International Journal of Scientific Research in Physics and Applied Sciences **Research Paper**
Vol.7, Issue.2, pp.71-74, April (2019) **E-ISSN:** 2348-3423
DOI: https://doi.org/10.26438/ijsrpas/v7i2.7174

Green synthesis of $MgFe_2O_4$ nanoparticles using Albumen as Fuel and their Physico-Chemical Properties

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Available online at: www.isroset.org

Received: 12/Apr/2019, Accepted: 20/Apr/2019, Online: 30/Apr/2019

Abstract— This research article reports the green synthesis of magnesium ferrite nanoparticle via auto-combustion using albumen as fuel. The synthesized nanoparticles are confirmed to process single phase and spinel structure with the help of powder X - ray diffraction (PXRD) and Fourier Transform Infrared Spectroscopy (FTIR). Which also determines the functional groups present in the nanoparticles. EDAX results provide the percentage composition of the elements in the synthesized sample. The Field Emission Scanning Microscope (FESEM) reveals the agglomerated nature of ferrite nanoparticles. Magnetic moment and retentivity were obtained using Vibrating Sample Magnetometer (VSM). Dielectric properties of the as prepared samples were measured by two-probe method for various frequencies ranging from 100Hz-1MHz.

Keywords— ferrites; albumen; PXRD; FTIR; FESEM; VSM; dielectric; retentivity; coercivity.

I. INTRODUCTION

Magnetic nanomaterials have been investigated intensively in recent years as they possess unique magnetic, electrical and optical properties [1]. Spinel ferrite nanoparticles are significant among the magnetic nanoparticles due to their thermal and chemical firmness in addition to the above characteristics. A typical spinal ferrite is formulized as MFe_2O_4 where M is a metal or a group of metallic elements with two dissimilar valences. The M^{2+} and Fe^{3+} cations will be distributed into tetrahedral and octahedral crystal sites of the spinel structure [2, 3]. In this current work egg white (albumen) has been used along with nitrates of iron and magnesium to produce $MgFe_2O_4$.

This method has been chosen after an in-depth study of solid state reactions. Nanoparticles can be synthesized by physical, chemical, mechanical and thermal methods using techniques like co-precipitation, sol-gel, combustion, ball milling etc. Synthesis of nanomaterials via green synthesis is superior to various other techniques as it is clean, eco-friendly with low reaction temperature and free from undesirable harmful by-products. Green synthesis of metal nanoparticles using plant extracts, animal byproducts and organisms such as bacteria and fungi have been rigorously adopted [4]. The egg white enriched with albumen was first time reported by Santi Maensiri et al for preparing transition metal substituted ferrites [5]. This technique is adapted in the present work to synthesize magnesium ferrite nanoparticles. The magnetic, electrical, optical, morphological and other properties of nanoparticles have been analyzed using different tools such as X - ray diffractometer, scanning election microscope, Vibrating sample magnetometer, Fourier transfer Infrared spectroscope etc.

Section I gives the introduction to ferrites and their methods of synthesis. Section II is a detailed account of the experimental procedure carried out for the synthesis of $MgFe_2O_4$ and the characterization techniques adopted. Section III presents and analyses based on the obtained results along with interpretations of suitable images and graphs. Section IV gives the conclusion of the research work with future directions.

II. EXPERIMENTAL PROCEDURE

Synthesis

Magnesium ferrite magnetic nanoparticles were prepared using ferric nitrate nonahydrate and magnesium nitrate hexahydrate of high chemical purity along with freshly prepared egg white. Egg white rich in albumen protein are recognized for their foaming and emulsifying features and it is easily soluble in water which makes it combine with metal ions easily. Egg white also serves as binder cum gel for shaping materials [6]. Egg White and double distilled water were mixed in 3:1 ratio and stirred vigorously at room temperature until a homogeneous solution was formed. Mg $(NO_3)_2$.$6H_2O$ and Fe $(NO_3)_3$.$9H_2O$ were mixed in 1:2 mole ratio of Magnesium and Iron and slowly added to the homogenous egg white solution, with continuous stirring at room temperature for nearly four hours. The mixed solution was then heated on a hot plate at 80^oC for several hours until a dried precursor was obtained. Then the as synthesized powder was calcined in a muffle furnace at 600^oC for 3 hours.

Characterisation

The crystallite phase of the magnesium ferrite was confirmed by X - ray diffraction analysis using XPERT PRO diffractometer. The Fourier Transform Infrared spectrum recorded in the wave number range of 4000 cm^{-1} to 400 cm^{-1} using Bruker IFS66V FT-IR spectrometer confirmed the spinel structure of the synthesized nanoparticles. The morphology of the prepared sample was studied using Field Emission Scanning Electron Microscopy. The magnetic parameters were measured using Vibrating Sample Magnetometer and the dielectric properties were studied using AGILENT 4284 A.

