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RESEARCH ARTICLE

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SYNTHESIS AND CHARACTERIZATION OF Mg-Cu-Zn FERRITES NANOPARTICLE BY CO-PRECIPITATION METHOD OXALATE AS PRECURSOR

M. SATYAVANI
Lecturer in Physics
D.N.R.College, Bhimavaram, Andhra Pradesh, India

ABSTRACT
Ferrite nanoparticles of basic composition $Mg_{0.40,}Cu_{0.25,}Zn_{0.50}Fe_2O_4$ were synthesized through co-precipitation wet synthetic method by using oxalate as precursor at 90°C then filtered and washed with distilled water. After drying, heat treatment was carried out for 5 hours at 750°C and the resulting compounds were characterized for structural properties using X-ray diffraction [XRD], scanning electron microscopy, transmission electron microscopy, and Fourier transform infrared spectroscopy [FT-IR]. The XRD result shows that all prepared samples crystallite size are in the range of 30 nm to 60 nm and lattice constant in the range of 8.20 to 8.40 nm. Fourier Transform Infra Red Spectroscopy results clearly indicate the Mg-Cu-Zn nano Ferrite are synthesized. Differential scanning calorimeter graphs shows the phase formation of all the samples.

Keywords: Mg-Cu-Zn ferrites, Oxalate, Co precipitation, Characterization

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INTRODUCTION

Ferrites or ferromagnetic oxides (also known as ceramics containing compounds of iron) are dark brown or gray in appearance and very hard and brittle in physical character. They are prepared by heat-treating various transition metal oxides or alkaline earth oxides with the ferric oxides. Many ferrites are spinels with the formula AB2O4, where A and B represent various metal cations, usually including iron Fe. Spinel ferrites usually adopt a crystal motif consisting of cubic close-packed (FCC) oxides (O^{2-}) with A cations occupying one eighth of the tetrahedral holes and B cations occupying half of the octahedral holes. Spinel ferrites are technologically very important magnetic materials having potential applications and have attracted intense interest in both the fundamental and the applied research points of view [1]. The last two decades have seen a remarkable progress in the synthesis of spinel ferrites nanocrystals, aiming at a better material with excellent chemical stability, low magnetic coercivity, moderate saturation magnetization, high permeability, high electrical resistivity and low eddy current [2]. The ferrite powders are prepared by the high temperature solid state reaction, sol –gel method, ball milling and co-precipitation method [3-6]. The synthesis of ferrites can be carried out using different methods but the low temperature synthesis and molecular level mixing is reported to be useful in obtaining desired magnetic properties and the reaction kinematics in a chemical process dependent on the temperature at which it is carried out.

Polycrystalline ferrite materials have wide application range in the field of electronic and communication industries due to their interesting

electrical and magnetic properties. The present study reports on the synthesis of Mg-Cu-Zn ferrite powders by oxalate co-precipitation method. In the present communication the results regarding IR absorption spectral analysis, SEM, TEM and XRD of Mg-Cu-Zn ferrites are discussed. This technique involves the formation of ferrites at much lower temperature and in less time than possible by the conventional ceramic method.

Materials and methods

Experimental

Synthesis of Mg-Cu-Zn ferrites were prepared by the oxalate co precipitation method. All used reagents were of AR grade. The starting materials were weighed according to the formula $Mg_{0.40,}Cu_{0.25,}Zn_{0.50}Fe_2O_4$ [7]. Suitable $(NH_4)_2C_2O_4{\cdot}H_2O$ and $NH_3{\cdot}H_2O$ were initially added into 0.1 mol/L FeCl3·6H2O solution under constant magnetic stirring to remain pH value of the solution at about 4. Then 0.9 mol/L $MgSO_4{\cdot}H_2O$, $CuSO_4.5H_2O$and 0.2 mol/L $ZnSO_4{\cdot}7H_2O$ were introduced to the above solution. The $(NH_4)2C_2O_4{\cdot}H_2O$ and $NH_3{\cdot}H_2O$ were re-added into the solution till its pH value of 8.0 At last, NaOH was added to the solution until its pH value of 14. Then the solution was refluxed for 5h. The obtained precipitated product was washed with distilled water until a clear solution, and then dried at 100˚C for 5 h.

