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Research Article

Nanocrystalline Ferrite (MFE_2O_4) For Photocatalysts and Thermal Properties by Homogeneous Co-Precipitation Technique

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Abstract

In this current research work, the preparation and properties of nickel ferrite nanoparticles were studied. Using nickel and ferric nitrates and citric acid, $NiFe_2O_4$ nanoparticles were prepared by a simple and cost-effective sol-gel auto-combustion method. The material used for this purpose was in the form of powder and also reagent grade chemicals were used for this manufacturing. Then the resultant powder was crushed into particles by using mortar and pestle. The phase formation of the $NiFe_2O_4$ was investigated by XRD and SEM techniques. X-ray diffraction (XRD) measurements were made it possible to approve the structural properties of the prepared samples. The morphology of these samples was explored through scanning electron microscopy (SEM). Thermal properties were studied by the thermal analyzer. The photocatalysts in an aqueous solution were studied under directly visible irradiation of the ferrite sample. After which the photocatalytic activity of the resultant product was observed by UV visible spectrometer.

Keywords: *Nanoparticles, X-ray diffraction, scanning electron microscopy, photocatalysts*

1. Introduction

Nano-science's fundamental job is to comprehend the processes and new characteristics of nano-systems and structure. It is possible to achieve new structural characteristics by decreasing their size, H. Laser emitters, quantity dots, nanotube of carbon, and thin films. Nano-science is manufacturing strategies by restricting their shape and size, and their representation on nano-scale apps and their efficiency varies on a big scale from that(Lapshin, 2011). Nickel ferrite ($NiFe_2O_4$) has an inverse spinel structure. The location of the divalent cations (Ni^{2+}) in the crystal structure is closely related to the magnetic properties of the nickel ferrite. However, nickel ferrite shows super-paramagnetic behavior and it has various applications such as gas-sensor, magnetic fluids, catalysts, magnetic storage systems, photomagnetic materials, magnetic resonance imaging, site-specific drug delivery, and microwave devices (McGaraughty et al., 2007). The properties of the synthesized materials are influenced by the composition and microstructure, which are sensitive to the preparation methodology used in the synthesis.

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Various methods such as citric acid combustion method sol-gel auto combustion method organic gel– thermal decomposition method, hydrothermal method, co-precipitation method, gel-assistant hydrothermal route thermolysis, wet chemical co-precipitation technique self-propagating microemulsion, and microwave synthesis have been developed to prepare nanocrystallite nickel ferrite (Yang et al., 2010). The morphology and particle size of the prepared sample is determined by HR-SEM and TEM. This method has the advantages of simple preparation, cost-effective and gentle chemistry route resulting in ultra-fine and homogeneous powder (Shobana et al., 2009).

[Nurseries *al*. (2011) was described that nickel ferrite nanocrystals were prepared from an aqueous solution containing metal nitrates and poly (vinyl pyrrolidone) (PVP) as a capping agent. To stabilize the particles, they were thermally treated at various temperatures from 623 to 823 K at which calcination occurred, thereby stabilizing the particles, controlling the growth of the nanoparticles, preventing their agglomeration, and creating a uniform distribution of particle sizes. The characterization studies were conducted by X-ray diffraction (XRD), Fourier transforms infrared spectroscopy (FT-IR), and transmission electron microscopy (TEM). Mahmoud *et al*. (2013) was used that nanocrystalline nickel ferrite

