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Solid State Synthesis of Nickel Ferrite ($NiFe_2O_4$) and their Characterization

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Abstract

Nanoparticles are small molecules with size ranging between 1to 100 nm. These find usage in wide range of industries from agricultural, biomedical, environmental and food. In the present work we used two methods first is practical method and second is green approach for the synthesis of nickel ferrite nanoparticles under chemical co-precipitation method. The synthesized powders were characterized by using X-Ray Diffraction (XRD). Crystallite size, X-Ray density, lattice constant and other lattice parameter also calculated. It reveals the presence of inverse spinel structure of ferrites but expansion of lattice dimension was observed in the case of nanoparticles which is synthesized by green approach because there is left shift in XRD peak as compared to nanoparticles which is synthesized by practical method.

Keywords: Ferrites, Co- Precipitation, Nanoparticles, Lattice parameter, XRD.

INTRODUCTION

Ferrites have gained importance because they possess the combined properties of magnetic materials and insulator. The soft ferrites with their tailorable properties have some stringent requirements[1, 2]. There are strict standards for the soft ferrites because of their customizable features. Soft ferrites have found excellent uses in microelectronics, including magnetic core memory systems and wave absorbers. Since fine-grained powder ferrites exhibit acute square hysteresis loop behavior and great stability in comparison to conventional approaches, they are useful in a wide range of microwave applications, including multilayer chip inductors. Ferrites with varying compositions have been produced using various substitutions to adjust their electrical, magnetic, and super magnetism characteristics[3-6]. The magnetic properties of ferrites are attracting a great deal of attention because of their versatile properties and applications in electronics, magnetic storage devices, and ferrite wave absorber and humidity sensors[7, 8].

Numerous wet chemical techniques, including as co-precipitation, sol-gel, combustion, hydrothermal, gel-assistant hydrothermal route, thermolysis, and micro emulsion, can be used to make ultra-fine ferrite powder[9-12]. Using a low process temperature and the co-precipitation approach, we created the nickel ferrite nanoparticles. The advantages of this technology include easy preparation, economical use of resources, and a mild chemical process that yields a very fine and uniform powder. Many researchers synthesized the nickel ferrite nanoparticles by different methods and at high temperatures only. But we synthesized the nickel ferrite nanoparticles at low temperature.

We synthesized two samples of Nickel Ferrite. In first Sample we used acetone and warm water for washing the precipitate but in second sample we only used warm water for washing the precipitate. So, we can conclude that the second sample is Green Synthesized. Because we use only water as a solvent and for washing the precipitate we also use only water.

EXPERIMENTAL

1. By Practical Method:

In this method the Nickel Ferrite Nanoparticles were synthesized by co- precipitation method. For this we have to prepare 0.4M solution of Anhydrated Ferric chloride ($FeCl_3$) and 0.2M solution of Anhydrated Nickel chloride ($NiCl_2$). Mix the both solution in a beaker and put the beaker in a magnetic stirrer and stir the solution but without heating. Then add 3M Sodium hydroxide solution and maintain the pH upto 11.5 to 12. The formed precipitates are washed with warm water and followed by acetone for many times. Then the precipitates are collected, dried and kept in oven for 30min at 80°C and then for 4 hours at 100°C to get the Nickel Ferrite Nanoparticles.

2. Acetone Free (Green Approach):

In this method also the Nickel Ferrite Nanoparticles were synthesized by co- precipitation method. For this we have to prepare 0.4M solution of Anhydrated Ferric chloride ($FeCl_3$) and 0.2M solution of Anhydrated Nickel chloride ($NiCl_2$). Mix the both solution in a beaker and put the beaker in a magnetic stirrer at constant temperature of 80°C for 45min and with that drop wise add 3M solution of Sodium hydroxide and also maintain the pH upto 11.5 to 12. Then formed precipitates are washed only with warm water for many times. Then the precipitates are collected, dried and kept in oven for 4 hours at 100°C to get the Nickel Ferrite Nanoparticles.

RESULTS AND DISCUSSION

For both methods the crystallite size of the Nickel Ferrite Nanoparticle sample were analyzed using powder X-ray diffractometer (XRD) using CuKα (λ= 0.154nm) radiation. The scanning was done in the 2θ range from 10° to 100°. Miller indices were also calculated and the lattice constant ‘a’ was computed using d values and with their respective [hkl] parameters.

Debye- Scherrer formula is used for measuring the crystallite size of Nickel Ferrite Nanoparticles[13],

$$D_{XRD} = K\lambda / \beta \, \mathrm{Cos}\theta$$

Where, λ= Wavelength of x-ray (0.154nm)

β= Full width at half- maximum in degree in the 2θ scale

θ = Bragg angle

And by using Bragg's Law we have calculated d spacing and after that we have calculated the value of lattice constant 'a' for both samples.

