Sol-gel Auto Combustion Synthesis and structural characterizations of Nickel Ferrite Nanoparticles by X-Ray Diffraction Technique

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Abstract: The method of synthesis plays an important role in governing the properties of spinel ferrite nanoparticles. The most common and advantageous method for preparation of nanoparticles is the sol-gel auto combustion method. In the present work, nickel ferrite nanoparticles were synthesized by optimizing various synthesis parameters using sol-gel auto combustion method. Citric acid was used as a fuel in the synthesis of nickel ferrite nanoparticles. The metal nitrate to fuel ratio was chosen as 1:3. The as prepared powder of nickel ferrite was annealed at 500°C for 4 h and characterized by X-ray diffraction technique to study the structural properties. The crystallite size, lattice constant, X-ray density, unit cell volume etc. structural parameters were evaluated and compared with the standard results. All these structural parameters agreed close to the literature values. The crystallite size obtained using Scherrer's formula shows that the prepared sample possesses nanocrystalline nature.

Index Terms - Nickel Ferrite, Sol-gel auto combustion, XRD.

I. INTRODUCTION

Magnetic nanomaterials have attracted attention in recent years due to their applicability in various areas, such as magnetic drug delivery, magnetic resonance imaging, magnetic recording media, or as catalysts, gas sensors, and for magneto-optical devices [1]. The development of novel magnetic nanoparticles is a subject of growing interest, owing to the outstanding properties displayed by such particles at nanoscale. Among magnetic nanomaterials, ferrites are the most important material used as soft magnet and low loss materials at high frequencies, due its magnetic properties, high chemical stability, low conductivity and reduced current losses [2].

On the basis of crystal structure, ferrites are grouped into three main categories namely, spinel ferrite, garnet and hexagonal ferrites. Among the ferrites, spinel ferrites are a class of compounds with the general formula of MFe_2O_4 (where M = Co, Ni, Zn, Mg, Mn etc.). Nanosized nickel spinel ferrites are currently of great research interest due to the ease of tailoring their magnetic properties by a systematic substitution of cations and by changing the average crystallite size through a proper choice of preparation techniques and post preparation treatments [3]. They belong to a technologically important class of magnetic materials which have high resistivity and chemical stability. These properties make them suitable for a wide variety of applications where other magnetic materials cannot be used.

In general, nickel spinel ferrite nanoparticles are prepared by sol-gel auto combustion method by various researchers. This method is more advantageous compared to other wet chemical methods [4]. However, the quality of the nanopowder which we obtain through sol-gel auto combustion method depends mainly on nature of fuel, fuel to oxidizer ratio, pH, annealing temperature etc parameters. These parameters affect the crystallite size, microstructure and morphology there by affect the properties of the nickel spinel ferrite nanoparticles. Taking into consideration all these synthesis parameters the aim of the present work is to synthesize nickel ferrite nanoparticles by optimizing necessary synthesis parameters such as pH, annealing temperature and fuel with a view to obtain high quality, homogeneous, smaller crystallite size nanoparticles which can be useful for diverse applications [5]. The prepared nanopowder was characterized by X-ray diffraction technique and other methods. Herein, the structural properties obtained by X-ray diffraction method are reported in this work.

II. EXPERIMENTAL METHOD

Materials

Nickel nitrate ($Ni(NO_3)_2\cdot 6H_2O$), ferric nitrate ($Fe(NO_3)_3\cdot 9H_2O$) and citric acid were used as a raw materials for sol-gel auto combustion synthesis of nickel ferrite nanoparticle. All the reagents used for the synthesis were of analytical grade (AR) and used as received without further purification.

Preparation

Nickel ferrite nanoparticle were synthesized by sol-gel auto combustion method using citric acid as a fuel. The stoichiometric proportions of metal nitrates to fuel (citric acid) ratio as 1:3 were taken into separate glass beakers. The mixed solution was stirred for 20 - 25 minutes to dissolve completely into distilled water. After complete dissolution they were mixed together. Ammonia was added drop-wise into the solution to adjust pH value at 7 and stabilize the nitrate-citric acid solution. Then the neutralized solution was constantly magnetically stirred and heated at 90°C - 100°C for 6 h on a hot plate. On the formation of sol-gel, very viscous gel the temperature was further raised up to 110°C so that the auto combustion of the dried gel started and finally powder was obtained. The flowchart of the sol-gel auto combustion synthesis for nickel ferrite nanoparticles is shown in fig. 1.

