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Effect of p^H Value on Structural and Morphological Properties Synthesized by Hydrothermal Technique

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Abstract

The present work carried out as to synthesize copper ferrite nanoparticles (CF) at pH 13 and verify the structural and, morphological properties of copper ferrite (CF) nanoparticles. In this direction we synthesized the copper ferrite nanoparticles via hydrothermal technique. The X-Ray diffraction pattern revealed the formation of cubic single phase structure. In addition, we studied the morphological properties with Field Emission Scanning Electron microscopy (FESEM), Transmission Electron Microscopy (TEM). The particle size and grain size were be calculated.

Keywords: Nanoparticles; Structure; morphological Properties; Hydrothermal Technique.

1. INTRODUCTION

It has been an established fact that in nowadays, the nanotechnology has grown rapidly owing to the more advanced properties of nano-materials than their bulk counterparts. For instance, the nanoparticles have showed much attention towards the applications like humidity sensors, enhanced photo catalytic performance, electromagnetic interference shielding devices, transformer inductors, drug-delivery systems, magnetic resonance imaging (MRI), multilayer chip inductors (MLCIs) and transformer core devices [1-5]. Especially, the ferrite nano-materials have been found to be promising candidates for various industrial applications such as

telecommunication devices, microwave devices, data-storage devices, electromagnetic devices, microwave absorbers, magnetic recording, biosensors and power transformers owing to their high thermal stability, enhance electrical, optical and magnetic properties [1-5].

In general, the ferrites are of spinel structure with a general formula AB_2O_4, where 'A' and 'B' are the tetrahedral and octahedral cationic positions respectively. The copper ferrite is one of the materials of this family. The authors' have got the idea of changing the p^H value is13 and furthermore to investigate the structural, morphology properties. Moreover, the literature survey expressed that there are no findings like varying p^H and studying various properties. Therefore, an attempt has been put forth by the authors' to study the effect of p^H on the structural, morphology properties of CF nanoparticles.

2. EXPERIMENTAL PROCEDURE

To prepare $CuFe_2O_4$ nano-particles at p^H =13, we have taken $Cu(NO_3)_26H_2O$, Fe $(NO_3)_3$ $9H_2O$ (each of 99.8 % purity, Sigma-Aldrich) and NaOH are taken as precursors. as stated by the calculated stoichiometric ratio, the copper nitrate and iron nitrates are mixed in di-ionized water (ml) with 1:3 (Nitrates: water) ratio. And then to achieve p^H =13 we added NaOH (gm) slowly to the mixer of the nitrates and water and hence the p^H is found be 13 of the mixtures. Further, the combinations fetched into a 300 ml Teflon-lined steel autoclave. Furthermore these are deposited in a hot-air oven and reaction is made at 150°C for 8 hr. Afterwards the solution of cupper ferrite nano-particles separated from autoclave by washing with acetone and distilled water for more than six times till it reaches the p^H = 7. Then the compound is again dried at 60°C for 6 hr so as to remove water percentage. Ultimately, the samples are characterized using X-ray diffract meter, field emission scanning & transmission electron microscope, for structural, morphological, properties respectively. The step wise flow chart diagram for the synthesis of copper ferrite nanoparticles is shown in Fig.1.

