One pot facile synthesis of nanosized Cu0.5Zn0.5Fe2O4 particles by direct precipitation method

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ABSTRACT: Nanosized Copper-zinc ferrites Cu0.5Zn0.5Fe2O4 have been synthesized by aqueous precipitation method and characterized by using XRD (X-ray diffraction), TGA/DTA (Thermo gravimetric analysis), SEM (Scanning Electron Microscopy)/ TEM (Transmission Electron Microscopy) and magnetic measurements by using VSM (vibrating sample magnetometer). XRD studies confirm the formation of cubic spinel structure. SEM (scanning electron microscopy)/ TEM (transmission electron microscopy) was used to characterize the microstructure of the ferrite samples. A homogeneous and fine grain microstructure was found. Magnetic measurements show that Ni0.5Cu0.5Fe2O4 is super paramagnetic in nature at room temperature and hence used in magnetic device. The particle size of synthesized Cu0.5Zn0.5Fe2O4 varied from 18nm to 68nm which is good agreement of the theoretically predicted size of nanomaterials. The method is easier more effective and convenient in comparison to the known methods of the synthesis Cu0.5Zn0.5Fe2O4 Nano materials like combustion synthesis, thermal cracking and conventional ceramic methods.

Key Words: Nanosized, Spinel, Ferrites, Super Para magnetism, copper – zinc Ferrite (Cu0.5Zn0.5Fe2O4)

1. Introduction:

Cu0.5Zn0.5Fe2O4 is a spinel ferrite. The general formula of the spinel ferrite is MeFe2O4 where Me usually represents one or, in mixed Ferrites more than one of the divalent transition metals Mn, Fe, Cu, Ni, Zn, Ca or Mg and Cd. Other combinations of equivalent valency are possible, and it is also possible to replace some or all the trivalent iron ions with other trivalent metal ions [16]. Ferrites are the fundamental functional materials of electronic industry. Ferrites can be cast into complex shapes and can be ground and will take fine finish. Ferrites are ceramic materials. Ferrites may be defined as magnetic materials composed of oxide containing ferric ions as the main constituent. This term is often restricted to materials which have cubic crystal structure of spinel but now a day it is also applied to magnetic oxides. Ferrous ferrites are an example of naturally occurring ferrite. Magnetic properties in ferrites arise from interactions between metallic ions occupying particular positions relative to oxygen ion in the crystal structure of the oxide. In majority of the present day magnetically soft ferrite the crystal structure is cubic and has the form of mineral spinel. Cu0.5Zn0.5Fe2O4 is developed for a wider range of applications where high permeability is the main requirement. The ferrite core memory was the basis of the IBM 360 computer which is standard for industry [6–15]. The application started in the field of telephony transmission operating in large frequency range from about 40 KHz. Most important use of ferrite for video recorder [15–21]. Today ferrites are used as noise filters in power lines of electronic equipments[22]. Ferrites are easy to manufacture, low costs ,small volume, high efficiency and with greater uniformity have applications in ceramic magnet as medical treatment, filter inductors , magnetic amplifiers, transformers, antenna cores, magnetic memories and fly back transformers.

2. Methods and materials

2.1 Chemicals:

All chemicals used in the experiment are analytic reagent grade. Ferric nitrate Fe(NO3)3, Copper Nitrate Cu (NO3)2 , Zn(NO3)2 and liquor ammonia were purchased from Merck, India. Deionized water was used throughout the experiment.

2.2 Synthesis of Cu 0.5Zn0.5Fe2O4 :

200 ml of 0.5 M solution of Fe(NO3)3 was mixed with 200 ml of 0.5M solution of Zn(NO3)2 and 200 ml of 0.5M solution of Cu(NO3)2 then aqueous ammonia was added drop wise with constant stirring until the pH of the solution reached to 10. The precipitate thus obtained were filtered on Buckner funnel and washed several times with distilled water. The precipitate were dried in oven at 70°C for 24 hrs and were calcined at 600°C in a muffle furnace for 5 hours. Obtained material was ground and sieved through 100 mesh size sieve.
2.3 Equipments:

An X-ray measurement was carried out using X-ray diffractometer system Philips PW 11/90, with nickel filtered CuKα (1 = 1.5405 Å).

