

STRUCTURAL CHARACTERIZATION OF CuFe_2O_4 NANOCOMPOSITES AND SYNTHESIS BY AN ECONOMICAL METHOD

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ABSTRACT: Copper ferrites were prepared through co-precipitation technique. The aim of this effort was to present a novel and economical method of preparation of copper ferrites via co-precipitation technique. Structural properties were studied with the help of XRD technique while micro structural study of the samples was carried out by SEM. The particle size was calculated with the help of Scherrer's formula using characteristic peaks. The SEM images showed uniformity and homogeneity of the synthesized CuFe_2O_4 particles.

Key words: Copper ferrite, Low cost ferrites, Co-precipitation

1. INTRODUCTION

Ferrites (CuFeO_4) have paramount advantages over other types of magnetic materials like high electrical resistivity and resultant low eddy current losses over a wide range of frequency. For the most favorable combination of low cost, high quality, high stability, and lowest volume, ferrites are considered to be the best core material choice for frequencies from 10 KHz to 50 MHz. Ferrites offer an unmatched flexibility in magnetic and mechanical parameters [1]. The development and consistent successes in switch-mode power supplies is continuously encouraging the ferrite industry to produce new, high quality ferrite cores capable of operating at increasingly higher frequency [2]. The ferrite particles can be used for the synthesis of temperature sensitive magnetic fluid with higher magnetization and magnetization-temperature gradient [3]. Of all magnetic materials, ferrites are the most useful because in addition to many magnetic properties, they are also good electrical insulator, unlike the ferromagnetic metals. Thus losses due to free electrons are eliminated. The crystallography, electrical and magnetic properties of ferrites depend upon the chemical composition as well as on the various heat treatments during the course of preparation [4]. Different studies have been made to synthesize copper ferrites by using different techniques. These studies revealed that the magnetic performances and microstructure depend considerably on chemical composition and sintering temperature of samples [5]. Ferrite shrinks when sintered, depending on the specific ferrite, this shrinkage can range from 10% to 17% in each dimension. The grain size significantly increases with increasing copper contents and sintering temperature T_s , also affects the densification, grain growth and initial magnetic permeability of the samples [6]. Hankare and co-workers [7] used oxalate precipitation method to synthesize single-phase Cu–Co ferrite. Wang et al. [8] used NaOH as precipitant to fabricate Cu-substituted NiZn ferrite and confirmed the formation of cubical spinel structure at all the temperatures for calcinations and sintering. Yang et al. [9] synthesized copper ferrite (CuFe_2O_4) by using three different techniques, citric acid-

assisted sol–gel method, solid-state reaction and co-precipitation method. Conventional oxide ceramic process have been applied by Ahmed et al. [10] to synthesize nanocrystalline copper ferrite and indicated that with a firing temperature of 1100 °C, the samples have higher bulk density (3.93 g/cm^3), whereas at 1200 °C higher saturation magnetization (45.2 emu/g) and lower co-ercivity (6.13 Oe). Various publications also explain successful synthesis of nanosized copper ferrites, and their characterizations [11, 12, 13]. The current effort has been focused to synthesize a low cost copper ferrite via co-precipitation technique. Of all these techniques, chemical co-precipitation seems to be the most convenient to synthesize nanoparticles because of its simplicity and better control over crystallite size and other properties of the materials [14]. Characterization has been done using X-ray diffraction and scanning-electron microscope.

EXPERIMENTAL SECTION

2.1 Chemicals. Ferrous Sulphate anhydrous (FeSO_4), Citric Acid ($\text{C}_6\text{H}_8\text{O}_7$), and Ethylene Glycol ($\text{C}_2\text{H}_4\text{O}_2$) were purchased from Merck where as Copper Sulphate (CuSO_4) and Sodium Hydroxide (NaOH) were purchased from Fischer and Sigma Aldrich respectively.

2.2 Instruments. X-ray diffraction (XRD) experiments were performed with PANalytical, Philips and scanning electron microscopic (SEM) images were captured by using JEOL-JSM 5910. For SEM each sample was prepared and coated by gold in Spi-Module Sputter Coater because Scanning Electron Microscopy requires conductor material to analyze the morphology of samples.

