Magnetic Properties of Nickel Ferrite Magnetic Nanoparticles Prepared via Glycine Assisted Sol-Gel Auto Combustion Route

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Abstract. The present research paper deals on the synthesis of nickel ferrite (NiFe₂O₄) nanoparticles using sol-gel auto combustion technique in which glycine was used a chelating agent. The prepared nanoparticles were annealed at 500°C for 4 h. and then used for structural, magnetic and other investigations. The X-ray diffraction technique was used for phase purity and structural determination. Room temperature X-ray diffraction pattern of NiFe₂O₄ nanoparticles shows all the reflections belonging to cubic spinel structure. The reflections are intense and slightly broader. No extra peak other than cubic spinel structure was observed in the XRD pattern. This indicates the single phase formations of the nickel ferrite nanoparticle. The refinement of XRD data has been carried out by using full proof programmer. The lattice constant was calculated using XRD data as well as Cohen’s least square method, the obtained lattice constant matches with the reported value. The other structural parameters like lattice strain, X-ray density etc. structural parameters were also calculated using standard relations.

The crystallite size was determined using Scherrer’s formula in which the most intense peak (311) was considered. The crystallite size was found to be 24 nm which indicates the nanocrystalline nature of the prepared nickel ferrite nanoparticles. The magnetic properties were investigated using the pulse field hysteresis loop (PFHL) technique at room temperature. The saturation magnetization, coercivity and remanence magnetization shows enhanced values. All the structural and magnetic properties results were compared with the reported data in which other chelating agent like citric acid etc.

1. Introduction

Over the last decade, nanosized magnetic materials, especially the ferrite nanoparticles, have been a topic of intense research mainly due to their unique magnetic properties which are different from those of their bulk counterparts. Among the three types of ferrites, spinel ferrites have attracted the attention of scientist and technologist due to their remarkable and useable properties which are useful in various industrial and technological applications [1-3]. The spinel ferrite has the general formula of MFe₂O₄.
where M is a divalent metal ion such as Co, Mn, Ni, Zn etc. In the class of spinel ferrite materials, nickel ferrite (NiFe$_2$O$_4$) with inverse spinel structure is one of the most promising candidate which is receiving even more attention and increasing demand as it can be used in novel large-scale applications such as Photocatalysis, magnetic recording media, magnetic sensors, magnetic memories, magnetic fluids, magnetic composites and catalysis etc [4-5]. Moderate saturation magnetization and wide band gap of nickel ferrite nanoparticles makes them appropriate for photocatalytic and waste water applications. The large tuning ability of nickel ferrite makes it possible to implement the performance of devices used in sensing and biomedical applications as a contrast agent for magnetic Resonance Imaging (MRI) and heat mediator for magnetic fluid hyperthermia [6]. Spinel ferrite nanoparticles have been synthesized using various methods, such as co-precipitation method, hydrothermal method, micro-emulsion method, sol-gel method, etc [7]. Among various synthesis methods, sol-gel auto combustion method has been widely used for synthesis of spinel ferrite nanoparticles [8]. The important structural, electrical, dielectric and magnetic properties of spinel ferrite are more sensitive to the synthesis parameters compared to synthesis method and cation distribution. The synthesis parameters that affect the properties of final product are type of fuel, fuel to oxidizer ratio, amount of fuel percentage, ignition temperature, water content in precursor, pH of the solution, sintering temperature, sintering atmosphere etc. The selection of fuel is an important criterion in obtaining the improved properties of the spinel ferrite nanoparticles. A good fuel should possess the properties like non-violent reactions and without evolution of toxic gases. Depending upon the fuel used, the nature of combustion differs from flaming to non-flaming. In spite of a large number of papers on sol–gel auto combustion synthesis of spinel ferrite using various fuels, no prominent literature is available on the use of glycine as a fuel for sol-gel auto combustion synthesis of spinel structured nickel ferrite nanoparticles. Glycine has high reducing capacity and it generates more number of moles of gases during the reaction. The specific surface area of the end product can be increased by addition of glycine in the combustion synthesis and also the resultant particle size can be controlled. It is easily available and has high exothermic nature, which makes it the most suitable material to be used as a fuel for synthesis of spinel ferrites. Thus, the present study reports on glycine assisted sol-gel auto combustion synthesis of nickel ferrite nanoparticles and investigations of their structural and magnetic properties.