Bragg's Law; $n\lambda = 2d\sin\theta$

For lattice constant 'a'

$$d = \frac{a}{\sqrt{h^2+k^2+l^2}}$$

For X-ray Density calculation the following formula is used[14];

XRD Density = $8M/ Na^3$

M= Molecular weight of the sample (234.38g)

N= Avogadro number ($6.023*10^{23}$)

a= Lattice constant in angstrom

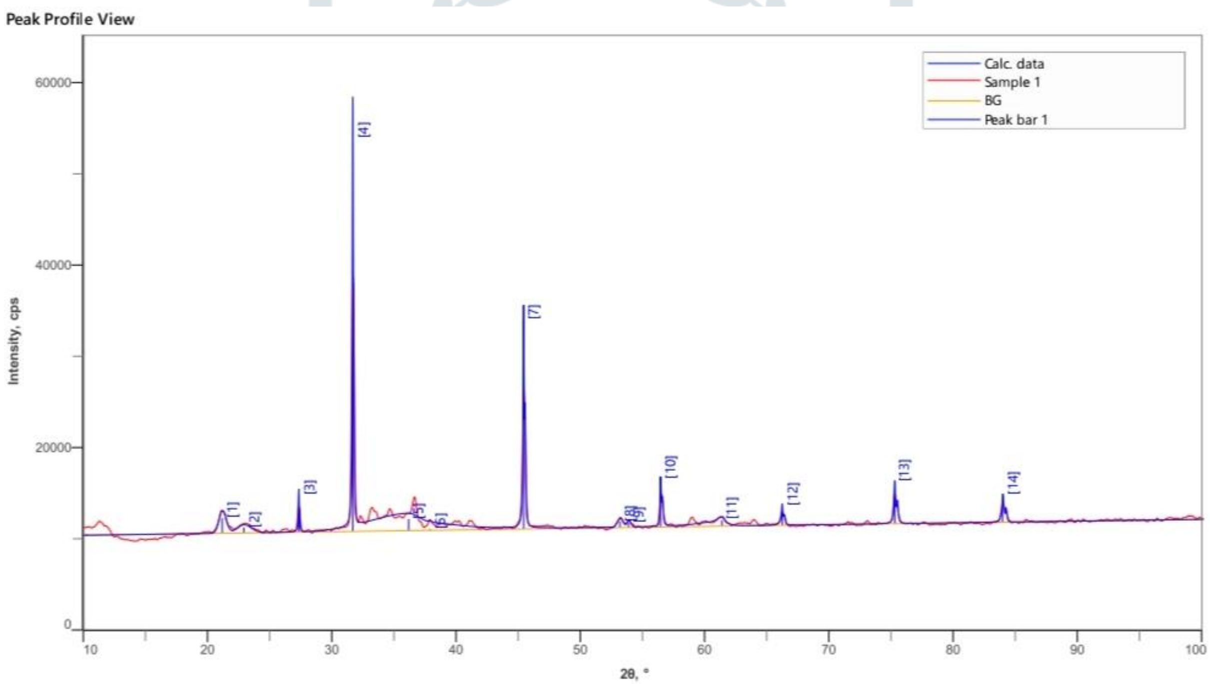

Fig.1. XRD pattern of the $NiFe_2O_4$ nanoparticles (sample 1)

S.No.	2θ (in degree)	d spacing (in angstrom)	Lattice constant 'a' (in angstrom)	[hkl]
1.	21.18	4.192	5.9283	[110]
2.	22.92	3.878	5.484	[110]
3.	27.336	3.2599	5.6445	[111]
4.	31.6707	2.82290	4.8894	[111]
5.	36.16	2.482	4.2988	[111]

6.	37.88	2.373	4.1100	[111]
7.	45.410	1.99564	3.9913	[200]
8.	53.16	1.721	3.848	[210]
9.	53.85	1.7040	3.8034	[210]
10.	56.420	1.62954	3.6436	[210]
11.	61.35	1.5099	3.6977	[211]
12.	66.211	1.4103	3.4538	[211]
13.	75.263	1.26158	3.0896	[211]
14.	83.933	1.15194	3.0468	[220]

Average value of lattice constant ‘a’ for sample 1 which is synthesized by practical method is **4.2092 angstrom.**

