

## Synthesis and magnetic investigation of cobalt ferrite nanoparticles prepared via a simple chemical precipitation method

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### ABSTRACT

In this research cobalt ferrite (CoFe<sub>2</sub>O<sub>4</sub>) nano-crystalline powders were prepared by simple chemical precipitation method using cobalt sulfate. The CoFe<sub>2</sub>O<sub>4</sub> nanoparticles were characterized by X-ray diffraction, scanning electron microscopy and Fourier transform infra-red spectroscopy. The crystallite size of CoFe<sub>2</sub>O<sub>4</sub> nanoparticles was calculated by Debye-Scherrer formula. The effect of precursor, capping agent, temperature and concentration on the morphology and particle size of the products was investigated. Starch and gelatin as green, safe, water-soluble and cost-effective capping agents were used. Alternative gradient field magnetometer confirms dominant influence of temperature on the morphology and magnetic domains. Results approve magnetic samples exhibit either ferromagnetic or super-paramagnetic behavior.

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### INTRODUCTION

Among the ferrite magnetic materials, cobalt ferrites have recently become the subject of research interest. This type of spinel ferrite has received special attention due to its properties, such as large magnetic anisotropy, high coercivity, moderate saturation magnetization and suitable chemical stability, as well as the adequate mechanical hardness. It possesses the so-called inverse spinel structure with one half of Fe (II) ions at the A site and the rest, together with Co (II) ions at the B site at room temperature. The magnetic properties of the cobalt ferrite nanoparticles have been found to be highly dependent on the size, shape and purity of these particles [1-10]. This type of spinel ferrite has received special attention due to its properties, like

removal of pollutant ions from aqueous systems or electronic devices. The magnetic properties of nanoparticles are sensitive to the synthesis issues such as method, reaction conditions, and particle size distribution [11-14].

A better understanding of magnetism is crucial not only for basic physics but also because of the great technological importance of ferro-magnets in information storage, color imaging, bio-processing, and ferro-fluids. Ferromagnetism occurs even for clusters with less than about 30 atoms [13-16].

In this research cobalt ferrite (CoFe<sub>2</sub>O<sub>4</sub>) nano-crystalline powders were prepared by chemical precipitation method using cobalt sulfate or cobalt acetate and green capping agent at 80 °C in solvent of water.

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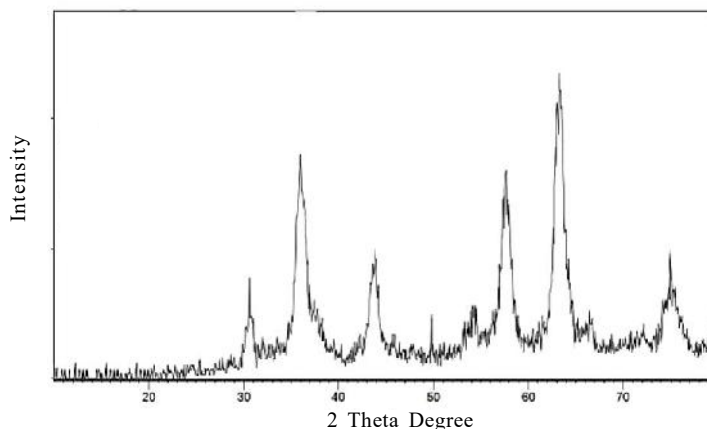


Fig. 1. XRD pattern of  $\text{CoFe}_2\text{O}_4$  nanoparticles

## MATERIALS AND METHODS

### Materials and characterization

Cobalt sulfate,  $\text{FeCl}_3 \cdot 9\text{H}_2\text{O}$ ,  $\text{NaOH}$ ,  $\text{NH}_3$  32%, ethylene glycol and acetone were purchased from Merck and all the chemicals were used as received without further purifications. 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 150 W was used for the ultrasonic irradiation. Room temperature magnetic properties were investigated using an alternating gradient force magnetometer (AGFM) device, made by Meghnatis Kavir Kashan Company (Iran) in an applied magnetic field sweeping between  $\pm 10000$  Oe. XRD patterns were recorded by a Philips, X-ray diffractometer using Ni-filtered  $\text{CuK}\alpha$  radiation. 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 (using a BAL-TEC SCD 005 sputter coater) to make the sample conductor.

