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Synthesis and characterization of $CuFe_2O_4$ nanoparticles

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ABSTRACT : Nanocrystalline Copper Ferrite ($CuFe_2O_4$) particles were successfully synthesized at room temperature using chemical co-precipitation method. These nanoparticles are synthesized without annealing. FTIR analysis was used for structural investigations. FTIR frequency data for the respective sites are also analyzed. The higher frequency band and lower frequency band are assigned to the tetrahedral and octahedral complex. X-ray diffraction pattern confirms the formation of single-phase cubic structure. Their corresponding reflection planes are also determined. The lattice constant calculated from XRD peaks is 8.398 Å. Crystallite size calculated from XRD peak broadening confirms an average particle size of 20 nm. Particle size measured using SEM show good agreement with the calculated Value. EDAX analysis was used for compositional studies.

INTRODUCTION

The importance of ferrites has well realized in the field of electronics and communication. The domain of application of ferrites has spread to a large variety in inductive cores of filters, power transformers, in switched mode devices (SMD) and in phase shifters in broadband application. In the recent past, miniaturization of magnetic components has been made possible due to the design of economic flat design power transformers (EDF) using ferrites [1]. Recently, processing of mixed ferrites has attracted the attention of material scientists. Many mixed ferrites have been newly prepared [2]. The properties of ferrite material are strongly affected by materials composition, microstructure and sintering conditions. The Cu–Fe–O system has been of long standing interest in Solid-state physics, mineralogy, ceramics, and metallurgy. By virtue of magnetic and semi conducting properties, Copper Ferrite ($CuFe_2O_4$) and its solid solutions with other ferrites are widely used in the electronic industry. Copper Ferrite has two crystallographic spinal structures. The high-temperature cubic phase (c-$CuFe_2O_4$) with a lattice parameter of 8.380Å, and the low-temperature tetragonal phase (t-$CuFe_2O_4$) with lattice parameters of a=8.216 Å and c=8.709 Å. The ideal inverse configuration consists of eight divalent (Cu^{2+}) ions on the octahedral (B) sites and 16 trivalent ($Fe^{3+)}$ ions equally splitting between the tetrahedral (A) and B sites per unit Cell [3]. The nanocrystalline ferrite is being synthesized by various techniques such as sol-gel, hydrothermal, ultrasonic emulsion, reverse micelle synthesis and combustion [4]. In the present work, we applied chemical co-precipitation method for synthesizing the nanoparticles of $CuFe_2O_4$. The open-ended nature of the results of the earlier studies has motivated a systematic characterization of these materials using SEM, FTIR, and XRD techniques.

EXPERIMENTAL PROCEDURE

The starting chemicals used in the present study were Cupric Chloride ($CuCl_2\ 2H_2O$) and Anhydrous Ferric chloride ($FeCl_3\ 6H_2O$). Mixed solution consisting of one mole of $CuCl_2{:}2H_2O$ and two mole of $FeCl_3{:}6H_2O$ has slowly heated to 70°C. Thereafter, the Oleic acid (5ml) was added for surface coating .The solution was allowed to cool down slowly with continuous stirring up to room temperature. Few drops of Nitric acid (HNO_3) were added to precipitate the formed coated particles. The precipitate was taken out and washed number of time with hot distilled water so as to remove sodium chloride. Finally, water was removed by washing it with acetone. This acetone wet-slurry was dispersed in 20ml of kerosene and heated at 50°C for 5 minutes. The resulting fluid was centrifuged at 12000 rpm for 15 minutes. For obtaining the dried particles, portion of the fluid was repeatedly washed with acetone.

