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SYNTHESIS OF COPPER FERRITE NANOPARTICLES WITH DIFFERENT CAPPING AGENTS

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ABSTRACT

In this work, we report the effect of capping agents, ethylenediaminetetraacetic acid (EDTA) and NNN Cetyl Trimethylammonium Bromide (CTAB), on the structure, morphology and characteristic properties of $CuFe_2O_4$ nanoparticles. The samples were prepared by sol-gel citrate method. Characterization of as prepared samples was carried out using X-ray diffraction (XRD) patterns. The average nanocrystalline sizes calculated using Scherrer formula indicated 25-30 nm and 40-45 nm for PEG:$CuFe_2O_4$ and CTAB: $CuFe_2O_4$ respectively. The SEM image shows that the agglomeration of PEG:$CuFe_2O_4$ nanoparticle is relatively higher when compared to that of the CTAB: $CuFe_2O_4$ nanoparticles because of the effect of capping agent on modifying partical sizes.

Keywords: *Capping agents, Nanoparticles, $CuFe_2O_4$, XRD*

I. INTRODUCTION

Spinel type of $M^{2+}M_2^{3+}O_4$ structures has attracted much attention due to its chemical stability and various potential applications. In general, ferrites have a chemical formula $MFeO_4$ (M=Zn, Cu, Ni, Co etc.). Among the many ferrites $CuFe_2O_4$ has many advantages especially in gas sensors, catalyst, conduction properties, fuel cell, superior magnetic properties.[1,2]

The copper ferrite bulk belongs to the inverse spinel structure [3,4] with 8 Cu^{2+} ions occupying octahedral B-site while 16 Fe^{3+} ions occupy equally the tetrahedral (A-sites) and octahedral (B-sites) site of the unit cell [5], this configuration is shown by $(Fe^{3+})A[Cu^{2+}Fe^{3+}]BO_4^{2-}$.The most widely used methods for synthesis of ferrite nanoparticles, with range of crystallite size between 1 to 100 nm, are sol-gel, co-precipitation and solid state methods [6]. The changes in the properties of the nanoparticles are mainly attributed to crystallite size due to the fraction of atoms on the surface compared to the bulk [5].

Most of the physical or chemical properties showed by these nanoparticles are because of their crystalline nature of materials. Further growth in their size is due to agglomeration of these crystallites to form primary particles. If this growth of particles is not controlled, then due to Ostwald ripening and Vanderwaals interactions between particles, they may agglomerate and settled. To control the growth of nanoparticles, organic stabilizers (polymers) ethylenediaminetetraacetic acid (EDTA), Ethanol, NNN Cetyl Trimethylammonium Bromide (CTAB), etc. can be added during the synthesis for capping agent. [7]

In this work, we report the preparation of nanocrystalline spinel ferrites $CuFe_2O_4$ synthesized by sol-gel citrate method using two different capping agent, ethylenediaminetetraacetic acid (EDTA) and NNN Cetyl Trimethylammonium Bromide (CTAB). The structural and morphological properties were investigated by X-ray diffraction, FT-IR and scanning electron microscopy. The obtained results are discussed in terms of the effect of capping agents and grain size.

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II. MATERIALS AND METHODS

AR grade copper nitrate hexahydrate $Cu(NO_3)_2.6H_2O$, ferric nitrate nonahydratetes $(Fe(NO_3)_3\ 9H_2O$, citric acid, ethylene diaminetetraacetic acid (EDTA), NNN Cetyl Trimethylammonium Bromide (CTAB), liquor ammonia with high purity of 99.99% and used without further purification.

Ferrite Nanoparticles $CuFe_2O_4$ were synthesized by sol-gel citrate method using PEG and CTAB capping agents**.** The accurately weighed amount of metal ion precursors, citric acid and PEG in desired stoichiometric proportions were dissolved separately in minimum quantity of distilled water. Citric acid and nitrate ions act as fuel and source of oxygen, respectively. The molar ratio of metal ion precursors to citric acid was kept 1:1. The individual solutions were then mixed together with constant stirring at pH = 7 by adding liquor ammonia . Stirring was done on magnetic stirrer for 2 hour at 80^0C to obtain homogeneous solution. Gel was obtained by constant stirring and heating the solution at 120^0C temperature in pressure vessel. The powder was finally calcinied at temperature 650^0C for 6 hrs in a muffle furnace. The same procedure was followed to prepare $CuFe_2O_4$ nanoparticles using the CTAB capping agent. $CuFe_2O_4$ nanoparticles prepared with the capping agents PEG and CTAB are named as (a) PEG: $CuFe_2O_4$and (b) CTAB: $CuFe_2O_4$and used in the discussion.