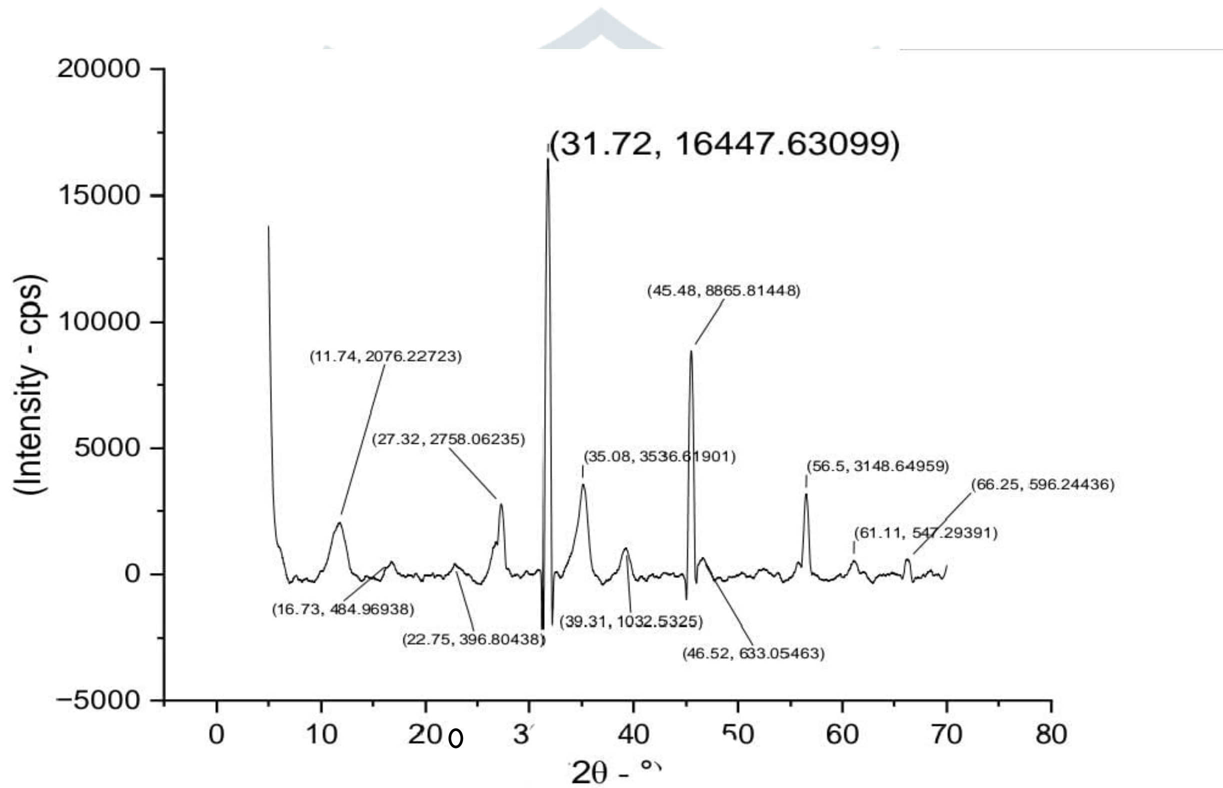

Fig.2. XRD pattern of the $NiFe_2O_4$ nanoparticles (sample 2)

S.No.	2θ (in degree)	d spacing (in angstrom)	Lattice constant ‘a’ (in angstrom)	[hkl]
1.	11.74	7.5	10.605	[110]
2.	16.73	5.3	9.1796	[111]
3.	22.75	3.9	7.8	[200]
4.	27.32	3.3	7.3788	[210]
5.	31.72	2.8	6.2608	[210]
6.	35.08	2.6	6.3674	[211]
7.	39.31	2.3	5.6327	[211]
8.	45.48	2.0	5.656	[220]
9.	46.52	2.0	5.76	[220]

10.	56.50	1.6	4.8	[221]
11.	61.11	1.5	4.743	[310]
12.	66.25	1.4	4.6424	[311]

Average value of lattice constant ‘a’ for sample 2 which is synthesized by acetone free method which is green approach for the synthesis of nickel ferrite nanoparticles is **6.568 angstrom.**

S.No.	XRD Parameters	Sample 1	Sample 2
1.	**K**	0.98	0.98
2.	**λ (in angstrom)**	1.54	1.54
3.	**Peak position (2θ) (°)**	36.16	35.08
4.	**FWHM (β) (°)**	5.21	25.5859
5.	**Crystallite size (nm)**	0.3047	0.062
6.	**Lattice constant ‘a’(in angstrom)**	4.2092	6.568
7.	**X- ray Density(kg/m^3)**	4.174	1.099

CONCLUSION

Nickel Ferrite Nanoparticles have been successfully synthesized by co – precipitation method at low temperature and successful formation of inverse spinel structure confirmed by X- ray diffraction analysis but in case of sample (2) expansion of lattice dimension was observed with decrease in crystallite size because there is left shift in XRD peaks as compared to the sample (1). The lattice plane, lattice constant, x-ray density and crystallite size in nm were also calculated. In second method some other lattice planes were found because this method is acetone free so we can say that it is a green approach for the synthesis of nickel ferrite nanoparticles because in this method we didn’t use any kind of chemical that is harmful to environment.

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