was synthesized from its stoichiometric metal nitrates and urea mixtures, using a microwave-assisted combustion method. The process was a convenient, inexpensive, and efficient method for the formation of $NiFe_2O_4$ nanomaterials. Hajaliloue*t al*. (2014) was that $NiFe_2O_4$ nanoparticles were synthesized by a mechanochemical reaction of NiO and Fe_2O_3 powders in a high-energy planetary ball milling machine. The XRD characterization results suggested that in the case of nano ferrite, milling up to 18 h formed $NiFe_2O_4$ particles of 10 nm with some residual Fe_2O_3 particles through a solid-state reaction. By extending the milling time to 30 h, the amorphous phase of $NiFe_2O_4$ was produced due to the high energy released during mechanical activation. Vasic*et al*. (2014) was described nickel ferrite nanoparticles were annealed to find the dependence of electric/magnetic properties on crystallite size. The following correlations of crystallite size with physical parameters were found: (a) lattice parameter decreases with the increase in size and it reaches value for bulk counterpart approximately for crystallites bigger than 7 nm, (b) ac electrical resistivity at room temperature increases with the increase in crystallite size, (c) for crystallites of ∼7 nm or smaller electrical resistivity has a maximum value at 50 °C, (d) the real part of permittivity at a selected frequency generally decreases with the increase in crystallite size and (e) magnetization increases with the increase in crystallite size. Deviation of stoichiometry, cation polyvalence, and cation redistribution with annealing are the main factors that influence the physical properties of Nickel ferrite nanoparticles.

Sutka*et al*., (2015) was used so–gel auto combustion to synthesize nickel ferrites of p-type and n-type conductivity by controlling their lative amounts of nickel and iron during synthesis. The obtained samples had been characterized by XRD, FE-SEM, electrical measurements, and XPS. We observe huge differences in the effect of grain size on the electrical resistivity between the p-type and the n-type material when the grain size increases from nano to micro-scale during annealing at temperatures from 900 ^oC to1300 ^oC. Shanmugavel*et al.* (2015) was analyzed nanocrystalline forms of nickel ferrite ($NiFe_2O_4$) that have been synthesized with the aid of a single-step chemical combustion method using citric acid as fuel in the 1:1 ratio. The single-phase formation of nickel ferrite was confirmed through powder X-ray diffraction (XRD). Kumar *et al*. (2016) Research on nickel ferrite nanostructures had drawn a great interest because of its inherent chemical, physical and electronic properties. In this study, we have synthesized rhombohedron – like nickel ferrite nanostructure by a rapid microwave-assisted combustion method using ethylene deaminate triacetin acid as a chelating agent. X-ray diffraction, Venkatesha*et al*. (2016) was discovered the nickel ferrite nanoplatelets were successfully synthesized by a simple microwave-assisted combustion method using trisodium citrate as a fuel. The prepared sample was chemically and structurally characterized by different techniques and the magnetic behavior was studied by field dependent magnetization measurement. Tangcharoen *et al*. (2016) was told that nano-crystalline Nickel ferrite ($NiFe_2O_4$) was synthesized in sol-gel and combustion methods with different chelating agents like PVA, citric acid, and urea. In the synthesis, the amount of chelating agent was taken exactly equal to the optimal output mass of the nickel ferrite which was unlike to conventional sol-gel or combustion method of synthesis. The powder X-ray diffraction technique was performed to identify the structure of the nickel ferrite and crystallite sizes of these samples were measured. Vinosha*et al*., (2018) About the diverse environment, application of ferrites (MFe2O4 = Ni, Cu, Mn, and Sr) in scientific and industrial categories, it is vital to optimizing its properties and hence a facile homogeneous co-precipitation route was used to formulate MFe_2O_4 nanoparticles to study its morphological, optical and magnetic perspective. The enviable phase pure spinel nanoparticles were deliberated by X-Ray Diffractometer (XRD), Fourier Transform Infrared (FTIR), Laser Raman, Transmission Electron Microscopy (TEM), Brunauer–Emmett–Teller adsorption-desorption isotherm (BET), and Vibrating Sample Magnetometer. XRD depicts the phase formation, crystallite size, lattice parameter, and the speck size was calculated by Scherrer formula.

2. Methods and materials

The present study was conducted in the Department of Physics, University of agriculture, Faisalabad. The purpose of the study was to prepare fine nickel ferrites powder through the co-precipitation technique. The X-ray diffraction analysis was performed in government college university, Faisalabad. The materials used in the present study are listed below:

2.1 Chemicals

The following chemicals were used for the completion of research work, nickel chloride, ferric chloride $FeCl_36H_2O$, NaOH, and distilled water.