### Synthesis of $\text{CoFe}_2\text{O}_4$ nanoparticles

0.2 g of  $\text{CoSO}_4$  (or  $\text{Co}(\text{CH}_3\text{COO})_2 \cdot 4\text{H}_2\text{O}$ ) and 0.43 g of  $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$  were dissolved in 100 ml of deionized water. 30 ml of  $\text{NaOH}$  solution (1M) was then slowly added to the solution until reaching pH to around 10. A brown precipitate was then centrifuged and rinsed with distilled water. Finally the obtained solution was remained at  $80^\circ\text{C}$  and its color goes from brown to black.

## RESULTS AND DISCUSSION

The XRD pattern of  $\text{CoFe}_2\text{O}_4$  nanoparticles is shown in Fig. 1. XRD analyses were performed to determine

the crystalline structure and phase formation of cobalt ferrite nanoparticles. The pattern confirms formation of pure cubic cobalt ferrite with JCPDS 22-1086 and space group of  $\text{Fd}3\text{m}$ .

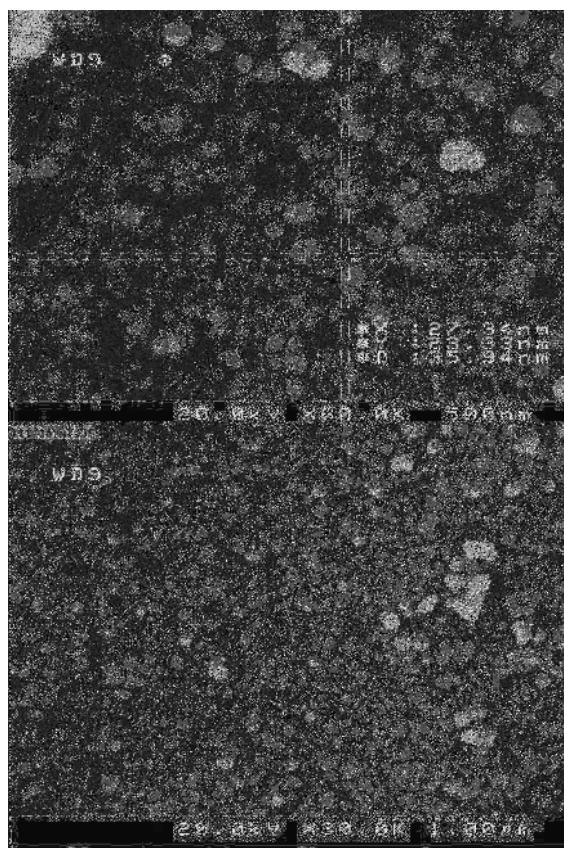


Fig. 2. SEM images of surfactant-free  $\text{CoFe}_2\text{O}_4$  nanoparticles

The nanoparticles crystallite size was calculated from X-ray line broadening using Debye–Scherrer equation [13]:

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

where  $\lambda$  is the X-ray wavelength (CuK $\alpha$  radiation equals to 1.54Å),  $\theta$  is the Bragg diffraction angle, and  $\beta$  is the FWHM of the XRD peak appearing at the diffraction angle  $\theta$ . The crystallite sizes calculated is about 27 nm.