2.3 Preparation of Copper Ferrites. Cu ferrites (CuFe_2O_4) were prepared according to the method adopted by Shin et al. [15] with minor changes. They synthesized CuFe_2O_4 with metallic chlorides and used KOH (5N) as precipitant by maintaining pH 10 but we used NaOH (1N) as precipitant and metallic sulphates by maintaining pH 12. The total molarity of the metallic ion solution was kept constant. The samples of CuFe_2O_4 were prepared by co-precipitation from CuSO_4 and FeSO_4 salts and citric acid was added to act as complexant. Shin and co-workers mixed the chloride salts with KOH at temperature of 353K but we mixed at room

temperature. The bath temperature was maintained at 85 °C. The blackish brown precipitates so obtained were washed several times with de-ionized water till filtered water of precipitates attained pH 7 and finally washed by acetone several times. The bath temperature was maintained at 85 °C. The final product was dried in oven for 6 to 8 hrs at 50 °C and grinded to fine powder with the help of pestle and mortar. The micro structural characteristics were examined via XRD technique and Scherrer's formula was used to find crystallite size ($t = 0.9 / B \cos \theta$, [16], where θ is the wavelength of the X-rays used. B is deduced from the characteristic peak at full width half maximum (FWHM).

3. RESULTS AND DISCUSSION

We have successfully employed co-precipitation technique and synthesized the Copper ferrites; results were compared with that of Shin et al's [15] results and were found in a close agreement. In the present work, synthesized copper ferrite showed all the peaks of XRD pattern in close agreement with JCPDS card no. 00-025-0283. There were additional peaks corresponding to extra phases such as γ - Fe_2O_3 which showed incomplete reaction. Crystallite size was estimated in nm [17]. The analysis of the peak positions and the relative intensities of the diffracted lines of the synthesized copper ferrites were compared with XRD pattern of Gomes et al. [18]. By comparing the XRD patterns of the prepared samples, it was found that these

were not indexed to single phase of spinal structure. Particle size of each sample was calculated with strongest diffraction peak (311), Fig.1. It was observed that broadening of diffraction peak (311) was directly relating to the particle size. So in this regard Scherrer's formula was used to find the crystallite size. The previous study clearly depicts that the pH of the solution has important factor regarding the ferrite formation. In the present study we maintain the pH of the solution at 12.

In sample K_{1A} four peaks out of seven major peaks have been matched with the standard pattern of CuFe_2O_4 . The peaks with miller indices (222), (400), (533) did not match with the standard pattern of CuFe_2O_4 . The following miller indices (422), (333) and (622) were not present in our synthesized CuFe_2O_4 . The synthesized CuFe_2O_4 was found out with FCC structure. Lattice constants have been presented in table 1.

In sample K_{2A} six peaks out of eight major peaks were found out in close agreement with the standard patterns of CuFe_2O_4 . The peaks with miller indices (111) and (422) did not match with the standard pattern of CuFe_2O_4 , Fig. 2. The following miller indices (400) and (300) were not present in the synthesized CuFe_2O_4 . The structure CuFe_2O_4 was found to be FCC. Lattice constants have been listed in table 2.