The process of precipitation can be explained by following chemical reaction

$MgSO_4 + 2H_2O + C_2O_4$------> $Mg\ C_2O_4H_2O + SO_4$

$Zn\ SO_4 + 2H_2O + C_2O_4$------> $Zn\ C_2O_4H_2O + SO_4$

$Cu\ SO_4 + 2H_2O + C_2O_4$------> $Cu\ C_2O_4H_2O + SO_4$

$Fe\ SO_4 + 2H_2O + C_2O_4$ ------> $FeC_2O_42H_2O + SO_4$

The precipitate was filtered through whatman filter paper No. 42. The filtrate was washed with double distilled water (pH=7.1) to remove unreacted chemicals. The residue was checked for the absence of sulphates using $BaCl_2$ test. The powder was finally sintered at 750°C for 5h followed by slow cooling the furnace.

The X-ray powder diffraction (XRD) patterns were obtained at room temperature by using philps PW-3710 X-ray powder diffracto meter. The diffraction patterns were recorded at steps size of 0.02° in angular range of 20°-100°. The crystallite size was calculated by Scherrer formula. The scanning electron microscopy was carried by analyze the microstructure of fractured surface of the pellets using the SEM JEOL-JSM 6360 MODEL, JAPAN. Infrared absorption spectra of powered samples were record in the range of 350-800 cm^{-1} using perkin-Elmer FT-IR spectrum one spectrometer by KBr pellet technique. The thermal decomposition of the as-synthesized powder was investigated by means of a differential thermal analyser (DTA, Linseis L62 thermal analyzer) at a heating rate of 10°C / min in air.

Results and discussions

The DTA curve of the prepared ferrite powder given in Figure 1. The large exothermic peak is observed in the range of 390-420°C indicates the decomposition of the oxalate complex during process. A peak at 530°C corresponds to endothermic peak in the solid state reaction of the resulting oxides [8].

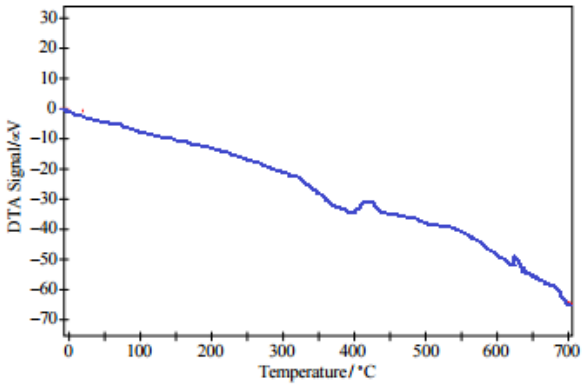

Figure 1: DTA curve of the as-ferrite Powder.