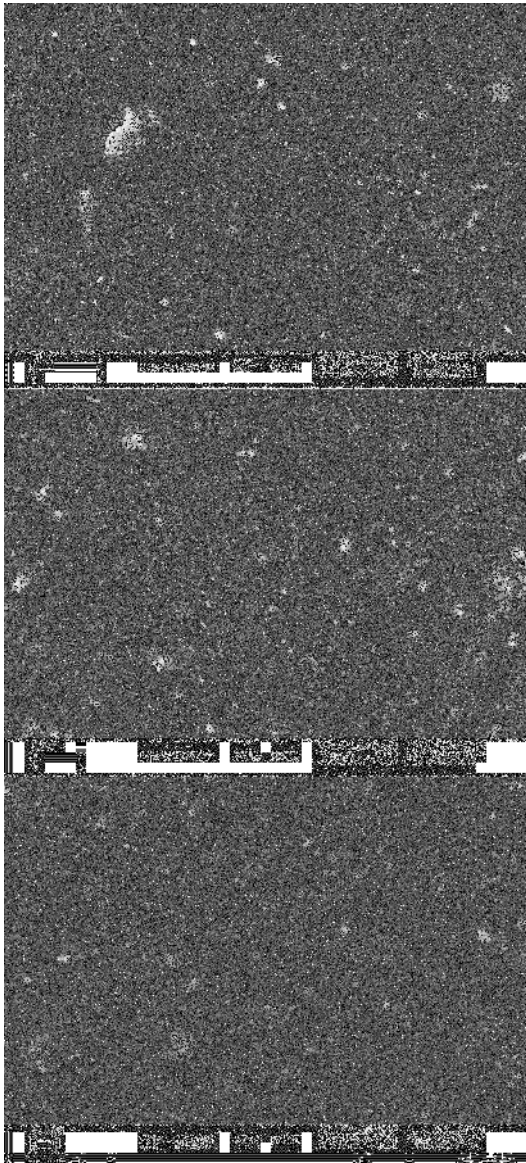


Fig. 3. SEM images of CoFe<sub>2</sub>O<sub>4</sub> nanoparticles at 400 ml of water (diluted sample)

SEM images of surfactant-free CoFe<sub>2</sub>O<sub>4</sub> are shown in Fig. 2., the results confirm formation of mono-disperse nanoparticles with average diameter less than 30 nm.

Influence of concentration was examined and diluted solution was prepared, SEM images of cobalt ferrite at 400 ml of water are shown in Fig. 3. The images show that the obtained cobalt ferrite nanocrystals have approximately spherical shape with an average diameter less than of 80 nm. The effect of capping agent on the morphology was investigated. Starch and gelatin as green, safe, water-soluble and cost-effective capping agents were used. SEM images of ferrite by gelatin are shown in Fig. 4 and outcomes show synthesis of mono-disperse nanostructures (around 60nm). SEM images of CoFe<sub>2</sub>O<sub>4</sub> with starch are shown in Fig. 5. Images depict formation of agglomerated product; by the way nano dimensions exist in the ferrite.

The influence of cobalt source on the morphology and particle size was examined. Fig. 6 illustrates

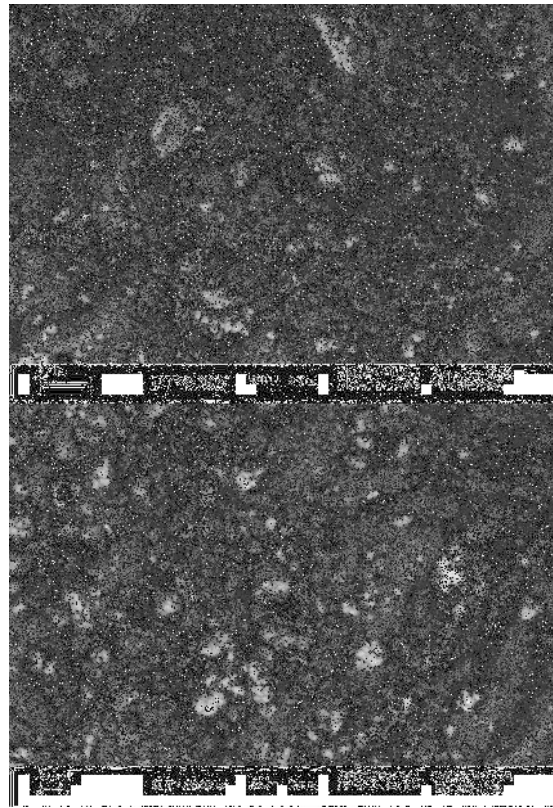


Fig. 4. SEM image of ferrite nanoparticles obtained by gelatin

product that obtained by cobalt acetate as another precursor. The images approve preparation of nanostructures with mediocre size around 60 nm near agglomerated product simultaneously.