RESULT AND DISCUSSION

Fig.1 shows X-ray diffraction patterns of the sample $CuFe_2O_4$, obtained at room temperature using Cu-k_α radiation. Every peak in the pattern can be indexed on a cubic unit cell. Careful analysis of the observed peak positions and intensities shows that $CuFe_2O_4$ has the cubic spinal structure. The diffraction peaks of the $CuFe_2O_4$ phase lost intensities and significantly broadened. This has made it difficult to distinguish the tetragonal from cubic structure. The full width at half maximum (FWHM) of the XRD peaks were used to calculate the crystallite size (D) using Scherrer's relation.

$$D = 0.9\ \lambda/\beta \cos\theta$$

Where ² is the broadening of the diffraction line measured at half maximum intensity (radians) and λ = 1.5406Å, the wavelength of Cu-K_α radiation. The lattice constant of $CuFe_2O_4$ nanocrystal were determined using the following relationship [5].

$$a = d_{hkl}\,(h^2 + k^2 + l^2)^{1/2}$$

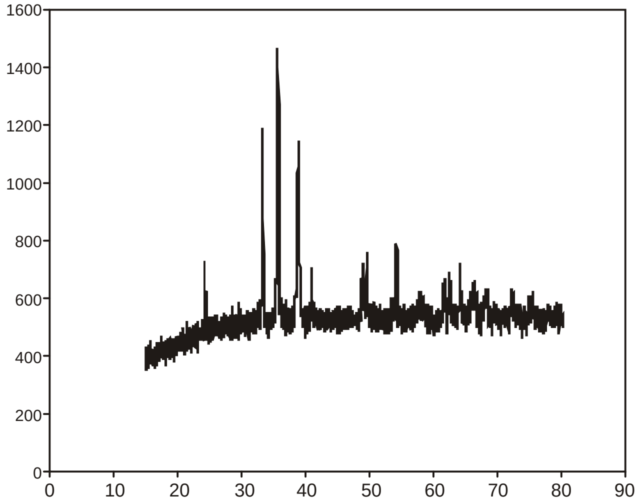

Fig.1. X-ray diffraction pattern of $CuFe_2O_4$.

The average particle size was found to 20nm. The calculated lattice parameter a = (8.398 ± 0.005) Å was found to be grater than the reported value. Different **d** values were calculated taking the lattice constant $a = b = c = 8.398 \pm 0.005$Å. The reduction in the **d** values can be explained on the basis of earlier studies [6]. The composional analysis was carried out using EDEX (Fig.2).

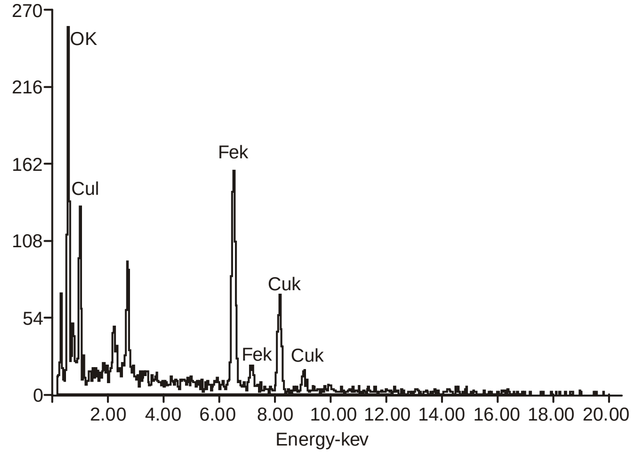

Fig.2. The EDAX spectra.

Element	Wt%	At%
OK	24.10	53.99
FeK	41.18	26.43
CuK	34.71	19.58
Matrix	Correction	ZAF