2.2 Apparatus

The apparatus used in research work is as follows.
The apparatus used in research work was beakers of different volumes such as 100 ml, 200 ml, 250 ml to make a solution of different salts, volumetric flasks, glass rods, funnels, burette, filter papers used for the filtration, electronic physical balance with 2-digit accuracy, Ph papers, magnetic stirrer, water bath, china dishes, oven, furnace, permanent magnet, and X-ray diffractometer.

Table 2.1: The Molecular weights of materials used in the study

Compound	Molecular weights (g/mol)
$NiCl_2$	129.5994 g/mol
$FeCl_3.6H_2O$	162.2 g/mol
NaOH	39.997 g/mol

2.3. Preparation of Samples

First of all, the apparatus like flask, beakers, burettes, and crucibles were cleaned with distilled water before initiating the synthesis of nanoparticles. This was carried due to avoid any contamination or pollutant in the samples. After that taken of nickel chloride (NiCl2) and ferric chloride was dissolved in 100ml of distilled water and stirred for half an hour on a magnetic stirrer. 4g of NaOH was dissolved in 100ml distilled water in another beaker and stirred for 1o minutes. The solution of NaOH was dissolved drop-wise to the above-prepared solution. Drop the solution NaOH until the PH of the solution was nearly 12. The PH of the solution was carried out with the help of PH paper. The solution was further stirred for 1 hour. After stirring, filter the solution by using filter paper. Precipitate collected on the filter paper was washed many times with distilled water. The prepared product was dried in an electric oven at a temperature of 60 °C for 6 hours to remove water contents. The sample was named sample 1, sample 2, etc. Pestle and mortar were used to grinded the sample after washing with distilled water. After samples were shifted into crucibles for annealing at 800°C for one hour in the furnace. The furnace was turned off and at the normal temperature, they were taken out from the furnace and put at room temperature for further cooling. At last, the fine material was obtained by grinding in pestle and mortar. All the prepared samples of ZnO were examined with the help of XRD to study their morphology.

Table 2.2: The quantities of nickel ferrites, ferric chloride, and NaOH are given in the below table.

Sr. No	$NiCl_2$	$FeCl_3.6H_2O$	NaOH
S-1	5.1839	12.976	4g
S-2	5.1839	12.976	4g
S-3	5.1839	12.976	4g

3. Results and Discussions

One method was adopted to synthesize nickel ferrite nanoparticles. By co-precipitation, three samples were prepared. In this method, nickel chloride, ferric chloride, and sodium hydroxide were used. Sodium hydroxide was acted as good material to produce precipitates. The equipment used in this method were cleaned up with the help of water. Water used to wash the apparatus should be condensed to protect the apparatus from dust or other impurities.

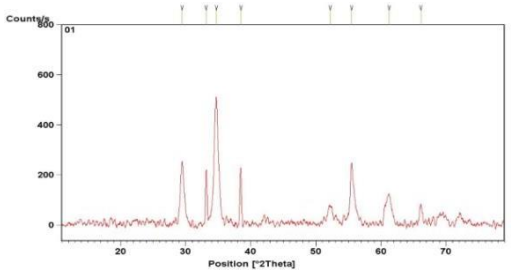
The synthesized materials were then subjected to the XRD technique to determine the structure of the material as well as the size of the material. The material should be in powder form to subject with XRD. The powder technique is favorable to examine the crystal materials. By using this method, diffraction peaks are obtained. Every material has its own particular peaks. $Ni_{0.4}Fe_2Fe_{0.8}O_4$were prepared by the co-precipitation method. The structural material was confirmed by XRD. It was observed that the sample is in pure form. No unwanted peaks were observed. Fine Ni ferrites powders were prepared by co-precipitation method using sodium hydroxide (NaOH) as a precipitating agent.