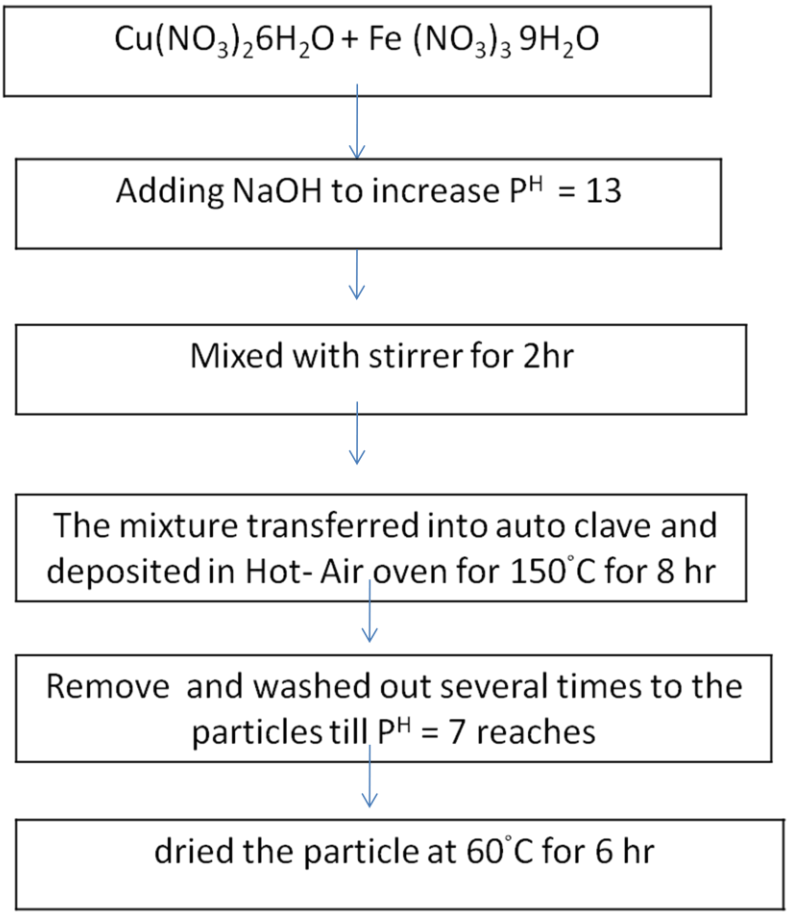

Fig.1. Flow chart for the synthesis of copper ferrite nanoparticles

3. RESULTS AND DISCUSSIONS

3.1 XRD Analysis

The X-ray diffraction patterns of CF nanoparticles with p^H is 13 were depicted in Fig.2. It indicates the variation of intensity (I) as a function of two-theta (2θ) angle. Obviously, it can be understood from the figure that the cubic phases with less intensities phases were identified for the p^H 13. This implies that hydrothermal reaction was not carried out successfully for that p^H values. The formed cubic (C) phases were in good consistent with the JCPDS: 25-0283 Cubic structures. Moreover, the cubic reflection planes (111), (220), (311), (222), (400), (331), (422), (511), (440), (620), (533) & (444) were indicated in the diffraction pattern. However, the cubic reflection planes expressed good apparent crystalline. In the JCPDS: 25-0283 the CF attained the cubic phases. The current investigation manifests that formation cubic phases

The average crystallite size 'D' is determined from average full width half maxima (FWHM) of all reflection peaks using Debye-Scherrer formula $D = k\lambda/\beta\cos\theta$ [1-9],

where β is FWHM, λ is wave length of $Cu_{K\alpha}$ source (0.15406 nm) and θ is diffraction angle. The average crystallite size (D) of all kinds of CF nanoparticles was found to be altering between ~17 to 60 nm. The microstrain $\varepsilon = \beta/4\tan\theta$ found to be 11.4 x 10^{-3} may be responsible for this (Table.1). Besides, the lattice constants (a = b = c) were calculated for the (311) cubic reflection planes using a standard relation: $a = d\,(h^2 + k^2 + l^2)^{1/2}$, where'd' is the interplanar distance and (hkl) are the miller indices [10-11]. The results attributed that the 'a' value were noticed to 0.8347 nm with in p^H 13.

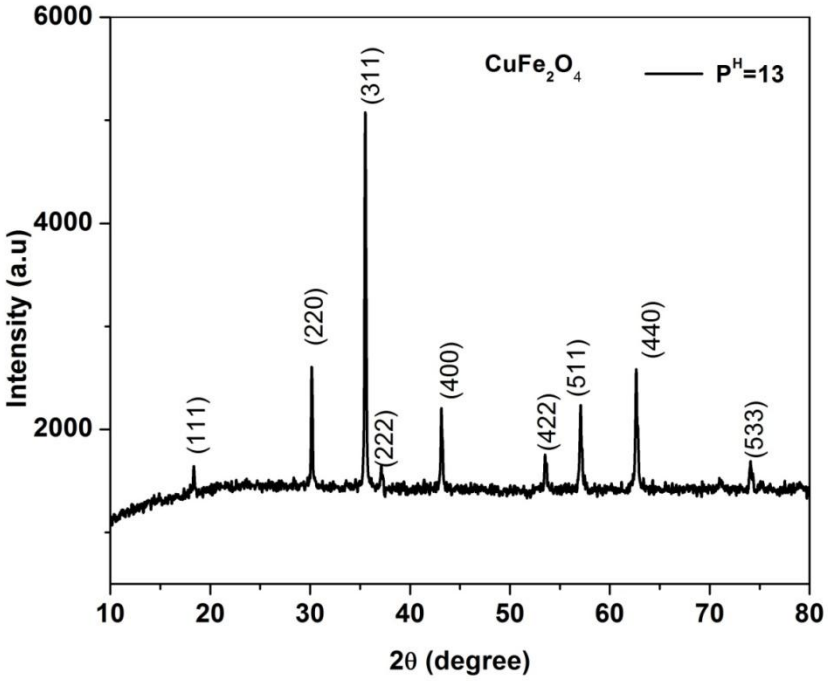

Fig.2. X-ray diffraction pattern of samples of copper ferrite nanoparticles at p^H=13