2. Experimental

The nanocrystalline spinel structured nickel ferrite (NiFe$_2$O$_4$) sample was prepared by sol-gel auto combustion method using glycine as a fuel. AR grade chemicals such as nickel nitrate (Ni (NO$_3$)$_2$), ferric nitrate (Fe (NO$_3$)$_3$) and glycine (C$_2$H$_5$NO$_2$) were used for the synthesis. The metal nitrates to fuel ratio was taken as 1:4.4. Ammonia solution was added to adjust the pH of the solution at 7. The as-synthesized powder is sintered at 500°C for 4 h and then used for further investigations. The prepared sample was characterized by X-ray diffraction (XRD) technique by Regaku model. The XRD patterns were recorded at room temperature in the 20 range of 20° to 80° using Cu-Kα radiation (λ = 1.54056 Å). The magnetic properties of the sample were measured using pulse field hysteresis loop technique (Magnata Company) at room temperature.

3. Results and discussion

3.1. X-ray diffraction analysis

The powder XRD pattern of the nickel ferrite nanoparticles synthesized by sol-gel auto combustion method using glycine as a fuel is shown in Fig. 1. It is noticed from the pattern that all the peaks correspond to nickel ferrite without any additional impurities. The peaks are indexed based on the JCPDS (#10-0325). The average crystallite size of the compounds is calculated using the Debye-Scherrer’s formula which is found to be 24 nm. Using the interplanar spacing (d) and the
corresponding Miller indices (h k l), the lattice constant (a) of the sample was calculated using standard relation and found to be 8.33 Å. Also, the Cohen’s least square method was used to determine the lattice constant values which are found to be 8.33 Å. The values of lattice constant measured from XRD data and Cohen’s least square method are in well agreement with each other. Also, the value of lattice constant well matches with that of reported in the literature [9]. Various structural parameters such as X-ray density (d_x), bulk density (d_B), porosity (P %) and unit cell volume (V) were calculated from the XRD data by standard relations. The X-ray density and bulk density was found to be 5.29 gm/cm³ and 3.29 gm/cm³ respectively. The percentage porosity was observed to be 17.03 %. The unit cell volume of the prepared sample was found to be 588.9 (Å)³.

Fig. 1 X-ray diffraction pattern of nickel ferrite nanoparticles

3.2. Magnetic properties

The magnetic properties of the prepared nickel ferrite sample were measured using pulse field hysteresis loop technique at room temperature. Obtained M-H curve of the prepared sample is shown in Fig. 2. From the M-H curve, the saturation magnetization (M_s), remanent magnetization (M_r) and coercive field (H_c) was found to be 48.24 emu/gm, 18.1 emu/gm and 59.67 Oe, respectively. The value of saturation magnetization is lower than the bulk value of 56 emu/gm [10]. The lower value of M_s exhibited by nickel ferrite with smaller particle size could be due to the surface disorder and modified cationic distribution. Another reason for this lower value of M_s of small sized nanoparticles is disordered spins which are present on the surface of the nanoparticles which prevents the core spin from aligning from field direction.

Table 1 shows the comparative table for the preparation of nickel ferrite nanoparticles by sol gel auto combustion method assisted via different chelating agents and current chelating agent i.e. glycine. It is evident from table 1 that, glycine can be used as fuel to enhance the properties of nickel ferrite nanoparticles in comparison to other fuels such as citric acid, urea and sodium azide. All the outcomes shows that glycine can be used as a fuel for synthesis of nickel ferrite nanoparticles with smaller size particles and improved magnetic properties which can be applied for various applications such as Photocatalysis, magnetic hyperthermia, magnetic sensors, magnetic fluids etc.
Fig. 2 M-H plot of nickel ferrite nanoparticles

Table 1 Comparative summary of nickel ferrite nanoparticles prepared by sol gel auto combustion method assisted via different chelating agents

| Sr. No. | Fuel                  | Crystallite size (nm) | Magnetization (emu/gm) | Coercivity (Oe) |
|---------|-----------------------|-----------------------|------------------------|-----------------|
| 1.      | Citric acid [11]      | 41                    | 37.80                  | 254.27          |
| 2.      | Urea [12]             | 25                    | 47.44                  | 170.07          |
| 3.      | Sodium azide [13]     | 25                    | 42.73                  | 252.36          |
| 4.      | Glycine (Present work)| 24                    | 48.24                  | 59.67           |

4. Conclusion

The spinel structured nickel ferrite nanoparticles were successfully synthesized by sol-gel auto combustion method assisted by glycine. Pure phase formation of nickel ferrite with no impurities was observed from X-ray diffraction pattern. The average crystallite size of the prepared sample was found to be 24 nm. The values of saturation magnetization ($M_s$) and remanent magnetization ($M_r$) were found to be 48.24emu/gm, and 18.1emu/gm respectively. All the structural and magnetic properties results were compared with the reported data in which other chelating agent like citric acid etc. Thus, it is observed from experimental results that glycine can be used as a fuel for synthesis of nickel ferrite nanoparticles with smaller size particles and improved magnetic properties which can be applied for various applications such as Photocatalysis, magnetic hyperthermia, magnetic sensors, magnetic fluids etc.

5. References

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