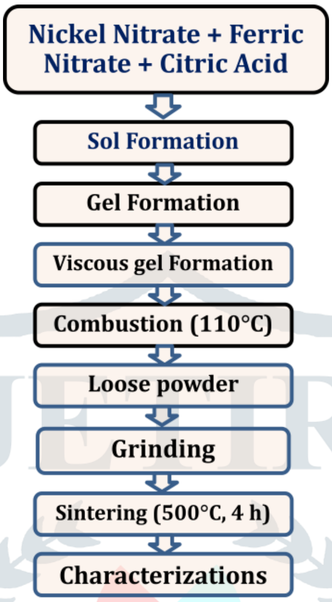

Fig. 1 Flowchart of nickel ferrite nanoparticles synthesis by sol-gel auto combustion

Characterizations

The prepared nickel ferrite nanoparticles were characterized by X-Ray diffraction (XRD) technique. The room temperature XRD pattern was recorded in the 2θ range of 20° and 80° with the appropriate wavelength. Using XRD data, various structural parameters such a crystallite size, lattice constant, unit cell volusme, X-ray density, bulk density, porosity etc parameters were calculated.

III. RESULTS AND DISCUSSION

X-Ray diffraction studies

The X-ray diffraction (XRD) pattern of $NiFe_2O_4$ nanopowder was recorded at room temperature using X-ray diffractometer in the 2θ range of 20° to 80°. **Fig. 2** depicts XRD pattern of the sample under investigation. All the peaks in the XRD pattern were indexed using Bragg's law. The XRD patterns reveal all the peaks belonging to cubic spinel structure suggesting that, the prepared samples possess single phase nature. All the peaks observed in the XRD patterns are intense and slightly broader reflecting nanocrystalline nature. The XRD pattern of pure nickel ferrite sample is well matched with that reported in the literature and JCPDS Card No. #22-1086 [6].

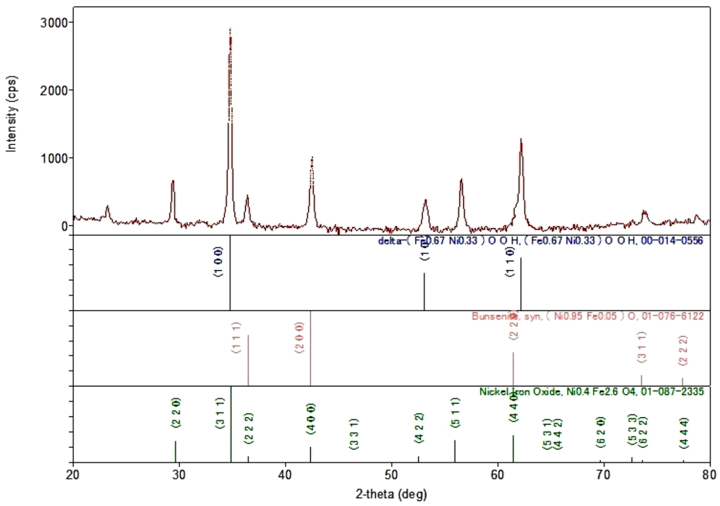

Fig. 2 X-ray diffraction pattern of $NiFe_2O_4$ nanoparticles

Crystallite size (t)

The crystallite size (t) of the prepared nickel ferrite nanoparticles was calculated using Debye-Scherrer's formula. The most intense peak i.e. (311) of the XRD pattern was used to determine the full width at half maxima (FWHM). It is found that, the crystallite size is in nanometric range confirming the nanocrystalline nature of the prepared nickel ferrite nanoparticles.

Lattice constant (a)

The lattice constant (a) was calculated using the interplanner spacing (d) value and Miller indices (h, k, l). The value of lattice constant is given in table 1 which show close agreement with the literature value.

Unit cell volume (V)

The unit cell volume (V) was calculated using lattice constant value. The value of the unit cell volume is given in table 1.

X-ray density (d_X)

The X-ray density (d_X) of the prepared nanoparticles was calculated using the values of molecular weight (M), Avogadro's number and lattice constant (a). The X-ray density value is given in table 1.

Bulk density (d_B)

The bulk density (d_B) of the prepared nanoparticles was measured using Archimedes principle. Toluene was used as a liquid medium. The value of the bulk density is given in table 1 and is found to be less than that of X-ray density.

Porosity (%P)

The porosity (%P) of the prepaid nanoparticles was calculated using the values of X-ray density (d_X) and bulk density (d_B). The value of porosity is given in table 1. The observed value of porosity is slightly higher may be due to agglomeration of particles.

Table 1 Values of crystallite size (t), lattice constant (a), unit cell volume (V), X-ray density (d_X), bulk density (d_B) and porosity (%P) of nickel ferrite nanoparticles

Sample/Parameters	t (nm)	a (Å)	V ($Å^3$)	d_X (gm/cm^3)	d_B (gm/cm^3)	% P
$NiFe_2O_4$	**32**	**8.338**	**579.68**	**5.371**	**3.752**	**30.14**

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IV. CONCLUSION

Nickel ferrite nanoparticles were successfully synthesized by optimizing various synthesis parameters using sol-gel auto combustion method. The crystallite size, lattice constant, X-ray density, unit cell volume, X-ray density, bulk density, porosity etc. structural parameters were evaluated with the help of XRD data and compared with the standard results. All these structural parameters agreed close to the literature values. The crystallite size obtained using Scherrer's formula shows that the prepared sample possesses nanocrystalline nature.

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