Williamson-Hall (W-H) plots (Fig.3) were drawn for βcosθ versus 4sinθ so as to calculate micro-strain (ε') and average crystallite size (D') with the help of following standard relation: $\beta\cos\theta = 0.9\lambda/4\varepsilon\sin\theta$ [5], where slope of straight line provides micro-strain while crystallite size is related to intercept value. Thus, this established a correlation between 'ε'' and 'D'' parameters. The achieved data is tabulated in Table.1. The results demonstrated that the numerical values of 'ε' and 'D' obtained using Scherrer method were almost in good agreement with the numerical values of 'ε'' 11.4x 10^{-3} and 'D'' 25 nm obtained using W-H plots. Therefore, one can alternatively find the developed micro strain as well as crystallite size using analysis of W-H plots.

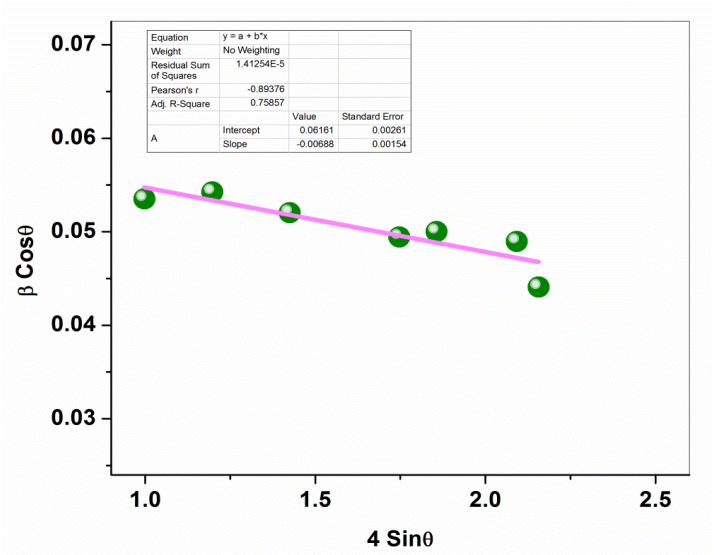

Fig.3. W-H plots of copper ferrite nanoparticles

Table.1 Data on structural and optical parameters of copper ferrites nanoparticles

x	13
a(Å)	8.347
D (nm)	25
strain (ε x10^3)	11.4
ρ_t (g/cm^3)	5.297
ρ_e (g/cm^3)	4.524
porosity (P)	0.146
G (nm)	132

3.2 Surface Morphology

The morphology of as fabricated copper ferrite nanoparticles of pH 13 was analyzed using FESEM. The FESEM photographs were shown in Fig.4. In the image shown, for CF nanoparticles with p^H = 13, almost Stone shape grains were to be observed. However, the grain size (Table.1) was measured using the linear intercept method: G = 3L/2MN, where L=the total test line length, M=the magnification, N=the total number of intercepts which the grain boundary makes with the line [12-19]. Moreover, the EDS provide the hidden elements and impurities in the samples.