III. RESULTS AND DISCUSSION

Powder X-ray diffraction Analysis

The PXRD pattern of $MgFe_2O_4$ nanoparticles is illustrated in Figure 1. The result obtained from XRD data agrees well with the standard data of Magnesium ferrite (JCPDS file No: 89-3084). The typical reflections at (220), (311), (400) (511) and (440) in the figure specify the existence of cubic spinel structure. The lattice parameter of the synthesized Magnesium ferrite nanoparticle is found to be a = 8.3893 $\pm$ 1Å using UNITCELL software. The particle size of $MgFe_2O_4$ ranges from 24 to 56 nm. X-ray density and hopping length of $MgFe_2O_4$ nanoparticles are obtained as P_x = 5.3020g/cc d_A = 3.6291 and d_B = 2.9632 respectively [7].

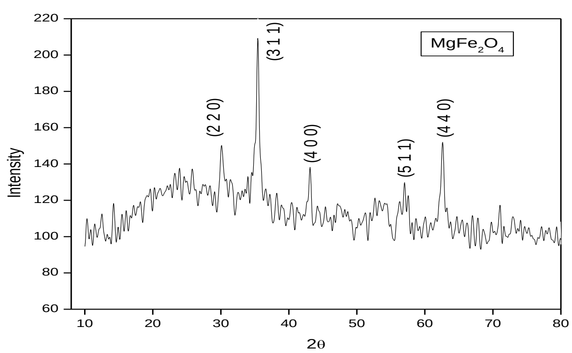

Figure 1. XRD pattern of $MgFe_2O_4$

FT-IR measurement

For a spinel structure the FT-IR spectrum recorded in the wave number range 4000 cm^{-1} to 400 cm^{-1} is expected to contain two main broad metal – oxygen bands, one (ν_1) in the range 600 cm^{-1} – 550 cm^{-1} due to the stretching vibrations of the tetrahedral metal – oxygen bond and the other one (ν_2) in the range 450 cm^{-1} - 385 cm^{-1} due to octahedral metal – oxygen bond [8]. Figure 2 represents the FTIR spectrum of $MgFe_2O_4$, which reveals the presence of $\nu_1$vibration [Fe–O] at 573 cm^{-1} and ν_2 vibration [Mg-O] at 437 cm^{-1}, thereby confirming the spinel structure of synthesized $MgFe_2O_4$. Their force constants are calculated as 2.4019 Nm^{-1} and 1.3970 Nm^{-1} respectively. The intensive broad band around 3450 cm^{-1} and less intense band around 1620 cm^{-1} in the spectra are due to O–H stretching vibration interacting through H bonds. The stretching vibration of the carboxylate group is observed around 1380 cm^{-1} and the band at around 1090 cm^{-1} corresponds to nitrate ion traces.

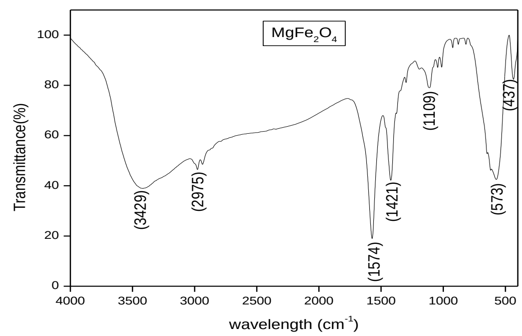

Figure 2. FTIR spectrum of $MgFe_2O_4$

FESEM Analysis

The morphology of the synthesized Magnesium Ferrite nanoparticles were recorded using FESEM. The FESEM image of $MgFe_2O_4$ at the magnification of 1µm and 500 nm are depicted in Fig. 3(a) and 3(b) respectively. The image 3 (a) displays the formation of squishy and crumbly magnesium ferrite powder at 1µm magnification. The image 3 (b) shows the formation of magnesium ferrite with multi spherical grain agglomerates or spherical clusters at 500 nm magnification. There is substantial degree of agglomeration in Magnesium ferrite nanoparticles. The agglomeration ensues in ferrite nanoparticles owing to its magnetic nature and the binding of primary particles seized together by frail surface interaction such as Vander Waals force [9]. Also the voids and apertures in the images may be ascribed to the discharge of enormous volume of gas created by the decomposition throughout the combustion.

