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Structural and Magnetic Characterizations of Citric Acid Assisted Sol-gel Synthesized Magnesium Ferrite Nanoparticles

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Abstract: The magnesium spinel ferrite ($MgFe_2O_4$) in nanocrystalline form was prepared by citric acid assisted sol-gel auto combustion method using AR grade nitrates of magnesium and ferric ions. In the synthesis process, the commonly used citric acid was taken as a fuel. The pH of the mixed citrate nitrate precusor solution was adjusted at 7 by adding complexing agent as ammonia. The metal nitrates to fuel (citric acid) ratio was chosen as 1:3. The as-prepared powder was sintered at 550°C for 4 h and same was used for further characterization. The structural characterization was carried out by X-ray diffraction technique. The single phase cubic spinel structure and nanocrystalline nature of the prepared magnesium ferrite nanoparticles was confirmed by analyzing the X-ray diffraction pattern recorded at room temperature (300K). Using the XRD data the lattice constant, X-ray density and other structural parameters were evaluated. The lattice constant, X-ray density and crystallite size of the $MgFe_2O_4$ nanoparticles was found to be in the reported range. The magnetic characterizations were made through pulse field hysteresis loop tracer technique. A typical hysteresis plot M-H curve recorded at room temperature reveals the ferrimagnetic behavior of the sample. Using M-H plot, the values of saturation magnetization (M_S), remenance magnetization (M_R), and coercivity (H_C) were obtained. The obtained values of magnetic parameters are in good agreement with the reported data in the literature.

Keywords: Magnesium ferrite, Sol-gel synthesis, X-ray diffraction, Magnetization.

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I. INTRODUCTION

In the recent year, magnetic nanoparticles of spinel ferrites have attracted the attention of scientist and technologist due to their interesting and superior properties in comparison with bulk sample. The magnetic nanoparticles show excellent chemical stability, high surface to volume to ratio, single domain nature and other useful properties [1]. These properties are useful in many technological devices and their use in fields of microwave, sensors, catalyst, medicine, biomedical, high frequency electronic devices etc [2-5]. The technological demand of the high performance devices has triggered the synthesis of the nanocrystalline spinel ferrite materials [6]. The important electrical and magnetic properties of spinel ferrite are sensitive to method of preparation and preparative parameters apart from chemical composition, nature and type of dopant [7-10].

Various wet chemical methods are available for making spinel ferrite in nanocrystalline form [11, 12]. These methods include prominently chemical coprecipitation, sol-gel auto combustion, hydrothermal, etc. Sol-gel auto combustion is more reliable, easy and cost effective technique compared to other synthesis techniques. The homogeneity, yield, phase purity, fine particle size distribution, etc characteristic features of sol-gel synthesis method are found to be better than other methods [13].

Spinel ferrite has a general chemical formula AB_2O_4 with a spinel structure in which (A) and [B] is the two interstitial sites namely tetrahedral and octahedral respectively. Unit cell of spinel ferrite consists of 8 formula units i.e. 56 atoms. Normally, (A) sites are occupied by divalent cations like Mg, Mn etc and trivalent cations as Fe^{3+} ions occupy octahedral [B] sites. In the literature, cobalt and nickel ferrite are extensively studied by various researchers. However, less attention is paid to the magnesium ferrite which has unique characteristics.

The magnesium spinel ferrite is a soft magnetic n type semiconductor and has many applications in catalysis, high-density recording, gas sensors, transformers and ferrofluids. In this paper, we focus on the synthesis, structural and magnetic properties of the nanocrystalline magnesium spinel ferrite and the experimental results so obtained are presented herein.

II. EXPERIMENTAL

The nanocrystalline magnesium spinel ferrite was prepared by using sol-gel auto combustion method. The following AR grade chemicals were used as raw materials for the synthesis of the magnesium spinel ferrite at low temperature using wet chemical synthesis route. Materials used: Magnesium nitrate ($Mg(NO_3)_2$), ferric nitrate ($Fe(NO_3)_2$) as source of magnesium and ferric ions respectively, citric acid ($C_6H_8O_7$) as fuel, distilled water as solvent and ammonia as complexing agent were used.

2.1 Preparation of nanocrystalline magnesium spinel ferrite

The metal nitrates to fuel (citric acid) ratio was taken as l: 3. Ammonia solution was added to adjust the pH of mixed solution of nitrates and citric acid at 7. Then the sol was stirred at a temperature around 110°C. The as-prepared powder was sintered at 500°C for 4 h and then used for further investigations as structural and magnetic properties.