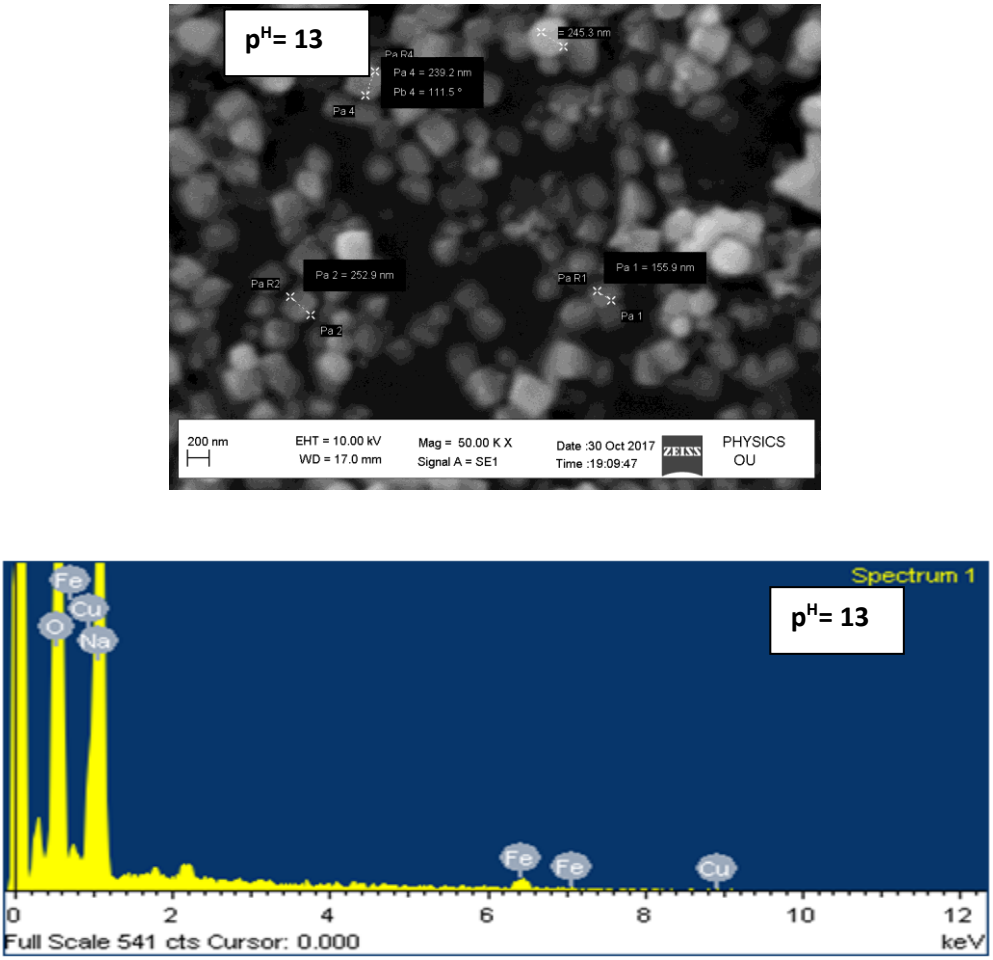

Fig.4. The FESEM and EDAX photograph of copper nano particle

Fig.5 depicts the HRTEM pictures of as prepared CF nanoparticles of 13 p^H. The particle size of samples was noticed to be altering between 23-56 nm. The CF nanoparticles revealed the irregular flat plates like particles. However, the magnetic interactions among the nanoparticles are responsible for the weak agglomeration identified in TEM pictures [4].

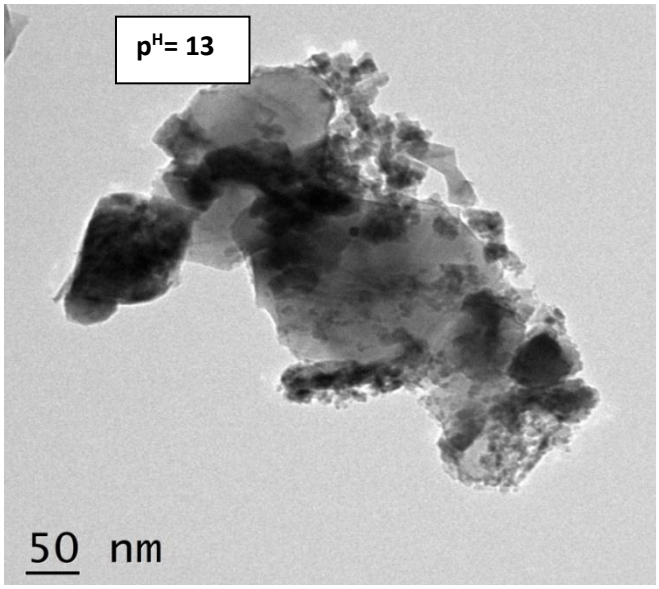

Fig. 5 TEM images of CF nanoparticles

4. CONCLUSIONS

The single (C) phase copper ferrite (CF) nanoparticles were synthesized via low temperature hydrothermal approach. Upon reaching the p^H value of 13 the cubic phases were appeared. The FESEM and HRTEM exhibited the formation almost homogeneous spherical shaped grains and particles respectively. The agglomeration phenomenon is responsible the huge variation between crystallite size and grain size.

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