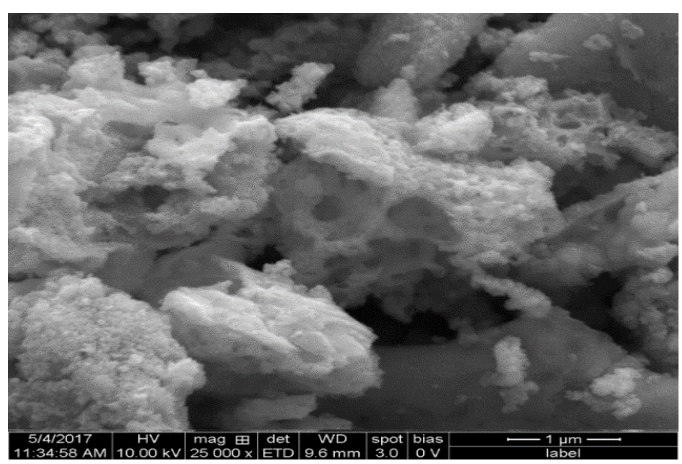

Figure 3. (a) FESEM image of $MgFe_2O_4$ at 1 µm magnification

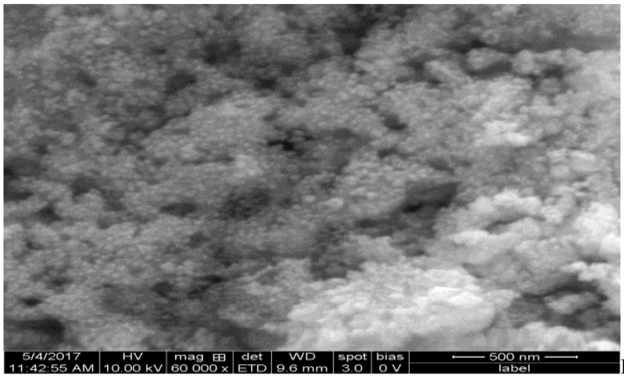

Figure 3. (b) FESEM image of $MgFe_2O_4$ at 500 nm magnification

EDAX Analysis

The elements present in the Magnesium Ferrite nanoparticles are analyzed using EDAX. The EDAX spectrum of $MgFe_2O_4$ is portrayed in Figure 4. The peaks at 0.7 eV and 6.4 eV confirm the existence of iron while the peak at 1.2 eV confirms the existence of magnesium in the sample. The presence of oxygen is revealed by the peak at 0.5 eV in the spectrum.

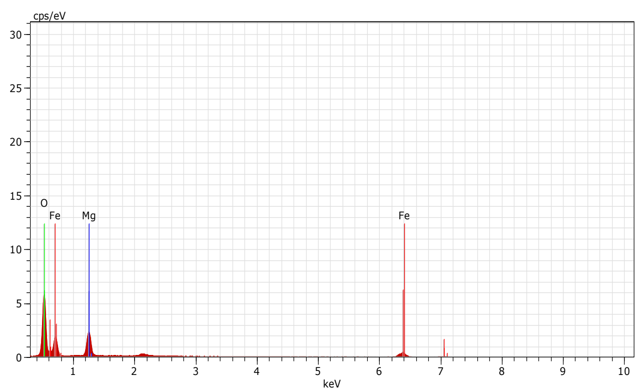

Figure 4. EDX spectrum of $MgFe_2O_4$

VSM Analysis

The magnetic properties are determined using Vibrating Sample Magnetometer. Figure 5 corresponds to the magnetic hysteresis loop for the Magnesium ferrite nanoparticles at room temperature. The hysteresis loop of the as synthesized $MgFe_2O_4$ is found to have less loop area which indicates that $MgFe_2O_4$ is a soft magnetic nanoparticle with potential application as magnetic memory devices. The magnetic moment, saturation magnetization, retentivity and coercivity of the $MgFe_2O_4$ nanoparticles are 0.0165, 0.46106 emu, 0.0039 emu and 85.710 G respectively [10].

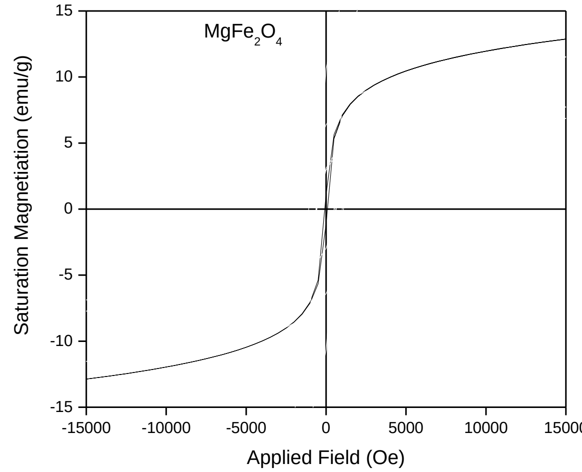

Figure 5. Hysteresis loop for $MgFe_2O_4$

Dielectric Analysis

The dielectric constant of $MgFe_2O_4$ is calculated from dielectric analysis. The capacitance of the parallel plate capacitor made by the electrodes, with the sample as the dielectric medium was measured. The capacitance was measured in the frequency range 100 Hz to 1 MHz at different temperatures ranging from 40°C to 150°C. Dielectric constant ε_r is calculated from the measured capacitance value using the following equation [11]

