 100 W was used for the ultrasonic irradiation. Scanning electron microscopy (SEM) images were obtained using a LEO instrument (Model 1455VP). Prior to taking images, the samples were coated by a very thin layer of Pt (BAL-TEC SCD 005 sputter coater) to make the sample surface conducting obtain better contrast and prevent charge accumulation. Room temperature magnetic properties were investigated using an alternating gradient force magnetometer (AGFM) device (Meghnatis Daghig Kavir Company) in an applied magnetic field sweeping between  $\pm 10000$  Oe.

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

$\text{Cu}(\text{NO}_3)_2$  (0.001 mol),  $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$  (0.002 mol) and 0.25 g cetyl trimethyl ammonium bromide are dissolved in 20 mL of propylene glycol. NaOH solution (20 mL, 1 M) is then slowly added to the mentioned solution under microwave radiation (600 W) for 10 minutes (30 s on, 60s off). The black precipitate is then centrifuged and rinsed with distilled water and left in an atmosphere environment to dry. The resulting powder is then calcinated at  $850^\circ\text{C}$  in an oven for 2 hours. A schematic diagram for experimental setup used for this microwave-assisted reaction is given in Fig. 1.



**Fig. 1.** Schematic diagram of preparation of magnetic  $\text{CuFe}_2\text{O}_4$  nanoparticles

For synthesis of nanocomposite, starch (0.95, 0.9 or 0.85 g) is first dissolved in water (10 mL). The magnetic nanoparticles (0.05, 0.10 or 0.15 g) are dispersed in water (5 mL) by ultrasonic waves. The nanoparticles dispersion is then slowly added to the starch solution. The new solution is then stirred for 8 hours. To evaporate the solvent, the product is casted on a glass plate and left for 48 hours (Fig. 2).

## 3. Results and discussion

The XRD pattern of magnetic nanoparticles is shown in Fig. 3 and is indexed as a pure cubic phase (space group:  $\text{Fd-}3\text{m}$ ). The experimental values are very

close to the literature (JCPDS No. 77-0010). The crystallite size measurements were carried out using the Scherrer equation (Eq. 1),

$$D_c = 0.9\lambda / \beta \cos\theta \quad (1)$$

where  $\beta$  is the width at half maximum intensity of the observed diffraction peak, and  $\lambda$  is the X-ray wavelength ( $\text{CuK}_\alpha$  radiation, 0.154 nm). The estimated crystallite size is about 32 nm. The XRD pattern of starch/magnetic nanocomposite is shown in Fig. 4. The  $\text{CuFe}_2\text{O}_4$  peaks confirm the existence of magnetic in the starch matrix.

The SEM images of the magnetic  $\text{CuFe}_2\text{O}_4$  nanoparticles, starch/ $\text{CuFe}_2\text{O}_4$  5% and 15% are shown in Fig. 5a-5d respectively. SEM images show that nanoparticles with average diameter of 60nm are obtained. The images confirm that the ferrite nanoparticles are dispersed in the starch matrix.

The room temperature magnetic properties of the samples were studied using an AGFM device. The magnetic nanoparticles exhibit a hysteresis loop (Fig. 6). The copper ferrite nanoparticle exhibits ferromagnetic behavior at room temperature with a saturation magnetization of 29emu/g and a coercivity of 136 Oe.

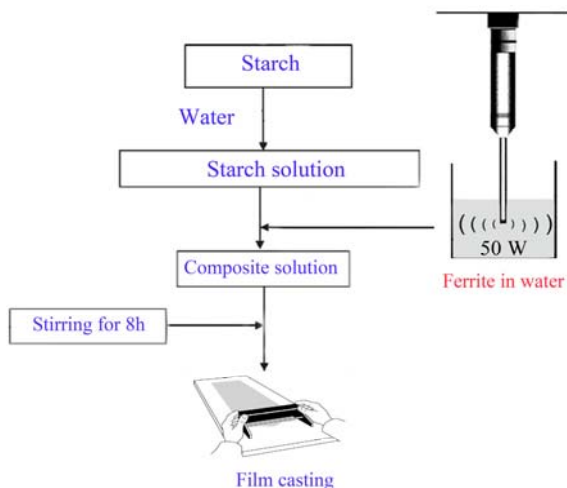


Fig. 2. Schematic diagram of preparation the nanocomposites

Based on our literature search, there are very few reports on magnetic starch nanocomposites. Therefore, we studied the magnetic interaction between the nanoparticles surrounded by starch chains. This interaction leads to a decrease (from 136 Oe to 66 Oe) of nanoparticle coersivities relative to pure copper ferrite nanoparticles. The hysteresis loops for different concentration of nanocomposites 5, 10 and 15 % are illustrated in Figs. 7-9 respectively.