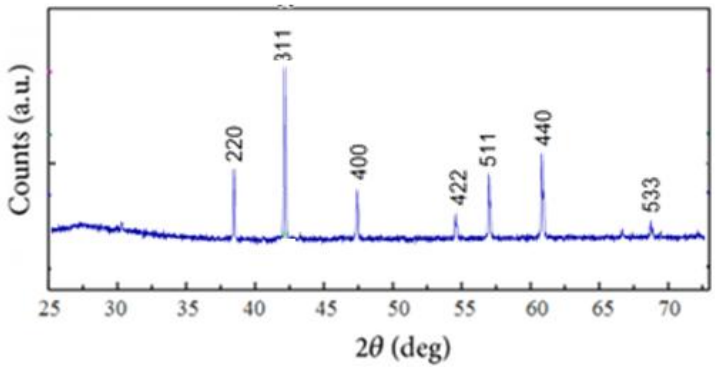

Figure 2. XRD pattern of $Mg_{0.40,}Cu_{0.25,}Zn_{0.50}Fe_2O_4$ nanoparticles

Figure 2 shows the XRD diffraction patterns of the $Mg_{0.40,}Cu_{0.25,}Zn_{0.50}Fe_2O_4$ nanoparticles at 353 K and at calcination temperatures at 750°C. The diffraction peaks show the reflection planes (220), (311), (400), (422), (511), (440) and (533) which are consistent with the standard powder diffraction reported from XRD library code (00-052-0279) and no other metal oxides could be identified. It can be said that formed sample is in the spinal phase with a face centered cubic structure (fcc). The particle size of the ferrite nanoparticles has been estimated from the XRD plane (311) by the Scherrer's formula: $d = 0.9\ \lambda/\beta \cos\theta$, where d is the average particle size in nm, β is the FWHM of the intensity measured in radians, λ is the X-ray wavelength and θ is the Bragg angle. The average crystallite size are in the range of 30 nm to 60 nm and lattice constant in the range of 8.20 to 8.40 nm

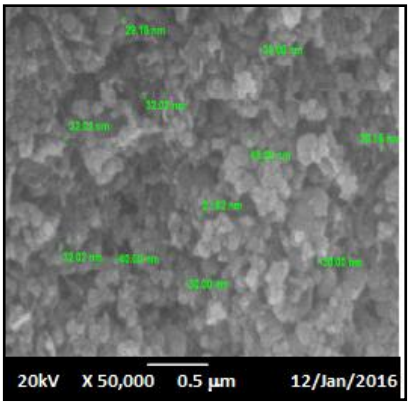

Figure 3: SEM Image of $Mg_{0.40,}Cu_{0.25,}Zn_{0.50}Fe_2O_4$ nanoparticles

The scanning electron microscopy studies were undertaken for the samples of $Mg_{0.40,}$ $Cu_{0.25,}$ $Zn_{0.50,}$ Fe_2O_4 nanoparticles and image is shown in Figure 3. It is evident from the SEM micrographs that these samples have uniform, almost spherical structural, morphology with a narrow size distribution of the particles. The particle size of these nano ferrites are in the range from 20 nm to 40 nm.

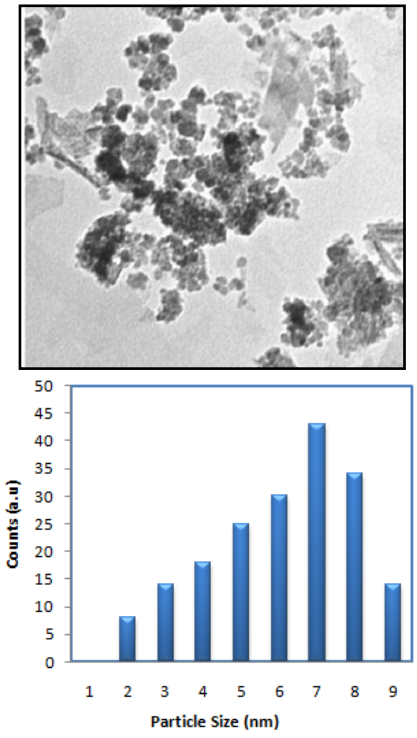

Figure 4: TEM micrograph for sample $Mg_{0.40,}Cu_{0.25,}Zn_{0.50}Fe_2O_4$ nanoparticle (above) and its particle size distribution (below)

Transmission electron micrograph TEM was performed for the prepared sample and it is represented in Figure 4(a). The micrograph reveals that the obtained particles are spherical in shape and have a dominant value of diameter of about 5 nm. The particle size distribution that is obtained

from TEM micrograph is represented by a histogram as shown in Figure 4 (below).