In this method nickel chloride, ferric chloride, and sodium hydroxide were used. Sodium hydroxide was acted as good material to produce precipitates. The equipment used in this method were cleaned up with the help of water. Water used to wash the apparatus should be condensed to protect the apparatus from dust or other impurities. The synthesized materials were then subjected to the XRD technique to determine the structure of the material as well as the size of the material. The material should be in powder form to subject with XRD. The powder technique is favorable to examine the crystal materials. By using this method, diffraction peaks are obtained. Every material has its particular peaks. $Ni_{0.4}Fe_2Fe_{0.8}O_4$were prepared by the co-precipitation method. The structural material was confirmed by XRD. It was observed that the sample is in pure form. No unwanted peaks were observed.

3.1 X-Ray Diffraction Studies

XRD data were used to determine the structural parameters of all the samples. This method gives information about the crystalline nature of the sample which includes miller indices, crystal structure, phase composition, nature of the sample (amorphous or crystalline). In this method, X rays are made to fall on the sample if the falling X rays satisfy Bragg's condition they get diffracted from the sample. The intensity of diffracted X-ray is plotted concerning scattering angle. Higher intensity ensures more atoms lie in that particular plane. XRD analysis of samples was conducted under the condition already described in materials and methods. XRD patterns of the prepared samples are given in the following figures. The relative intensities, d spacing, miller indices, and FWHM values were also obtained for each sample (arulmuruganet *al*., 2005).

Sample 1



Graph 3.1.1: XRD pattern of co- precipitated $Ni_{0.4}Fe_2Fe_{0.8}O_4$ferrite of sample S-1 at600°C.

Lattice constants

Table 3.1.1 Lattice constants of co- precipitated $Ni_{0.4}Fe_2Fe_{0.8}O_4$ nanoparticles for sample 1

Sr.No	Angle (2θ)	d-spacing	Miller indices	Lattice constant
1.	34.7121	2.58436	(3 1 1)	8.5713
2.	55.4596	1.65685	(3 3 3)	8.6092
3.	61.2326	1.51376	(4 4 0)	8.5631

Particle sizes

Table3.1.2 Particle sizes of co-precipitation $Ni_{0.4}Fe_2Fe_{0.8}O_4$ nanoparticles for sample 1

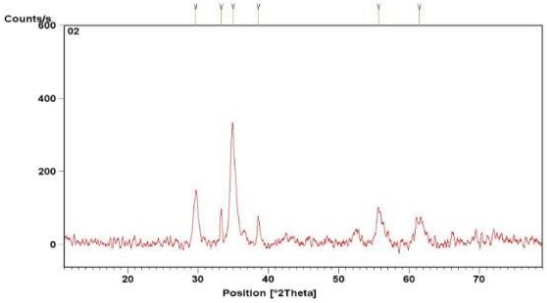
Sr.No	B=FWHM (Radians)	2θ	θ	T(nm)
1.	8.239	34.7121	17.3560	1.762
2.	5.4932	55.4596	27.7298	2.8511
3.	0.0164	61.2326	30.6163	9.820

X-ray Density

Table 3.1.3 Density of X-ray for $Ni_{0.4}Fe_2Fe_{0.8}O_4$ nanoparticles heated at 600°C of S-1

Serial No.	Angle (2θ)	Molecular Weight(M)	Lattice constant(a)	X-ray Density
1.	34.7121	176.82536	8.5713	3.716×10^{-24}
2.	55.4596	176.82536	8.6092	3.6825×10^{24}
3.	61.2326	176.82536	8.5631	3.74×10^{-24}

Sample 2



Graph 3.1.2: XRD pattern of co-precipitated $Ni_{0.4}Fe_2Fe_{0.8}O_4$ferrite of sample S-2 at 700°C.