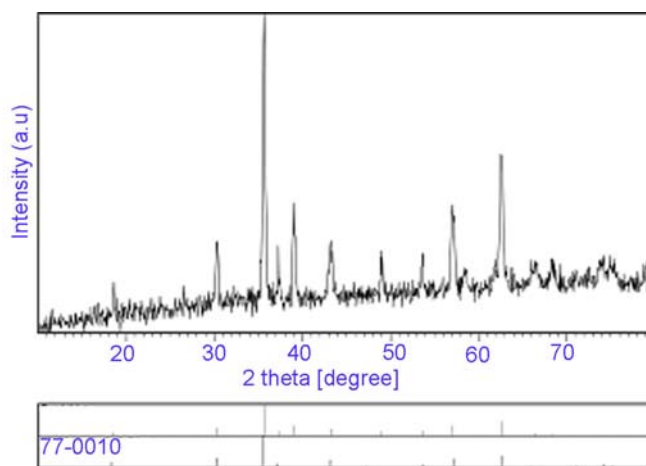


Fig. 3. XRD pattern of  $\text{CuFe}_2\text{O}_4$  nanoparticles

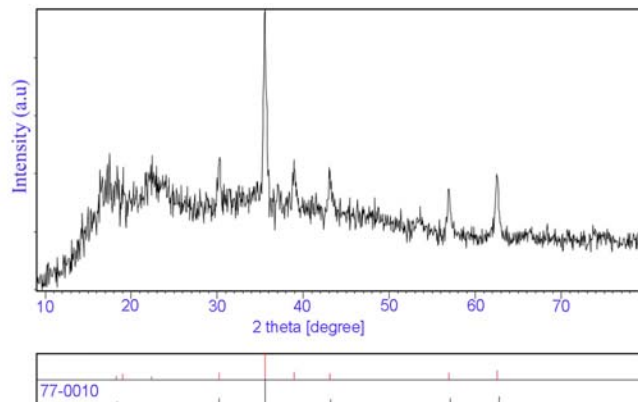
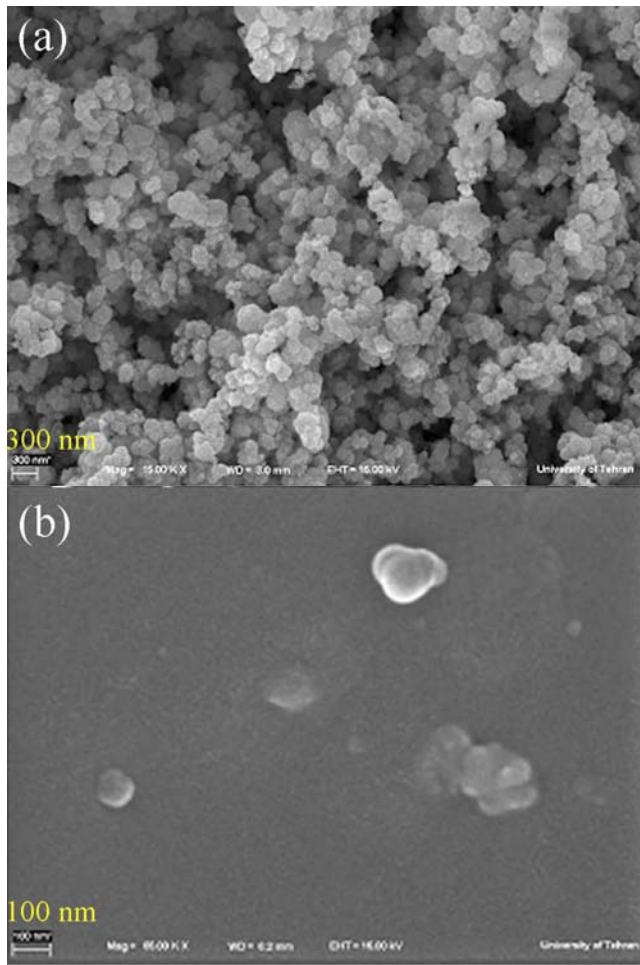