The crystalline size of Zinc ferrite was calculated using Scherrer equation.

\[ t = \frac{K\lambda}{B \cos \theta} \]

Where \( t \) is the average crystallite size of the phase under investigation, \( K \) is the Scherrer constant (0.89), \( \lambda \) is the wave length of X-ray beam used, \( B \) is the full-with half maximum (FWHM) of diffraction (in radians) and \( \theta \) is the Bragg’s angle.

Transmission electron micrograph (TEM) were recorded on Hitachi H7500. The samples were dispersed in ethanol and then treated ultrasonically in order dispersed individual particles over a gold grid. The surface morphology of Ni₉₅Cu₀₅Fe₂O₄ prepared by precipitation method was investigated by using scanning Electron Microscope Quanta 200 FEG (FEI Netherlands).

The magnetic properties of the solid was measured at room temperature using a Vibrating sample Magnetometer Model 155.

3. Results and discussions:

3.1 X-ray studies:

The X-ray diffraction pattern of synthesized Cu₉₅Zn₀₅Fe₂O₄ Nano particles is depicted in Fig. (1). X-ray diffraction pattern of Cu₉₅Zn₀₅Fe₂O₄ pure indicated that Cu-Zn ferrite in the form of Cu₀₅Zn₀₅Fe₂O₄ (Fig. 1). In X-ray diffraction, some prominent peaks were considered and corresponding d-values were compared with the standard i.e. JCPDS (Joint Committee on Powder Diffraction Standards) (File card 86.). Table 1. X-ray diffraction shows that metal oxide is pure Cu₀₅Zn₀₅Fe₂O₄ having cubic spinal structure. Table 1 gave a verification of well defined crystalline phase of spinal structure of Cu-Zn ferrite. The average crystallite size was determined from the broadening of the most intense peaks using Debye Scherer equation (Cullity 2001) and values shown in Table 1 and it also supports the SEM/TEM observations. The crystallite size was found within 18-68 nm. The powder x-ray patterns confirm the single-phase spinal structure for synthesized material. Thickness of the crystal has been calculated using Debye Scherrer’s formula and it support the TEM observations.

3.2 Thermal Analysis:

Thermal analysis includes a group of techniques in which a physical property of a substance is measured as a function of temperature or time while the substance is subjected to a controlled temperature programme. The analysis involves thermogravity (TG), differential thermal analysis (DTA) and derivative Thermogravimetry (DTG). Thermal Gravimetric studies of the calcined oxides prepared were done between a temperature range of 10-1000°C under N₂ atmosphere. The TGA/DTA curves of the oxides are shown in Fig. (2). The maximum total weight loss observed for Nickel oxide and their corresponding temperature is summarized in Table (2). Results showed that in the synthesized oxides shows some weight loss and ferrite undergoing decomposition, dehydration or any physical change. In DTA curve also, there is exothermic peak which shows phase transition, solid state reach on any chemical reaction occurred during heating treatment.

3.3 SEM/TEM studies:

SEM studies were carried out to study the morphology of the sample Fig. (3a,b) shows the SEM micrographs of Cu₀₅Zn₀₅Fe₂O₄. The micrographs of Cu₀₅Zn₀₅Fe₂O₄ (Fig. 3a,b) displayed spherical particles with high agglomeration.