Lattice constants

Table 3.1.4: Lattice constants of Co- precipitated $Ni_{0.4}Fe_2Fe_{0.8}O_4$ nano particles for sample 2

Sr.No	Angle(2θ)	d-spacing	Miller indices	Lattice constant
1.	29.6653	3.01151	(2 2 0)	8.5178
2.	38.5907	2.33308	(2 2 2)	8.0820
3.	61.4753	1.50712	(4 4 0)	8.5255

The mean lattice constant of sample 2 was 8.5.

Particle sizes

Table 3.1.5 Particle sizes of co-precipitation $Ni_{0.4}Fe_2Fe_{0.8}O_4$ nanoparticles for sample 2

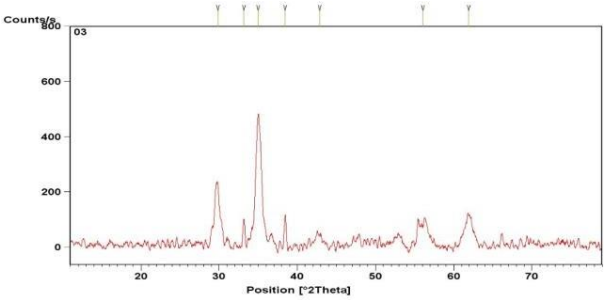
Sr.No	B=FWHM (Radians)	2θ	θ	t(nm)
1.	0.0109	29.6653	14.8326	13.153
2.	8.239	38.5907	19.2953	11.412
3.	0.02679	61.4753	30.7376	6.0191

X-ray Density

Table 3.1.6 Density of X-ray for $Ni_{0.4}Fe_2Fe_{0.8}O_4$ nanoparticles heated at 700°C of S-2

Serial No.	Angle (2θ)	Molecular Weight(M)	Lattice constant (a)	X-ray Density
1.	29.6653	176.8253	8.5178	3.8023×10^{-24}
2.	38.5907	176.8253	8.0820	4.451×10^{-24}
3.	61.47533	176.8253	8.5255	3.7920×10^{-24}

Sample 3



Graph 3.1.3XRD pattern of co- precipitated $Ni_{0.4}Fe_2Fe_{0.8}O_4$ferrite of sample S-3 at800°C.

Lattice constants

Table 3.1.7 Density of X-ray for $Ni_{0.4}Fe_2Fe_{0.8}O_4$ nanoparticles heated at 700°C of S-2

Sr.No	Angle (2θ)	d-spacing	Miller indices	Lattice constant
1.	34.9988	2.56384	(3 1 1)	8.5032
2.	42.8851	2.10887	(4 0 0)	8.4354
3.	56.0391	1.64109	(3 3 3)	8.5273

Particle Sizes

Table 3.1.8 Particle sizes of co-precipitation $Ni_{0.4}Fe_2Fe_{0.8}O_4$ nanoparticles for sample 3

Sr.No	B=FWHM (Radians	2θ	θ	t(nm)
1.	8.2390	34.9988	17.4994	17.638
2.	2.1971	42.8851	21.4425	67.774
3.	0.02746	56.0391	28.0195	5.717

X-ray Density

Table 3.1.9 Density of X-ray for $Ni_{0.4}Fe_2Fe_{0.8}O_4$ nanoparticles heated at 800°C of S-3

Serial No.	Angle (2θ)	Molecular Weight(M)	Lattice constant (a)	X-ray Density
1.	34.9988	176.8253	8.5032	3.8220×10^{-24}
2.	42.8851	176.8253	8.4354	3.9149×10^{-24}
3.	56.0391	176.8253	8.5273	3.7896×10^{-24}

3.2 Scanning electron microscopy (SEM)

SEM Stand for scanning electron microscopy. It is one of the widely used methods for material characterization especially to study their morphology and topological features. This method utilizes the emission of electrons from a cathode when the high potential is applied across its end. This gives information about the surface morphology of the specimen. Backscattered electrons are high-energy electrons resulting from the reflection or backscattering from the volume of the specimen. As they come from the comparatively more depth inside the specimen, they carry information about the topology of the specimen change in specimen property with the depth can also be analyzed with detection of backscattered electrons.