The results of EDAX confirm the initial composition. The FTIR spectra shown (Fig.3) further confirm the presence of various functional groups in the synthesized compound. FTIR spectra were recorded in the range of 400–4000 cm^{-1}. The IR bands of solids are usually assigned to the vibration of ions in the crystal lattice [7]. Two main broad metal oxygen bands are seen in the IR spectra of all spinals, and ferrites in particular. The FTIR spectrum has two absorption bands at approximately 400-600cm^{-1}, which correspond to the octahedral and tetrahedral sites of positive ions of $CuFe_2O_4$, respectively. Waldron *et. al.* [8] reported that the higher absorption band ~595 cm^{-1} corresponds to the intrinsic vibrations of tetrahedral complexes and the lower absorption band at ~447 cm^{-1} is attributed to the vibrations of octahedral complexes. The different values of absorption peaks for octahedral and tetrahedral complexes of $CuFe_2O_4$ crystals are due to the different values of $Fe^{3+} - O^{2-}$ distance for octahedral and tetrahedral sites. The band at ~3356$cm^{”1}$ corresponds to OH^{--} ions.

The other two peaks occurring at approximately 2922-2852 cm^{-1} were assigned to the anti-symmetric and symmetric CH_2-vibrations of the carbon chains, respectively. The absorption band at approximately 1457 cm^{-1} corresponds to the $-CH_2$ bending vibration. SEM micrographs of the synthesized $CuFe_2O_4$ nanocrystal are shown in Fig.4. It is evident by the SEM micrograph that the $CuFe_2O_4$ have almost uniform spherical structure morphology with a narrow size distribution of the particles. The size of the nanoparticles calculated using SEM micrograph were found to be in the range of 20-40nm, which is in good agreement with the particle size calculated using XRD data.

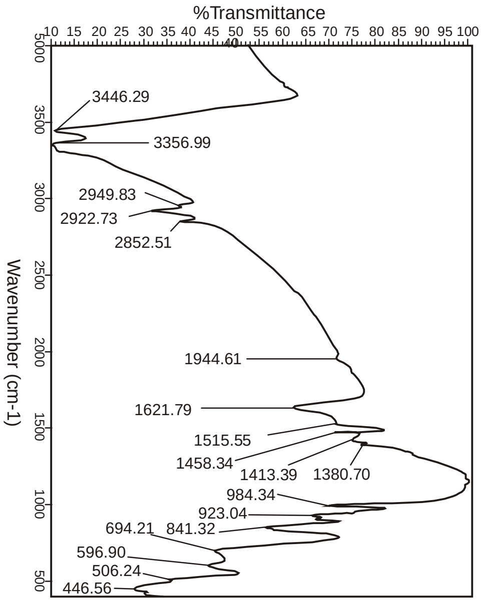

Fig.3. FTIR Spectra.

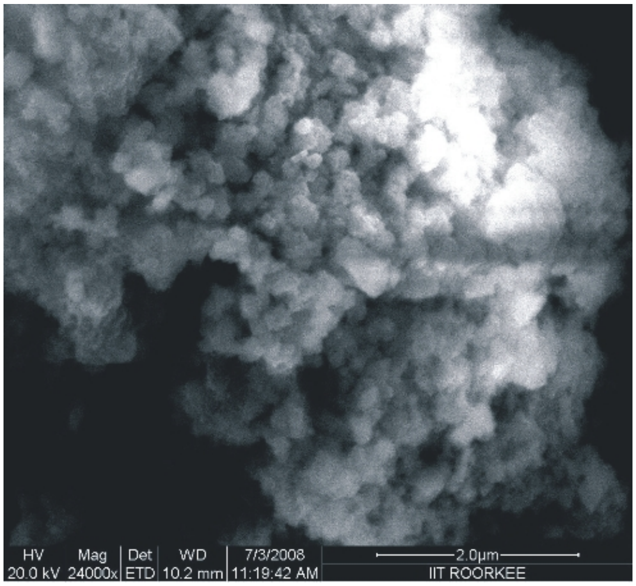

Fig.4. SEM image of $CuFe_2O_4$.

CONCLUSION

XRD pattern of the sample prepared by chemical co-precipitation method shows nanocrystalline nature of the sample. It has been observed that the material prepared is having the lattice constant of 8.398 Å. The average particle size resulting synthesized were observed to be 20nm, which is well supported by the SEM micrographs.

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