

INFLUENCE OF PRECURSORS ON STRUCTURE AND MAGNETIC PROPERTIES OF CuFe_2O_4 OBTAINED BY COPRECIPITATION

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ABSTRACT

Nanoparticles of copper ferrites were obtained by co-precipitation method using two precursors $\text{Cu}(\text{CH}_3\text{COO})_2$ and $\text{Cu}(\text{OH})_2$ with $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$. In this paper, we try to demonstrate the influence of precursors on structure and magnetic properties of CuFe_2O_4 obtained by co-precipitation allow the preparation of high reactive ferrite nanoparticles whose composition, microstructure, size and properties can be rigorously controlled in order to obtain the special requirements of various advanced applications.

INTRODUCTION

Copper ferrite (CuFe_2O_4) is one of the important spinel ferrites MFe_2O_4 because it exhibits phase transitions, changes semiconducting properties, shows electrical switching and tetragonal variation when treated under different conditions in addition to interesting magnetic and electrical properties with chemical and thermal stabilities[1]. The method of preparation plays a very important role in determining the chemical, structural and magnetic properties of spinel ferrites [2]. The essential requirements of obtaining well controlled uniformity and high-purity materials encouraged the development of wet chemical methods, such as coprecipitation[3,4], hydro/solvothermal synthesis[5-6], micro-emulsion[7] and sol-gel technique[8].

It is used in the wide range of applications in gas sensing [9], catalytic applications[10], Li ion batteries[11] high density magneto-optic recording devices, colour imaging, bio-processing, magnetic refrigeration and ferrofluids, for the removal of acid orange II and catalytic regeneration[12-13].

MATERIALS and METHODS

In this paper we synthesized two fine copper ferrite particles using co-precipitation technique, using for precursors $\text{Cu}(\text{CH}_3\text{COO})_2$ and $\text{Cu}(\text{OH})_2$ with $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$. The copper ferrite particles were prepared by regular co-precipitation as follows, $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ (purity 99%) with $\text{Cu}(\text{CH}_3\text{COO})_2$ (purity 99%) and $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ with $\text{Cu}(\text{OH})_2$ were taken in a $\text{Cu}/\text{Fe} = 1:2$ mole ratio. The materials were dissolved in distilled and de-ionized water. The two solutions of $\text{Cu}(\text{CH}_3\text{COO})_2$ and $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ (P5) were mixed together on magnetic stirrer with continuous stirring at a moderate speed for 30 minutes and the same way the second solution $\text{Cu}(\text{OH})_2$ with $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ (P6). In another beaker, solution of 1M NaOH was prepared. After this addition of

NaOH up to pH 12 at continuous stirring a dark precipitate was obtained. The precipitate was filtered, washed with deionized water and then dried at 60°C for 4h. A scanning electron microscope SEM was used for observing the sample morphology spectra were analyzed using a FEI, Inspect-S microscope. Elemental X-ray powder diffraction was performed at room temperature (20±2°C) on the PANalytical X'Pert Pro MPD diffractometer using CuK α radiation ($\lambda=1.54$ Å) between 20 and 100° (2 θ) with an integrated step scan of 0.016° (2 θ). Magnetic studies were carried out using a conventional induction method [15], in AC magnetic fields up to 4 kOe

RESULTS

Figure 1 presents the X-ray diffraction pattern of the powders synthesized by co-precipitation method (P5) and (P6).