SEM analysis of sample 1

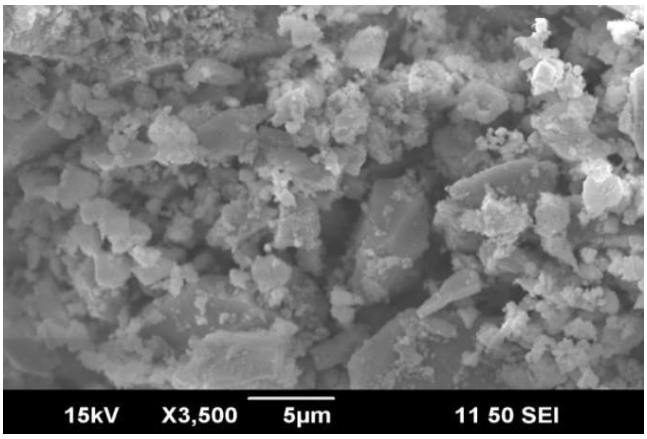


Figure 3.2.1: Scanning electron microscopy for $Ni_{0.4}Fe_2Fe_{0.8}O_4$ nanoparticle of S-1 co-precipitated at 600°C

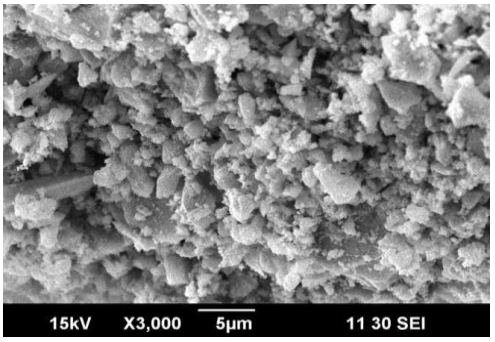


Figure 3.2.2: Scanning electron microscopy for $Ni_{0.4}Fe_2Fe_{0.8}O_4$ nanoparticle of S-1 co-precipitated at 600°C

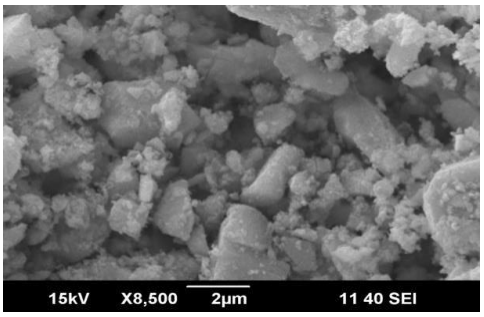


Fig 3.2.3: Scanning electron microscopy for $Ni_{0.4}Fe_2Fe_{0.8}O_4$ nanoparticle of S-1 co-precipitated at 600°C.

SEM analysis of sample 2

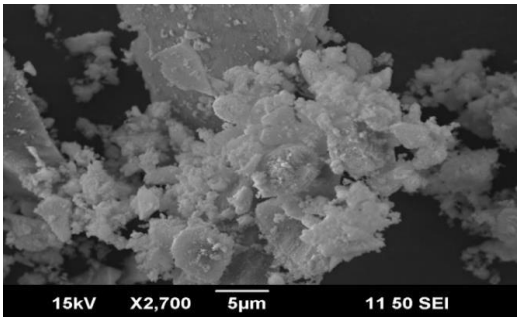


Fig 3.2.4: Scanning electron microscopy for $Ni_{0.4}Fe_2Fe_{0.8}O_4$ nanoparticle of S-2 co-precipitated at 700°C

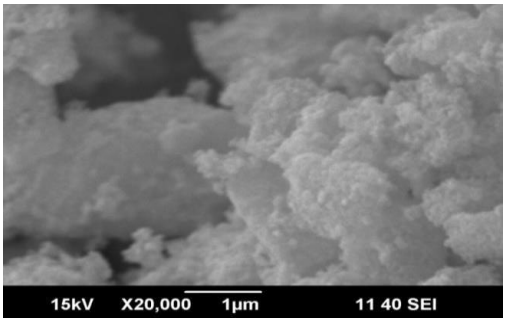


Fig 3.2.5: Scanning electron microscopy for $Ni_{0.4}Fe_2Fe_{0.8}O_4$ nanoparticleof S-2 co-precipitated at 700°C