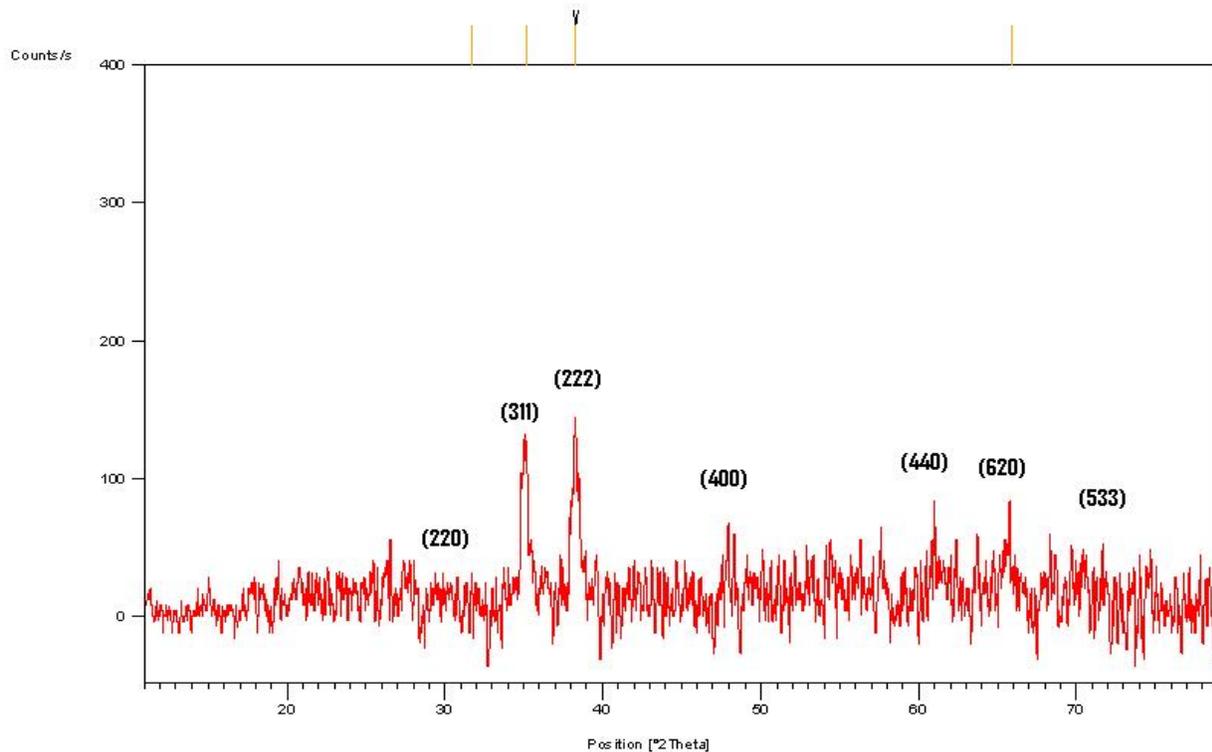


Fig. 1: XRD Pattern of Sample K_{1A}

Table 1: Peak analysis of XRD Pattern of Sample K_{1A}

2θ (deg)	θ (deg)	I/I _{max} %	(hkl) Miller indices	d-value	a (Å)
31.706	15.853	17.14	(220)	2.822	7.98
35.121	17.561	72.59	(311)	2.555	8.47
38.301	19.151	100.0	(222)	2.351	8.14
65.911	32.955	14.29	(531)	1.416	8.38

The above table confirmed the miller indices and lattice constants. The average lattice constant was 8.24(Å). The X-ray density was calculated 5.67g/cm³.

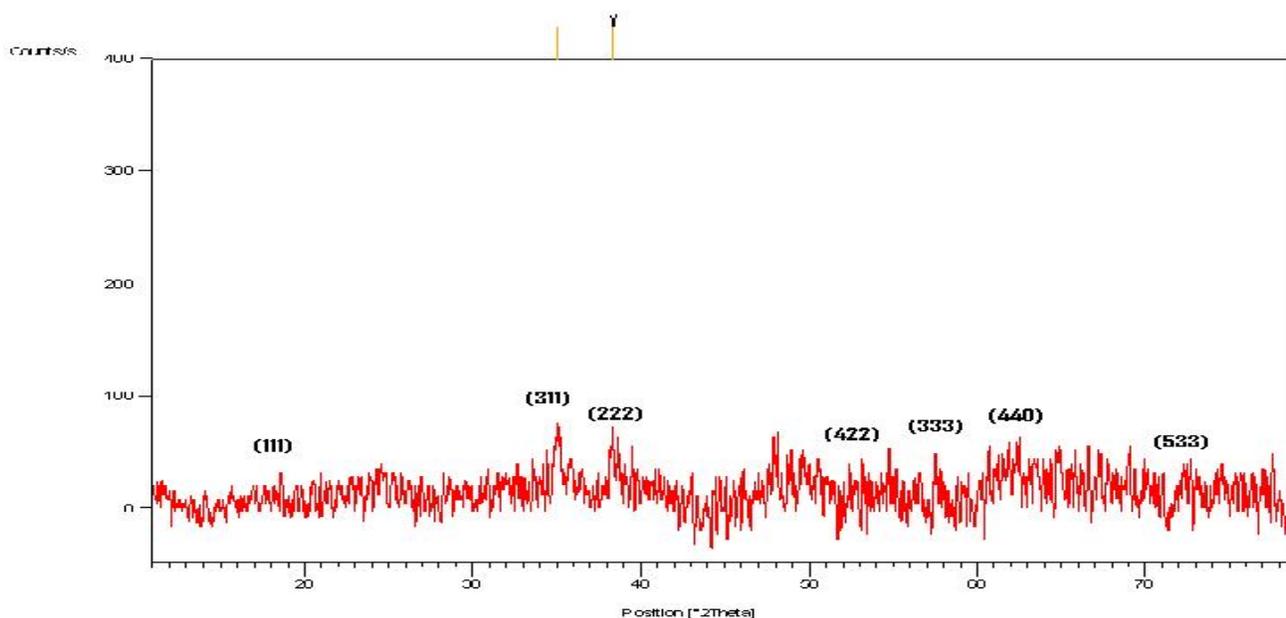
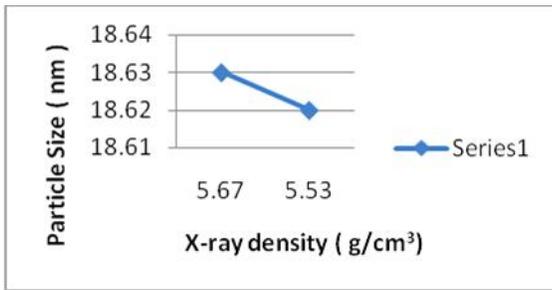


Fig. 2: XRD Pattern of Sample K_{2A}

Table 2: Peak analysis of XRD Pattern of Sample K_{2A}

2θ (deg)	θ (deg)	I/I _{max} %	(hkl) Miller indices	d-value	a (Å)
35.069	17.535	100.0	(311)	2.228	8.48
38.331	19.165	34.43	(222)	2.348	8.14

The above table confirmed the miller indices and lattice constants. The average lattice constant was 8.31(Å). The X-ray density was calculated 5.53g/cm³.