$$\varepsilon_r = \frac{tC_p}{A\varepsilon_0}$$

where t is thickness of the sample, C_p is the capacitance, ε_0 is the permittivity of free space and A is the area of the sample. The plot of the variation of dielectric constant vs frequency at different temperatures for $MgFe_2O_4$ nanoparticle is given in Figure 6. From Figure 6 it can be noted that dielectric constant decreases with increase in temperature.

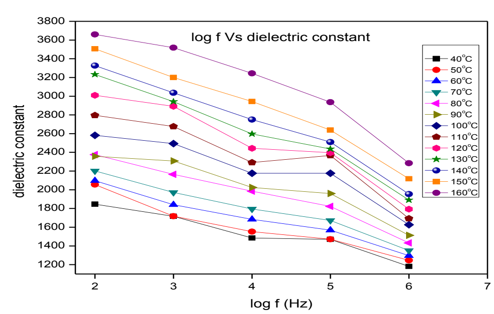

Figure 6. Variation of dielectric constant versus frequency at different temperature for $MgFe_2O_4$

The plot of the variation of dielectric loss with frequency is given in Figure 7. The dielectric loss is found to increase with increase in frequency and increase in temperature.

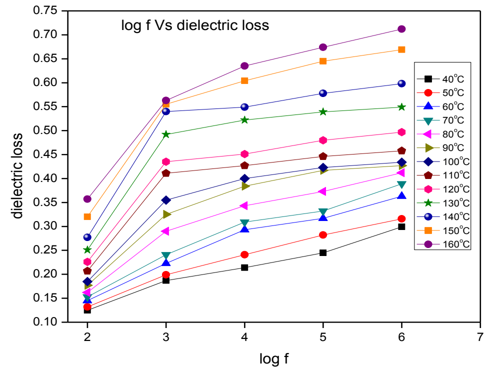

Figure 7. Variation of dielectric loss versus frequency at different temperature for $MgFe_2O_4$

The AC conductivity is calculated using the relation

$$\sigma_{ac} = \omega \varepsilon_0 \varepsilon' \tan\delta$$

where, σ_{ac} is permittivity of free space, ω is the angular frequency and δ is the loss factor. The plot of the variation of ac conductivity with frequency is given in Figure 8. The AC conductivity is observed to increase with increase in applied frequency and temperature.

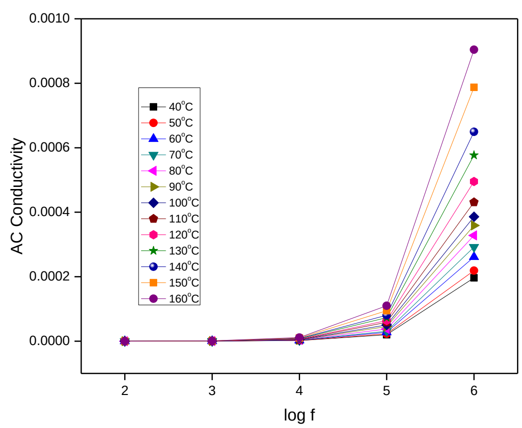

Figure 8. Variation of AC Conductivity versus frequency at different temperature for $NiFe_2O_4$

IV. CONCLUSION

Magnesium ferrite nanoparticles have been synthesized through green synthesis route using egg white as the eco-friendly precursor. The egg white protein albumen has acted as fuel in the auto combustion method. PXRD results confirmed the formation of magnesium ferrite $MgFe_2O_4$ nanoparticles with cubic spinel structure and having particle size ranging from 24 to 56 nm. The FESEM micrographs exposed high degree of agglomeration with spherical multi grains. The EDAX spectra clearly confirmed the presence of Mg, Fe and O in $MgFe_2O_4$ nanoparticles. The magnetic parameters like coercivity, retentivity and magnetic moment were measured using VSM. The dielectric constant and dielectric loss of the particle decrease with increase in the frequency of the applied signal while the AC conductivity increases with increase in frequency. As $MgFe_2O_4$ shows the characteristics of soft magnetic nanoparticles, extensive research may be done towards its application as magnetic memory devices. Also magnetic nanomaterials antibacterial agents and photo catalysts, study may be directed in these areas too.

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