III. STRUCTURAL AND MORPHOLOGY STUDY

Structural analysis of synthesized powdered samples was carried out using X-ray diffractometer (Philips 3710, PAN alytical) using Cu-Kα radiation with wavelength (λ = 1.5406 ˚A), and the surface morphology of the samples was investigated with a Scanning Electron Microscope (JEOL-JXA-8100 SEM).

IV. RESULT & DISCUSSION

The synthesized ferrite $CuFe_2O_4$ was calcinied at an operating temperatures 650^0C for 6 hrs to study the effect of calcination temperature on particle size. The powder XRD patterns of PEG: $CuFe_2O_4$ and CTAB: $CuFe_2O_4$ are shown in Figure 1. The XRD peaks of the prepared samples were indexed by comparing with the JCPDS card No: [034-0425] [8] which confirms the formation of $CuFe_2O_4$. XRD pattern shows that synthesized nanoparticles exhibit mixed phase of CuO and $CuFe_2O_4$ where the $CuFe_2O_4$ belongs to face centered regular spinel cubic structure.

For all samples 2θ value for the most intense peak at (211) plane ranges at 35.50^o, a characteristic of cubic spinel ferrite. The most intense peak at (211) is used to determine the average crystallite size of nanoparticles. There is no impurity peak found indicating the formation of pure sample. The average crystallite size of the samples was determined by using the Debye-Scherer's formula. $D_{hkl} = 0.9\ \lambda\ /\ \beta\ \cos\ \theta$ [9] where, D_{hkl} = crystallite size, λ is wavelength of the X-ray radiation, β is the full width at half maximum (FWHM) of the most intense diffraction peak and θ is Bragg's angle. The value of crystallite size calculated for PEG: $CuFe_2O_4$ and CTAB: $CuFe_2O_4$ is 25-30 nm and 40-45 nm respectively.

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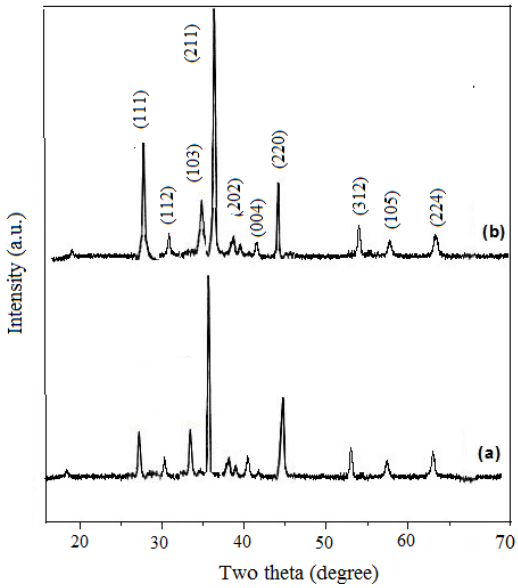

Figure 1. XRD patterns of (a) PEG: $CuFe_2O_4$ and (b) CTAB: $CuFe_2O_4$ nanoparticles calcined at 650 ^{0}C

FTIR spectroscopy was used to determine the structure and investigation of the chemical species present. As per figure 2, showed, broad band peaks at 430 and 470 cm^{-1} are related to metal-oxide stretching vibrations of the tetrahedral and octahedral sites in crystalline structures, respectively. [10] The peak at 1546 cm^{-1} and broad band peak at 3440 cm-1 are assigned to the bending and stretching vibration of O–H related to the adsorbed water.