Fig. 4. XRD pattern of starch/ $\text{CuFe}_2\text{O}_4$  nanocomposite

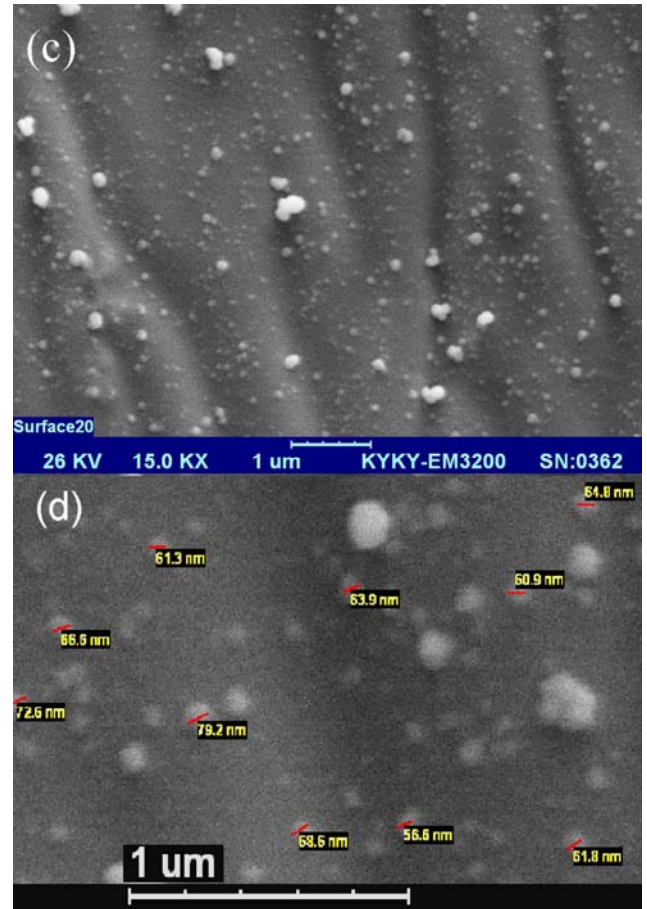
In order to make 1g of magnetic nanocomposite 10%, 0.1 g of copper ferrite nanoparticles is added to 0.9 g of starch. Thus, the nanocomposite magnetization (defined as the magnetic moment per

unit volume) is about one tenth of that obtained for  $\text{CuFe}_2\text{O}_4$  nanoparticles.

The saturation magnetization of magnetic nanoparticles is much higher than that of obtained for the starch/magnetic nanocomposites. The results also indicate that forming the nanocomposite and distributing the magnetic nanoparticles into the starch matrix leads to a decreased coercivity.



**Fig. 5.** SEM image of (a)  $\text{CuFe}_2\text{O}_4$  nanoparticles (b) starch/ $\text{CuFe}_2\text{O}_4$  nanocomposite 5%



**Fig. 5c-d.** SEM images of starch/ $\text{CuFe}_2\text{O}_4$  15% nanocomposite

A possible explanation could be as follow: The magnetic moments of the nanoparticles are agglomerated by the surrounding starch matrix chains so that a lower magnetic field is required for the alignment of the single domain nanoparticles in the field direction.

The coercivity of starch-magnetic 15% nanocomposite (82 Oe) is also higher than that of the other starch/ $\text{CuFe}_2\text{O}_4$  nanocomposites (66 Oe and 70 Oe). Coercivity of magnetic nanocomposites highly depends on the magnetic nanoparticle distribution into the starch matrixes. Since these distributions are different the coercivities also differ from one nanocomposite to the other, though the magnetic nanoparticles are

similar (but in different distributions). On the other hand, the nanocomposite magnetization (defined as the magnetic moment per unit volume) is much lower (about one tenth) than that obtained for copper ferrite nanoparticles [14-15].