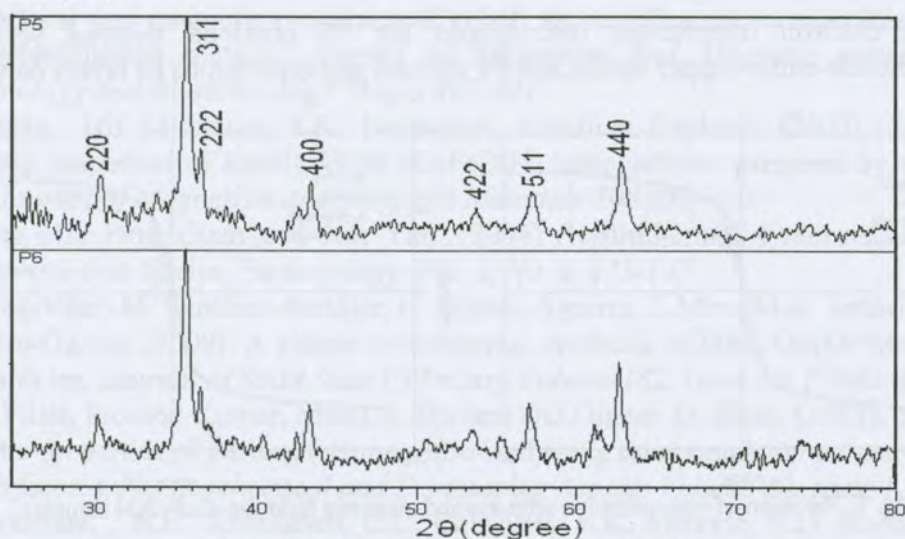


Fig. 1. XRD patterns of the CuFe₂O₄ samples

The diffraction peaks are intense, revealing a good crystallization degree for copper ferrite. From the XRD patterns, the average crystalline sizes for the spinel phase were estimated from the broadening of the strongest diffraction peak using Scherrer equation given by:

$$D = K\lambda/\beta\cos\theta_{\beta} \quad \text{with} \quad \beta^2 = \beta_a^2 - \beta_b^2 \quad (1)$$

where D is the average crystallite size, K is the shape factor (we take $K=0.9$), λ is the X-ray wavelength used, β is the measure of the broadening of the peak in a diffraction pattern, β_a and β_b are the full-width at half-maximum of the XRD line of the sample of a standard specimen respectively at around the same Bragg's angle and θ_{β} is the Bragg's angle in degree. Using this relation (eq. (1)), the average crystallite sizes, for the samples using a (3 1 1) reflection have been estimated for sample P5=21.8nm and for the sample P6=14nm

The SEM images from Fig. 2(a) shown low degree of agglomeration that in Fig 2(b)

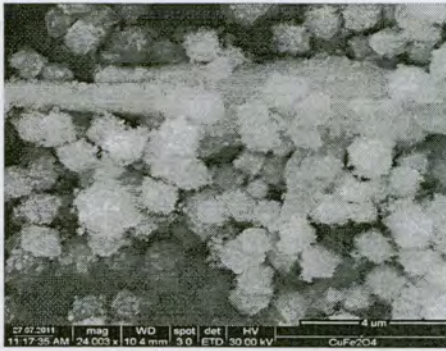


Fig. 2 (a)



Fig. 2 (b)

Fig. 2.(a) and (b) SEM micrographs of the CuFe₂O₄ sample

The scanning electron microscopy micrographs for P5 obtained showed very fine and homogenous pseudo-cubic copper ferrite, and P6 showed agglomerations of layers copper ferrite.

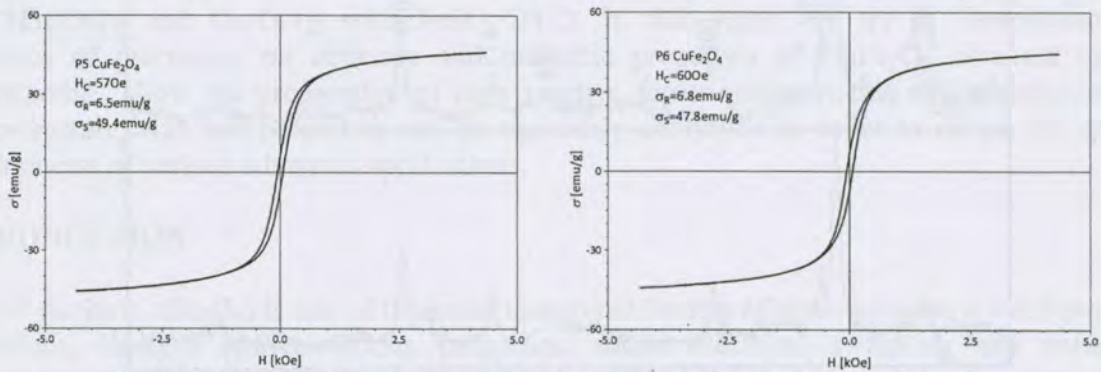


Fig. 3. Variation of magnetization with applied magnetic field for CuFe₂O₄ samples

The magnetic measurements made on the samples indicate a small coercive field that presents almost superparamagnetic behavior and the specific saturation magnetization for P5 is 49.4emu/g and for P6 is 47.8emu/g.

CONCLUSIONS

Nanoparticles of copper ferrites were obtained by co-precipitation method using two different precursors of Cu we established the following characteristics:

- The precursors have strongly influenced the morphology, crystallite size, microstructures.
- Saturation magnetization values for P5-49.4, for P6-47.8 and magnetic coercivity smaller for sample P5-570e and for P6-600e.
- The crystallite size for sample P5 is 21.8nm and for the sample P6 is 14nm.
- The sample P5 very fine and homogenous pseudo-cubic copper ferrite and P6 presents agglomerations of layers copper ferrite.

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