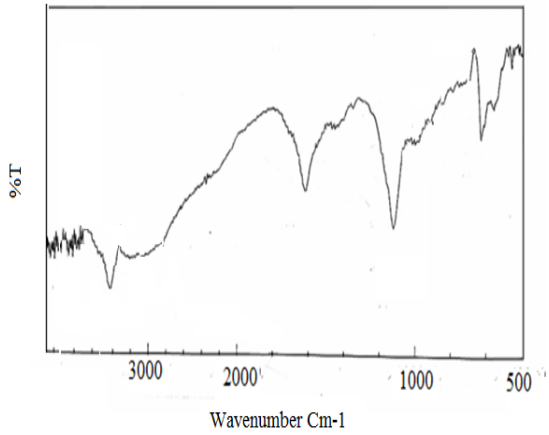

Figure 2. FT-IR spectrum of PEG: $CuFe_2O_4$ nanoparticles calcined at 650 ^{0}C.

The morphology studies of the prepared $CuFe_2O_4$ nanoparticles was investigated by SEM. The SEM image of (a) PEG: $CuFe_2O_4$ and (b) CTAB: $CuFe_2O_4$ nanoparticles is shown in Figures.3a,b respectively. The SEM images shows agglomerated of nanoparticles. The small size of nanoparticles leads to high agglomeration because of its high surface energy. The SEM image shows that the agglomeration of PEG:$CuFe_2O_4$ nanoparticle is relatively higher when compared to that of the CTAB: $CuFe_2O_4$ nanoparticles because of the effect of capping agent on modifying partical sizes.

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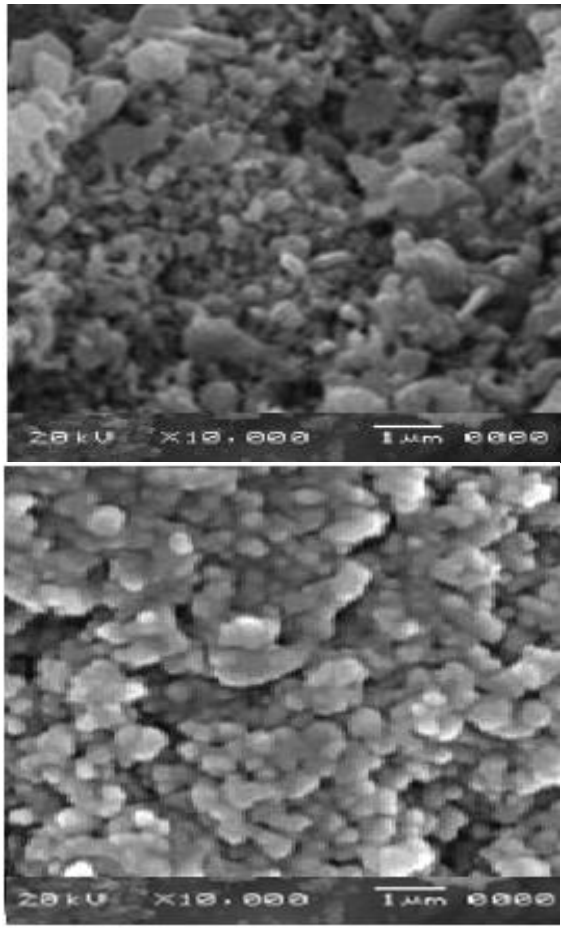

Figure 3. SEM images of (a) PEG: $CuFe_2O_4$ and (b) CTAB: $CuFe_2O_4$ nanoparticles calcined at 650 ^0C

V. CONCLUSIONS

It can be concluded that pure $CuFe_2O_4$ nanoparticles have been prepared by using conventional and simple sol-gel citrate method using two different capping agent, ethylenediaminetetraacetic acid (EDTA) and NNN Cetyl Trimethylammonium Bromide (CTAB). XRD patterns confirmed that the PEG:$CuFe_2O_4$ and CTAB: $CuFe_2O_4$ nanoparticles were synthesized having a size 25-30 nm and 40-45 nm. FT-IR result showed that nanoparticles were completely calcined after the heat treatments. The SEM image shows that the agglomeration of PEG:$CuFe_2O_4$ nanoparticle is relatively higher when compared to that of the CTAB: $CuFe_2O_4$ nanoparticles because of the effect of capping agent on modifying partical sizes. Further, low cost, simplicity, short time of production, purity and homogeneity of final product are some of its advantages.

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