Chemical Co-precipitation Synthesis and Characterizations of Nickel Ferrite Nanoparticles

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Abstract: In the present work, spinel nickel ferrite (NiFe₂O₄) nanoparticles were prepared through chemical co-precipitation method taking nitrates as raw materials. The molar ratio of nickel to ferric was kept at 1:2 (0.25 M and 0.5 M respectively) proportion. The pH of the mixed solution was maintained at 7 by adding 2 M NaOH solution. The prepared powder was sintered at 600°C for 4 h and same is used for further characterizations. X-ray diffraction pattern shows the entire diffraction peak belonging to cubic spinel structure. No extra peak other than cubic phase was seen in the XRD pattern. The analysis of XRD pattern confirms the formation of single phase material. The crystallite size, lattice constant, X-ray density etc. structural parameters were deduced using XRD data and found to be in the reported range. The surface morphology was studied through scanning electron microscopy technique. The grain size and specific surface area was determined using SEM analysis which proves that, the prepared nickel ferrite particles are nanocrystalline in nature.

Keywords: Nickel Ferrite, Chemical co-precipitation, XRD, SEM.

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I. INTRODUCTION

Ferrite belongs to a class of magnetic materials which exhibit both electrical and magnetic properties. Owing to their excellent high electrical resistivity, low eddy current and dielctric losses, high saturation magnetization, high Curie temperature etc. ferrite are used on large scale in many technological applications [1-2]. They can be used in transformer cores, antenna rods, memory chips, microwave devices, sensors and catalysts [3]. Recently, ferrites in nanocrystalline form have gained much importance in many areas due to their smaller size, large surface area and superparamagnetic behaviour. Currently, they are used as targeted drug delivery, contrasts agents in MRI, hyperthermia, magnetic sensors, catalysts etc [4]. In view of their multifunctional properties and applications they are being studied by many researchers.

The main objective of these researchers is to improve the properties of ferrite nanoparticles for desired applications. It is well known fact that, the properties of ferrite are dependent on the various parameters such as method of preparation, preparative parameters and conditions, nature and type of dopant [5]. In addition to this, the distribution of cations over the available sites is also responsible for change in the properties of ferrites. A careful control on the synthesis parameters will result in improved properties.

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). Spinels ferrites possess unit cell consists of cubic, close-packed package of oxides reveals two types of interstices namely tetrahedral (A) site and octahedral (B) sites. The unit cell of spinel ferrites consists of 32 oxygen ions, 16 trivalent iron ions and 8 divalent metal cations.

Ferrite nanoparticles are prepared by wet chemical methods such as sol-gel auto combustion, chemical coprecipitation, micro emulsion etc. These methods produce fine particles with nanometric scale. Among these methods, chemical coprecipitation method is more advantageous as compared to other methods. This method is simple, economic and produces high quality homogeneous fine powder [6].

In this work, nickel ferrite nanoparticles were synthesized by chemical coprecipitation and characterized by X-ray diffraction and scanning electron microscopy technique. The results on crystallite size, lattice constant, grain size, specific surface area etc. is reported in this work.

II. EXPERIMENTAL

Materials

Nickel nitrate (Ni $(NO_3)_2 \cdot 6H_2O$), ferric nitrate (Fe $(NO_3)_3 \cdot 9H_2O$) and NaOH were used as a raw materials for chemical coprecipitation 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

AR grade chemicals such as nickel nitrate (Ni (NO₃)₂), ferric nitrate (Fe (NO₃)₃), sodium hydroxide (NaOH), acetone ((CH₃)₂CO) and DI water were used as a raw materials for the synthesis. Aqueous solution of nickel nitrate and ferric nitrate in stoichiometric ratio 1:2 were mixed together and stirred for 1 h to get a homogeneous mixture. The pH of the mixture was adjusted to 7 by adding drop by drop solution of 2 M NaOH. The obtained precipitate was washed several times using doubled distilled water and ethanol to remove the impurities. Further, the precipitate was centrifuged and the centrifuged precipitate was then dried and grinded to form the homogeneous and fine powder. The prepared powder was sintered at 600°C for 4 h and same is used for further characterizations.

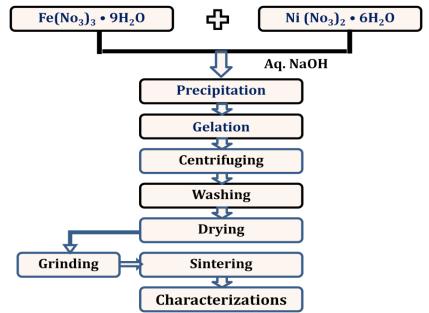


Fig. 1 Flowchart of chemical co-precipitation synthesis of nickel ferrite nanoparticles

Characterizations

The prepared nickel ferrite nanoparticles were characterized by X-Ray diffraction (XRD) technique. The room temperature XRD pattern was recorded in the 20 range of 20° and 80° with the appropriate wavelength. Using XRD data, various structural parameters such a crystallite size, lattice constant, unit cell volume, X-ray density, bulk density, porosity etc parameters were calculated. The morphology of the prepared nickel ferrite nanoparticles was studied through scanning electron microscopy technique. The SEM images were used to determine the grain size and surface energy of prepared nickel ferrite nanoparticles.

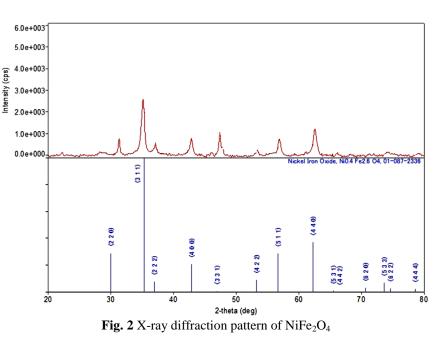
III. RESULTS AND DISCUSSION

3.1 Structural analysis

The X-ray diffraction (XRD) pattern of NiFe₂O₄ nanopowder was recorded at room temperature using X-ray diffractometer (Philips) in the 2 θ 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 #74-2081 [7].

Sample/ Parameters	t (nm)	a (Å)	V (Å ³)	d _X (gm/cm ³)	d _B (gm/cm ³)	% P	G (nm)	S (cm ² /gm)
NiFe ₂ O ₄	28	8.337	579.47	5.387	3.762	30.16	58	15.57

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



3.2 Morphological analysis

Morphology of the prepared nickel ferrite nanoparticles was studied through the scanning electron microscopy (SEM) technique. The SEM image of the nickel ferrite nanoparticles is shown in fig. 2. The SEM image shows the spherical morphology with some agglomeration. The grain size was calculated using linear intercept method. The value of grain size (table 1) is in nanometric range showing the nanocrystalline nature of the prepared sample. The specific surface area is calculated using the standard relation and the value of the same is given in table 1.

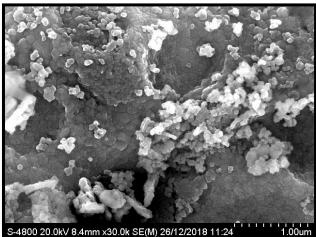


Fig. 3 SEM image of nickel ferrite nanoparticles

IV. CONCLUSIONS

Nanoparticles of nickel ferrite were successfully prepared using chemical coprecipitation method. The analysis of X-ray diffraction pattern confirms the formation of single phase cubic spinel structure. The lattice constant, X-ray density and unit cell volume are closely agreed with the reported values. SEM image show spherical grains with average grain size 58 nm.

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