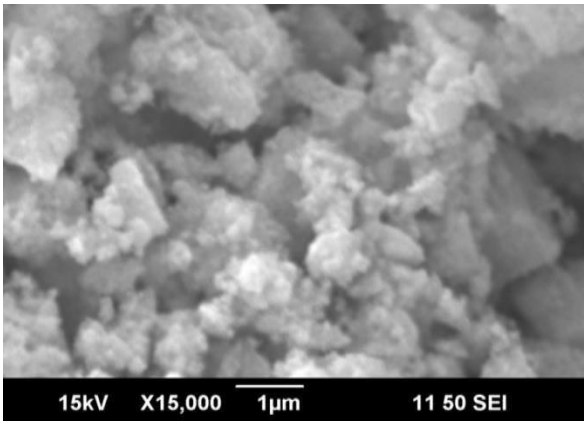


Figure 3.2.6: Scanning electron microscopy for $Ni_{0.4}Fe_2Fe_{0.8}O_4$nanoparticleof S-2 co-precipitated at 700°C

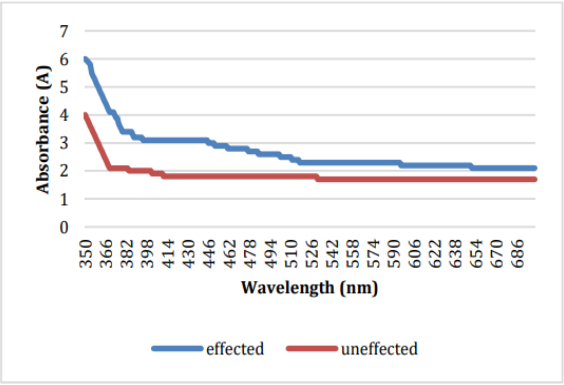
3.3 Photocatalytic activity

By measuring absorption spectra of prepared samples, the reaction of $Mg_{0.5}Zn_{0.5-x}Ni_xFe_2O_4$ on methylene blue, which is dye by nature, is observed. The photocatalytic degradation efficiency can be considered using equation;

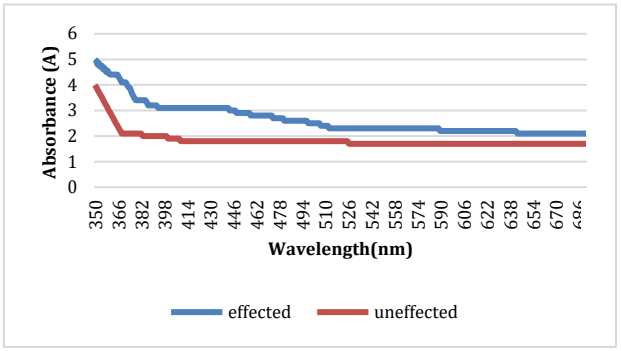
Degradation efficiency (%) $=[(c_o-c)/c_o]\times100$

Where c_o hereby be the initial concentration of dye and c is the concentration for certain expose time after degradation. For reasons of concentration (c) being directly proportional to absorbance (A), the c_o / c can be replaced by A_o / A. The reduction in absorption strength shows that dye degradation has increased (Kumar *et al.,* 2014).

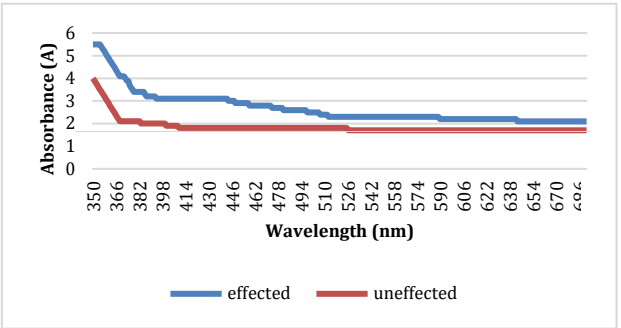
The results for dye degradations are shown in graphs 4.4, 4.5, and 4.6 respectively.