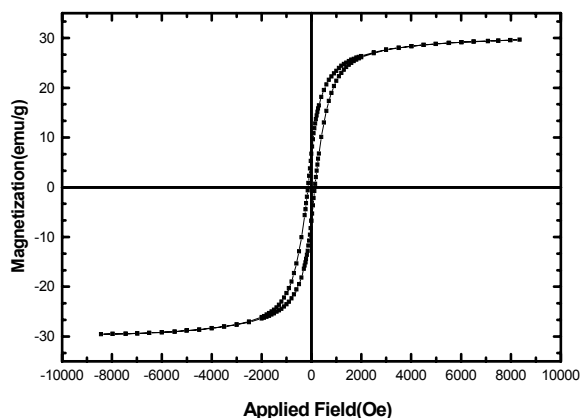


Fig. 6. Room temperature hysteresis loop of magnetic CuFe<sub>2</sub>O<sub>4</sub> nanoparticles

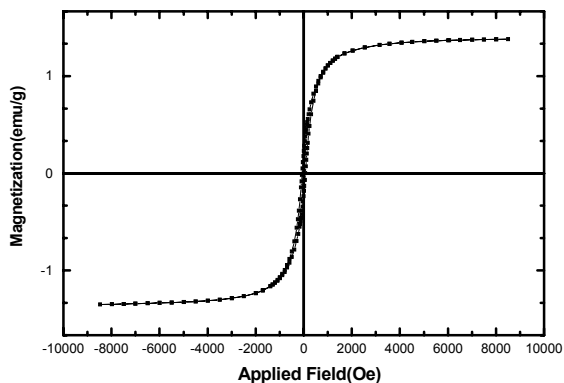


Fig. 7. Room temperature hysteresis loop of magnetic starch-CuFe<sub>2</sub>O<sub>4</sub> (5%) nanocomposite

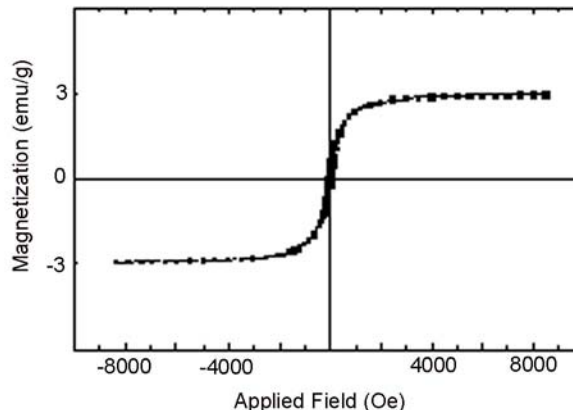


Fig. 8. The hysteresis loop of starch-CuFe<sub>2</sub>O<sub>4</sub> (10%) nanocomposite

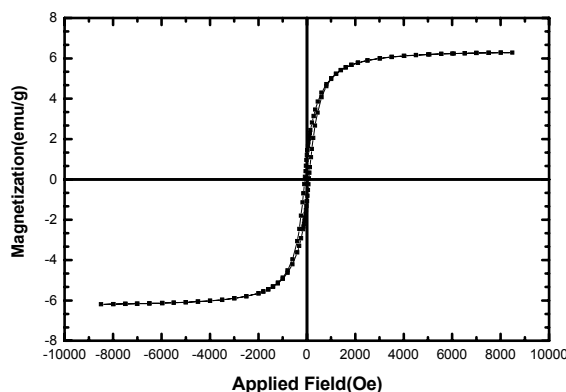


Fig. 9. The hysteresis loop for starch/CuFe<sub>2</sub>O<sub>4</sub> (15%) nanocomposite.

#### 4. Conclusion

CuFe<sub>2</sub>O<sub>4</sub> nanoparticles are synthesized by a simple microwave-assisted reaction. The CuFe<sub>2</sub>O<sub>4</sub> nanoparticles exhibited ferromagnetic behaviour with a saturation magnetization of 29 emu/g and a coercivity of 136 Oe at room temperature. CuFe<sub>2</sub>O<sub>4</sub> nanoparticles were then added to starch polymeric matrix to make magnetic nanocomposites. The nanocomposites were characterized by XRD and SEM spectroscopy. It seems agglomeration of CuFe<sub>2</sub>O<sub>4</sub> nanoparticles into the starch matrix decreases the coercivity (136 Oe to 66 Oe). A

maximum coercivity of 82 Oe was found for 15% of  $\text{CuFe}_2\text{O}_4$  distributed in the starch matrix.

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