2.2 Characterizations

The phase purity and crystallinity of the prepared magnesium spinel ferrite was checked by X-ray diffraction technique at room temperature in 2θ range of 20°-80° with Cu Kα radiation of wavelength λ = 1.5046 Å. The magnetic properties of the nanocrystalline magnesium spinel ferrite were studied by pulse field hysteresis loop tracer technique at room temperature. The external applied magnetic field was of the order of 5000 Oe.

III. RESULTS AND DISCUSSION

3.1 X-ray diffraction

The X-ray diffraction pattern of prepared $MgFe_2O_4$ spinel ferrite nanoparticles is shown in figure l. The XRD pattern shows the reflections belonging to cubic spinel structure; no impurity peaks have been observed in the XRD pattern of $MgFe_2O_4$. From the primary analysis of XRD pattern the single phase formation of $MgFe_2O_4$ was confirmed. The Miller indices (h k l) corresponding to Bragg's angles along with their interplanar spacing (d) values, intensity and relative intensity ratios obtained from the XRD data are represented in table 1. It is observed from the table I that the interplanar spacing value decreases with increasing Bragg's angle. Also it is observed that the plane (311) is of the most intensity peak as compared to other planes as (220), (222), (400), (422), (511) and (440). The various structural parameters such as lattice constant, unit cell volume, X-ray density, bulk density, porosity, crystallite size etc were calculated using the XRD data.

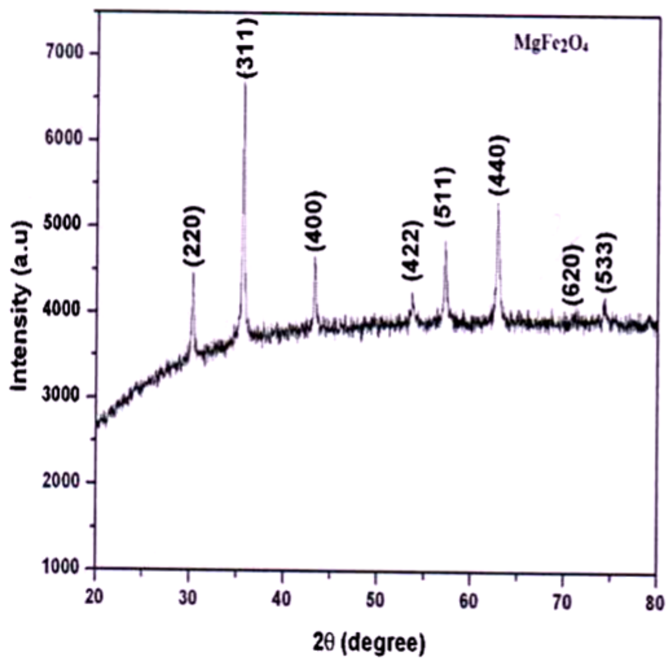

Fig. 1 XRD pattern of $MgFe_2O_4$ recorded at room temperature

Table 1 Miller indices (h k l), Bragg's angle (2θ), Interplanar spacing (d), Intensity (I) and relative intensity ratio (I/Io) of $MgFe_2O_4$

h k l	2θ (degree)	d (Å)	I (a.u.)	I/I_O
(220)	30.309	2.9465	4471.8	65.4
311)	35.658	2.5158	6842.6	100.0
(400)	43.256	2.0899	4666.5	68.2
(422)	53.675	1.7062	4264.2	62.3
(511)	57.198	1.6092	4877	71.3
(440)	62.754	1.4794	5330.6	77.9
(620)	71.166	1.3238	4112	60.1
(533)	74.298	1.2755	4219.5	61.7

Lattice constant (a)
Using the values of inter planar spacing (d) and the corresponding Miller indices, the lattice constant (a) was calculated using standard relation

$$a = d\sqrt{h^2 + k^2 + l^2}\ \text{Å} \qquad \ldots 1$$

Where, d is interplanar spacing, (h k l) is Miller indices. The obtained value of the lattice constant is 8.341 Å.

Unit cell volume (V)
The unit cell volume was calculated by using the following equation,

$$V = a^3 \text{Å}^3 \qquad \ldots 2$$

Where, V is the unit cell volume, 'a' is the lattice constant. The calculated value of the unit cell volume is found to be 580.36 cm^3.