Fig. 7 shows the FT-IR spectrum of the surfactant-free  $\text{CoFe}_2\text{O}_4$  nanoparticles. The peaks at 283 and  $582\text{ cm}^{-1}$

are the characteristic absorption of Fe-O and Co-O bonds. Other absorption peaks at  $3327\text{ cm}^{-1}$  which corresponding the hydroxyl adsorbed on surface of the materials. This result has a suitable agreement with other works [16-20].

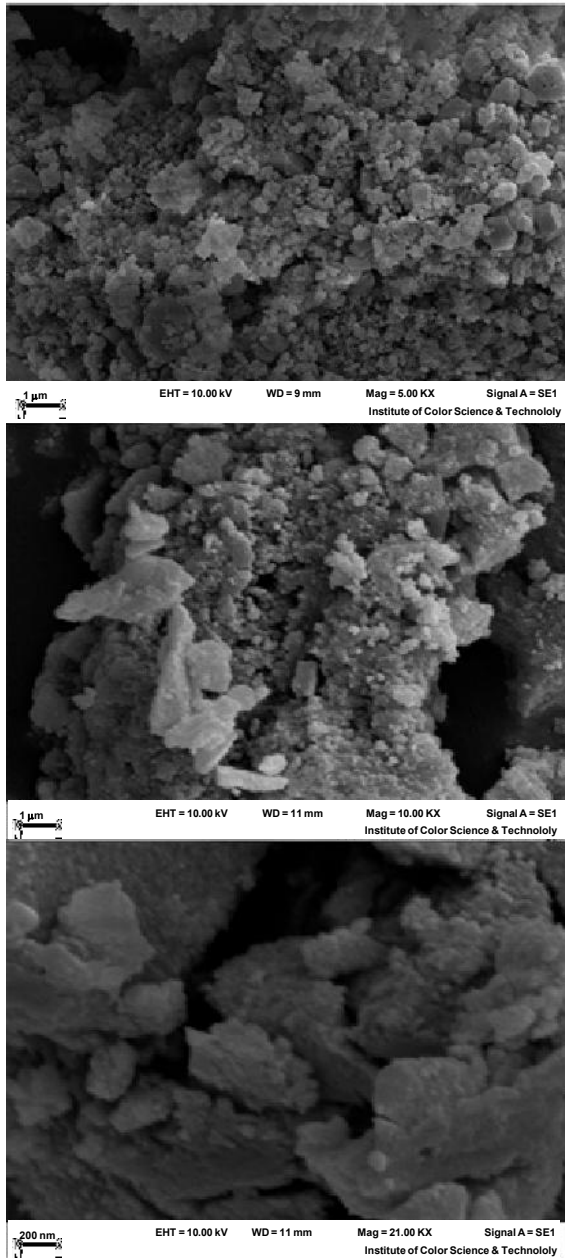


Fig. 5. SEM image of  $\text{CoFe}_2\text{O}_4$  nanoparticles synthesized by starch

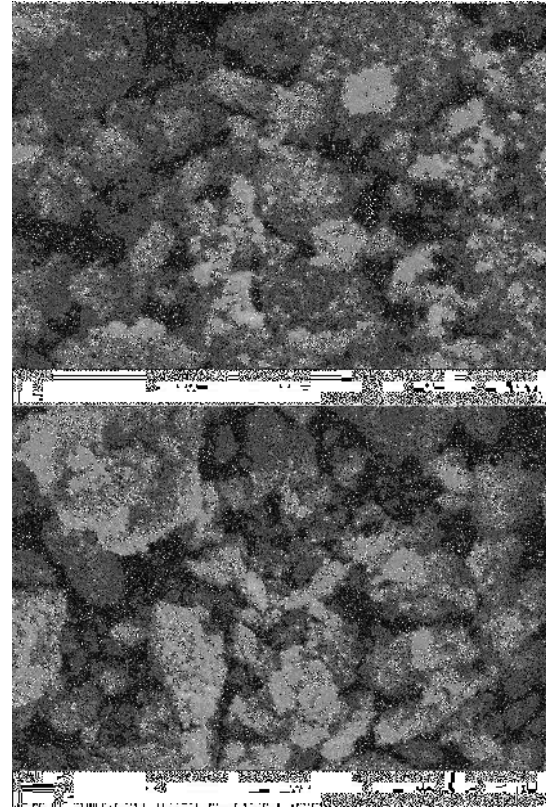


Fig. 6. SEM image of  $\text{CoFe}_2\text{O}_4$  nanoparticles synthesized by cobalt acetate