Graph 1: Relationship between particle sizes and X-ray density

Samples #	Particle size (XRD) (nm)
K _{1A}	18.63
K _{2A}	18.62

T
a
b

Table 3: Particle sizes obtained from X-ray diffraction technique

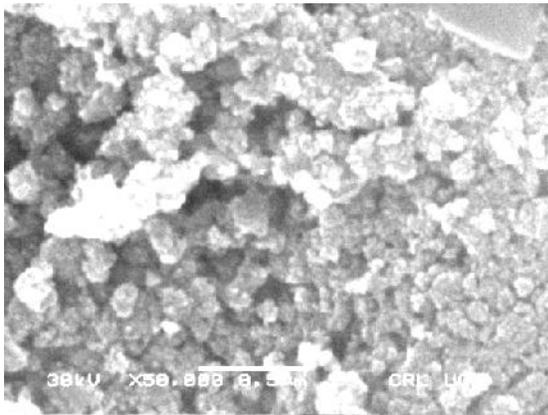


Fig. 3: SEM Analysis of Sample K_{1A} at 50,000X The above figure shows the homogeneity and uniformity of particle sizes. This Scanning Electron Microscopic image also confirms the particle sizes of the nanomaterials.

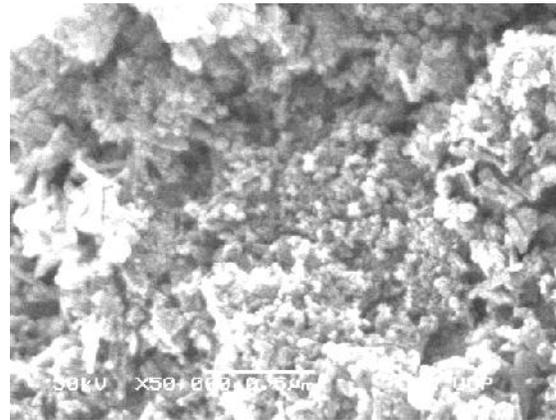


Fig. 5: SEM Analysis of Sample K_{2A} at 50,000 magnification. This Scanning Electron Microscopic image also shows the amorphous structure of synthesized Copper ferrite.

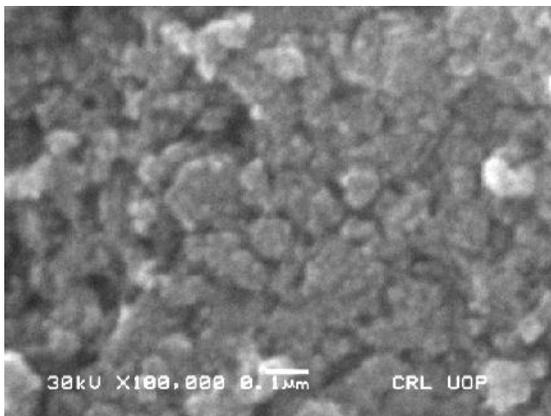


Fig. 4: SEM Analysis of Sample K_{1A} at 100,000X.

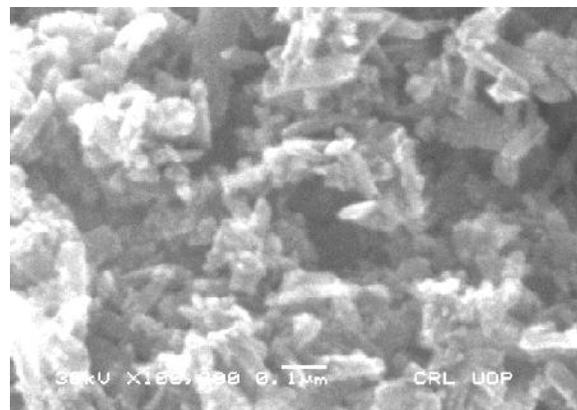


Fig. 6: SEM Analysis of Sample K_{2A} at 100,000X.

4. CONCLUSION

Homogeneous CuFe₂O₄ nanoparticles were prepared by simple and economical chemical co-precipitation method at room temperature. It is reported that the water bath temperature, 85 °C, is the temperature where the most suitable results can be obtained.

ACKNOWLEDGMENT

The authors would like to acknowledge Department of Physics University of Agriculture Faisalabad for technical assistance and greatly indebted to the University Sains Malaysia (USM) for providing state of the art facilities and *graduate assistantship (GA).

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