X-ray density (dx)
The values of lattice constant and molecular weight were used to determine the X-ray density of nanocrystalline $MgFe_2O_4$ spinel ferrite. By using the following relation, the X-ray density was calculated,

$$d_X = \frac{Z \times M}{V \times N_A} \quad gm/cm^3 \quad \ldots 3$$

Where, d_x is X-ray density, Z is the number of molecules per unit, M is molecular weight of the sample, V is the unit cell volume, N_A is the Avogadro's number. The calculated X-ray density is 4.5778 gm/cm^3.

Bulk density (d_B)

The bulk density of $MgFe_2O_4$ spinel ferrite was measured using Archimedes principle. The value of bulk density was found to 3.8240 gm/cm^3. The small difference between the X-ray density and bulk density values is due to the existence of inter and intra granular porosity of the sample.

Porosity (%P)
The values of X-ray density, bulk density were used to obtain percentage porosity using the following equation,

$$P = 1 - \frac{d_B}{d_X}\ \% \qquad \ldots 4$$

Where, d_B is the bulk density, d_x is the X-ray density. The percentage porosity of the magnesium spinel ferrite was 16.46 %.

Crystallite size (t)
The crystallite size was calculated using Debye-Scherer's formula as given below. The most intensity plane (311) was considered for the determination of the crystallite size.

$$t = 0.9\ \lambda / \beta \cos\theta \qquad \ldots 5$$

Where, λ is wavelength of the Cu-Kα radiation, β is the full width of the half maximum, θ is Bragg's angle. The crystallite size calculated by Debye Scherrer's formula was of the order of 22 nm.

3.2 Magnetic properties
The magnetic hysteresis curve of nanocrystalline $MgFe_2O_4$ spinel ferrite is shown in fig. 2. The hysteresis curve was used to determine saturation magnetization (M_S), remanence magnetization (Mr), remanence ratio (Mr/M_S), coercivity (H_C) and magneton number (n_B). The magneton number has been calculated by using the formula,

$$n_B = (M.W. \times M_S)/5585 \quad \ldots 6$$

Where, M.W. is molecular weight of the sample, M_s is the saturation magnetization. In a cubic system of ferrimagnetic spinels, the magnetic order is due to a super exchange interaction mechanism occurring between the metal ions in the A and B sub-lattices. The $MgFe_2O_4$ has a normal spinel structure in which Mg^{2+} occupy tetrahedral sites and Fe^{3+} ions are equally distributed in tetrahedral and octahedral sites with their spin in the opposite direction. The Mg^{2+} ions can exist in both sites but strongly prefer to occupy octahedral site. The magnetic moment of Mg^{2+} is zero, so in $MgFe_2O_4$ magnetic couplings purely originate from the magnetic moment of Fe cations. The values of all these magnetic parameters are saturation magnetization 28.83 emu/gm, remanence magnetization 0.02 emu/gm, remanence ratio 0.0007, coercivity 84.98 Oe, and the magneton number l.03 μ_B for the prepared nanocrystalline magnesium spinel ferrite are in the reported range.

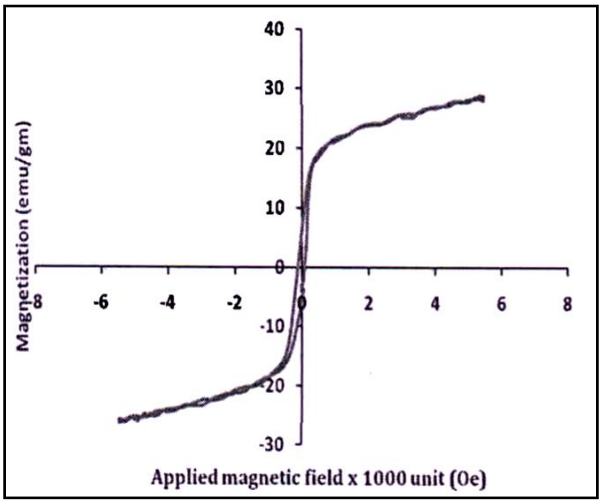

Fig. 2 M-H plot of $MgFe_2O_4$ recorded at room temperatures

IV. CONCLUSION

The nanocrystalline magnesium spinel ferrite prepared successfully using sol-gel auto combustion technique. The XRD pattern revealed the formation of a single phase cubic spinel structure. The calculated crystallite size suggested the nanocrystalline nature of the prepared $MgFe_2O_4$ spinel ferrite. The magnetic properties of the nanocrystalline magnesium spinel ferrite are in the reported range.

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