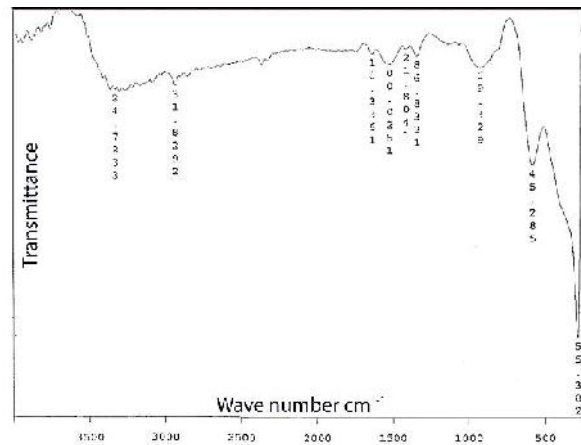
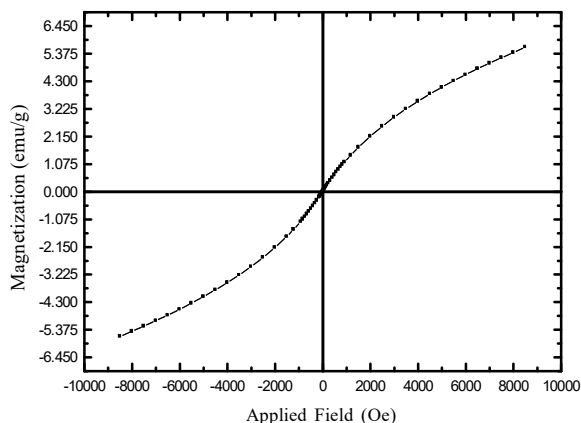
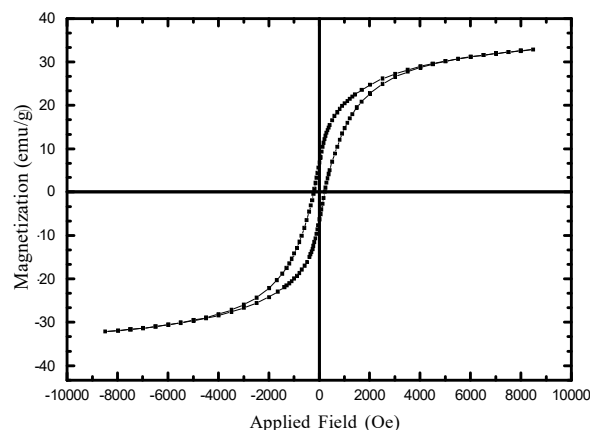


Fig. 7. FTIR spectrum of ferrite nanoparticles

Fig. 8. AGFM of  $\text{CoFe}_2\text{O}_4$  nanoparticles.Fig. 9. AGFM of  $\text{CoFe}_2\text{O}_4$  nanoparticles at calcination temperature of 500 °C.

Room temperature magnetic property of uncalcinated sample was studied using AGFM instrument and is shown in Fig. 8. The result indicates that, before calcination, the samples exhibit a super-paramagnetic property, with a saturation magnetization about 5.4 emu/g and a very small coercivity (less than 50 Oe). Room temperature magnetic property of calcinated product at 500 °C is illustrated in Fig. 9. The outcomes interestingly show the effective role of temperature on the magnetic domains. The obtained ferrite illustrates ferromagnetic property with a saturation magnetization around 32 emu/g and a coercivity about 150 Oe.

## CONCLUSION

In conclusion, synthesis and magnetic characterization of  $\text{CoFe}_2\text{O}_4$  nanoparticles was reported. Effect of precursor, green capping agent, temperature and concentration on the morphology and particle size of the products was investigated. AGFM confirmed significant influence of temperature on the morphology and magnetic domains. Results approve magnetic samples exhibit either ferromagnetic or super-paramagnetic behavior.

## CONFLICT OF INTEREST

The authors declare that there are no conflicts of interest regarding the publication of this manuscript.

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