Graph 3.3.1 Photo-catalytic degradation of methylene blue using $Mg_{0.5}Zn_{0.375}Ni_{0.125}Fe_2O_4$ nano ferrites irradiated under light for the time interval of 40 minutes. The absorbing strength reduces as light radiation increases its exposure time. This shows an improvement in the effectiveness of degradation.



Graph 3.3.2 Photo-catalytic degradation of methylene blue using $Mg_{0.5}Zn_{0.25}Ni_{0.250}Fe_2O_4$ nano ferrites irradiated under light for a time interval of 40 minutes. The absorbing strength reduces as light radiation increases its exposure time. This shows an improvement in the effectiveness of degradation



Graph 3.3.3 Photo-catalytic degradation of methylene blue using $Mg_{0.5}Zn_{0.125}Ni_{0.375}Fe_2O_4$ nano ferrites irradiated under light for a time interval of 40 minutes. The absorbing strength reduces as light radiation increases its exposure time. This shows an improvement in the effectiveness of degradation.

Table 3.3.1 Absorption efficiency of $Mg_{0.5}Zn_{0.5-x}Ni_xFe_2O_4$ nanoparticles

Sample code	S-L Ratio	Adsorption efficiency(%)
S-A	0.6-50	65
S-B	0.6-50	58
S-C	0.6-50	61

In this research work nickel ferrite ($NiFe_2O_4$) nanoparticles at the different concentrations for (x= 0.4, 0.8) were successfully synthesized through co-precipitation technique at a constant temperature of 500. By x-ray powder diffraction analysis, the structural characterization of the $NiFe_2O_4$ samples was calculated. These studies were performed by calculating particle sizes, the lattice constant, the density of x-ray, and the volume for nickel ferrite ($NiFe_2O_4$) at different concentrations. The average lattice constant for all samples was calculated by wurtzite structure that in the range of 8.30-8.40 which close to 8.476. The average particle size for all samples initially increases then decreases gradually. The density of x-ray for all samples was calculated that in the range of 5.30x10-24-5.40x10-24.

Table 3.3.2 Comparative data of methylene blue degradation using different ferrites

Photo-catalyst	MB (mg/L)	Photo-catalyst (gm/L)	Irradiation time (min)	Source	Degradation %	Ref.
$Ni_{0.6}Zn_{0.4}Fe_2O_4$	20	0.30	90	UV light	94.00	(Padmapriya*et al.,* 2016)
$MgFe_2O_4$	10	0.60	180	Solar simulator (400–700 nm)	26.00	(Dom *et al.,* 2011)
$ZnFe_2O_4$	10	0.60	180	Solar simulator (400–700 nm)	32.00	(Dom *et al.,* 2011)
$CaFe_2O_4$	10	0.60	180	Solar simulator (400–700 nm)	28.00	(Dom *et al.,* 2011)
$BaFe_{12}O_{19}$	10	0.75	360	LED Lamp (400–700 nm)	26.00	(Valero *et al.,* 2016)

4. Conclusion

In this work, the structural properties of the $NiFe_2O_4$ nanoparticles have been investigated. The Ni ferrites have a significant influence on the structural properties of nanocrystalline $NiFe_2O_4$ prepared by using the co-precipitation method. Scanning electron micrographs indicate the increase in grain size up to x = 0.5 and later on it decreases with Ni content.

The Structural, morphology, and magnetic properties of the obtained materials have been studied. The formation of the single-phase is confirmed by the XRD technique. SEM and EDS measurements showed the chemical composition, element distribution, and homogeneity of the nanocrystalline powder.

5. References

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