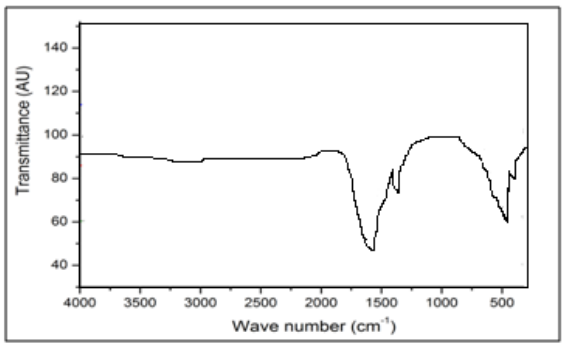

Figure 5: FTIR Spectra of $Mg_{0.40,}Cu_{0.25,}Zn_{0.50}Fe_2O_4$ nanoparticle sample

The FT-IR spectra of $Mg_{0.40,}Cu_{0.25,}Zn_{0.50}Fe_2O_4$ nano-ferrite sample is shown in fig.5. The presence of the bands in the range 375-600 cm-1 in the spectra confirms the formation of spinel phase [9-10]. Some additional bands around 3400-3200 cm^{-1}, 1200-1500 cm^{-1}, 1500-1700$cm^{-1}$and 2100-2400^{-1}are also present in the FT-IR spectra of the samples. These bands correspond to the stretching and bending modes of –OH group, C-H bond bending in plane mode, N-H bond in bending mode and C≡C bond in stretching mode respectively. The FTIR spectra are found to exhibit two major bands in the range 375-600 cm^{-1}. The high frequency band (*v*1) is in the range 560-580 cm-1 and the lower frequency band (*v*2) is in the range 390 - 410 cm^{-1}. These bands are common characteristics of spinel structure. The vibration of unit cell of the cubic spinel can be constructed in the tetrahedral (A) site and octahedral (B) site. The absorption band (v1) is caused by stretching vibrations of the tetrahedral metal-oxygen bond and absorption band (v2) is caused by the metal-oxygen vibrations in octahedral sites [11]. The change in band position is expected because of the difference in the Fe3+– O2- distances for tetrahedral and octahedral complexes. It is found that Fe–O distance of A-site (1.89 Å) is smaller than that of the B-site (1.99 Å).This can be interpreted by more covalent bonding of Fe3+ ions at A-sites than B-sites.

Initial permeability

It's known that the permeability of polycrystalline ferrite can be described as the superposition of domain wall and pin rotation components. At small grain sizes, the grain become monodomain and the reversal of magnetization can only occur through spin rotation.

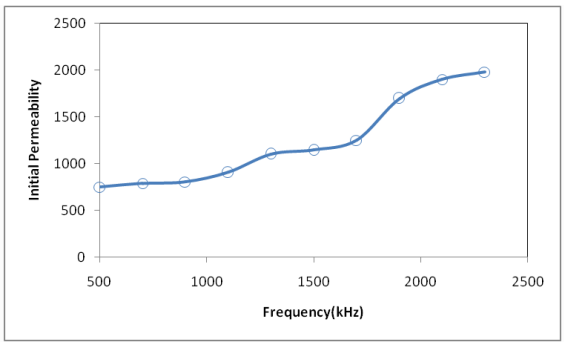

Figure 6: Initial permeability versus frequency.

Figure (5) shows the variation of the initial permeability with frequency in the region 10-2000 kHz. We connect the augmentation of the initial permeability near 2 MHz to the relaxation process in the RF region

Conclusions

The investigation in the present work indicates that the sol-gel method is efficient for synthesizing $Mg_{0.40,}Cu_{0.25,}Zn_{0.50}Fe_2O_4$ nanoparticles possessing unique spinel structure. It's regarded that the fine particle morphology of the powder synthesized by this method is responsible for its higher sintering activity. The very fact that single phase ferrite could be obtained directly by citrate precursor without any additional heat-treatments above 750^{o}C is a significant achievement considering the variety of applications of the $Mg_{0.40,}Cu_{0.25,}Zn_{0.50}Fe_2O_4$. The highly active powders could be sintered at relatively low temperatures to obtain highly dense and homogeneous polycrystalline ferrites for high frequency applications.

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