TEM studies were carried out to find the exact size of the synthesized Cu₀₅Zn₀₅Fe₂O₄ nanoparticles Fig. (4a,b,c,d,e,f,g) shows the TEM images of the synthesized Cu₀₅Zn₀₅Fe₂O₄ nanoparticles. It shows that the size of the obtained Nano particles is in the range 18-68 nm. Most of the particles are in the range 21-50 nm. TEM images indicate that Cu₀₅Zn₀₅Fe₂O₄ samples were all spherical particles with uniform grain size distribution.
3.4 Magnetic measurements:
Magnetic properties of nanometer sized particles have attracted considerable attention in recent years because of their unique properties. The size of the magnetic particles decreases below a critical length. Domain formation was no longer energetically favored and the particle existed as a single domain. Magnetic nanoparticle have aroused increasing interest among researchers of various fields due to their extensive applications such as in information storage system, medical diagnostics, ferrofluid technology etc. This is mainly because of the properties of nanoparticles differ from those of the corresponding bulk material. The magnetic measurements of Cu$_{0.5}$Zn$_{0.5}$Fe$_2$O$_4$ was carried out at room temperature and it has been shown that the magnetic measurements shows that the prepared Cu$_{0.5}$Zn$_{0.5}$Fe$_2$O$_4$ nanoparticles posses good super paramagnetic behavior at room temperature (300K) with saturation magnetization MS value 51 (fig. 5). Previously reported values of MS For Cu$_{0.5}$Zn$_{0.5}$Fe$_2$O$_4$ Nanoparticles prepared by various methods has been reported in Table (2). The value of MS ranging from 15-90 emu/g shows that Ms strongly depends on the methods of synthesis.

This MS value at room temperature is good and comparable with methods of synthesis as thermal decomposition method (MS value 43 at 300 K and MS value 68.5 emu-1 at 10k) ball milling (MS value 20.7 in at 4.2k) and other co-precipitation routes which shows a maximum MS 47.8 at 4.2 k (fig.5). Magnetic Hysteresis curve clearly indicate the soft nature of the prepared sample saturation magnetization ms value increase with time.

4. Conclusion:
Cu$_{0.5}$Zn$_{0.5}$Fe$_2$O$_4$ nanoparticle with cubic spinel structure are synthesized successfully by aqueous precipitation method. From SEM/TEM studies it is found that particles have average size 18-68nm. Magnetic measurements shows that Cu$_{0.5}$Zn$_{0.5}$Fe$_2$O$_4$ super paramagnetic in nature having saturation magnetization (MS) value 51 emu/g. This method is beneficial over the existing methods of synthesis of Nano particles because other methods require expensive materials, highly skilled labor and specialized instrumentation. Therefore, the proposed precipitation method is cheaper, easier, very promising and may have extensive applications.

Table-1 X-RAY DIFFRACTION DATA FOR Cu$_{0.5}$Zn$_{0.5}$Fe$_2$O$_4$

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<th>S. No.</th>
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<th>d=λ / 2sinθ (Reported)</th>
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Table: 2: OBSERVATIONS OF WEIGHT LOSS FOR Cu$_{0.5}$Zn$_{0.5}$Fe$_2$O AT CORRESPONDING TEMPERATURE RANGE

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<th>Sr.No.</th>
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TABLE 3: PRACTICAL SIZE OF SYNTHESIZED Cu0.5Zn0.5Fe2O4 AT DIFFERENT SCALES

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<th>S.No.</th>
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<th>Scale (20nm)</th>
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Figure 1: X-Ray diffraction spectra of Cu0.5Zn0.5Fe2O4 Particles
Figure 2: TGA-DTA of Cu$_{0.5}$Zn$_{0.5}$Fe$_2$O$_4$ Particles

Figure 3(a): SEM micrographs of Cu$_{0.5}$Zn$_{0.5}$Fe$_2$O$_4$ Particles
Figure 3(b): SEM micrographs of Cu0.5Zn0.5Fe2O4 Particles
Fig. 4 (a,b,c,d,e,f,g,h) TEM micrographs of Cu$_{0.5}$Zn$_{0.5}$Fe$_2$O$_4$ Particles
Figure 5: Magnetic measurement of synthesized Cu$_{0.5}$Zn$_{0.5}$Fe$_2$O$_4$ particles

References
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