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Synthesis of Nickel Ferrite Nano Particles Using Azadirachta Indica Leaves Extract and Its Antimicrobial Potential against Human Pathogens

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Abstract

Green synthesis of nanoparticles objectives are reducing generated waste and instigating sustainable processes. Green processes using minor reaction conditions and a harmless precursor has been emphasized in the development of nanotechnology for promoting environmental sustainability. This present study is proposed with an objective to synthesize $NiFe_2O_4$ nano particles using green synthesis method using Azadirachta Indica for the first time various characterization techniques such as Powder X-Ray diffraction, UV, FTIR Scanning electron microscopy and anti-microbial studies are done of the prepared $NiFe_2O_4$ nano particles. Anti-microbial studies are done in bacterias such as E.coli, Pseudomonas aeruginosa, Enterobacter, Bacillus cereus and Staph aureus. Antifungal activities of the nanao particles are studied using Candida albicans, Aspergillus niger and Aspergillus Flaves and the results are discussed with detailed explanation.

Key words: green synthesis, nanoparticles, XRD, UV, FTIR, SEM, Anti-Microbial studies, antifungal studies.

Introduction

Spinel ferrite has the common formula MFe_2O_4 where M represents a divalent metal ion. They were cubic in structure and have wide technological applications. Over the last decade, ferrite nanoparticles have attracted attention due to their high permeability, electrical resistivity and electromagnetic properties make them suitable for many purposes such as magnetic storage, microwave devices, biosensors, drug delivery, disease diagnosis, gas sensors, energy conversion, photo catalysts and magnetic separation (Wang 2011, Gupta 2017). Nickel Ferrite ($NiFe_2O_4$) is an industrially important soft ferrite with properties that make it ideal not only for soft magnetic core materials in power transformers but also as a photocatalyst. It has low coercivity and high electrochemical stability. $NiFe_2O_4$ has an inverse spinel cubic structure where Fe^{3+} ions occupy both tetrahedral (A) and octahedral sites (B), and Ni^{2+}ions occupy octahedral sites (B). Spinel ferrite nanoparticles has been developed intensively using several preparation techniques such as sol-gel combustion and Co-precipitation (Hirthna2018, Joshi2014), combustion (Phadatare 2013), solvothermal (Dong2015), thermal decomposition and seed growth (Lasheras2016), hydrothermal synthesis (Tang 2014), solid-state reactions([Shahid 2013), and honey-mediated sol gel combustion (Yadav 2017). Due to growing ecological concerns, there is a growing interest in green synthesis of inorganic materials in which simple environmentally friendly methods are used. Nano particle are prepared using natural extracts as capping agents and water as a solvent. Spinel's ferrites are produced in this method have a smaller particle size are stable, smooth and have a homogenous structure. In this work we made an attempt to synthesis $NiFe_2O_4$ nano particles using leaves and the results are reported.

Synthesis of $NiFe_2O_3$ Nanoparticles Using Azadirachta Indica leaves

The synthesis of $NiFe_2O_4$ nano particles consists of two parts which is the preparation of leaf extract and the second part is the synthesis of $NiFe_2O_4$ nano particles.

Preparation of Leaves extract

Leaves of Azadirachta Indica (neem) are collected from the regions around Tirunelveli, Tamilnadu, India. Fresh and healthy leaves are weighed and are thoroughly washed with deionised water to remove dust particles and sun dried to remove moisture content and these leaves were cut into small pieces. Further, the purified neem leaves was mixed with deionised water in a conical flask and kept in water bath at constant temperature of about for 3 hours. Later the obtained leaf extract was vacuum filtered through whatmann filter paper.

Synthesis of Nickel Ferrite Nanoparticles

. Ferrous sulphate heptahydrate ($FeSo_{4.}7H_2O$) and Nickel chloride ($NiCl_2$) are used as precursor for synthesizing nickel ferrite nanoparticles solution. The solution was stirred on a rotatory shaker for 8 hours. The immediate appearance of a black discoloration within the mixture indicates the formation of nickel ferrite nano particles. The resultant mixture was filtered using the whatmann filter paper and the precipitate obtained was cleaned twice by deionsed water and then kept in a hot air oven under the temperature of 300º C for 24 hours. The resulted sample was calcinated using muffle furnace at 800°C for 4 hours. Characterization analysis of biologically synthesized nickel ferrite nano particles are carried out using XRD,UV, FTIR, SEM and antimicrobial studies.

Results and Discussions

In order to get the information about various properties of prepared nanoparticles,

various characterization studies have to be done. The structural properties of nanoparticles are

analyzed from Powder XRD studies, UV and FTIR. The morphology of the nanoparticles is analyzed from SEM analysis. The inhibition growth of microorganism such as Ecoli, Pseudomonas, Enterobactor, Bascillus and Candida albicans, Aspergillus niger and Aspergillus Flaves are analyzed using anti- microbial studies.

Powder X- ray Diffraction

X-ray diffraction is the most common study to analyse the crystal structure of nanoparticles. The grain size and the dislocation density structure of the nanoparticles are investigated with a powder

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diffractometer with radiation at a diffraction angle (θ) between 20º to 80º.From the powder X-ray spectrum, finely sharp peaks are observed, which indicates the crystalline perfection of the $NiFe_2O_4$ nanoparticles. For the $NiFe_2O_4$ nanoparticles the peaks observed 30.28°, 33.151°, 35.635°, 43.40°, 54.057°, 57.505°, 62.421°, 71.917°, 75.420°, 82.914°, 88.501° well matches with the (h k l) values and was represented in Fig 1. The peaks of the diffraction patterns are compared with the standard available data for the the obtained nano particles has confirmation of the structure, and the lattice constant a =b =c = 8.337 with the use of **JCPDS Card no 74-2081** (Binu 2011).

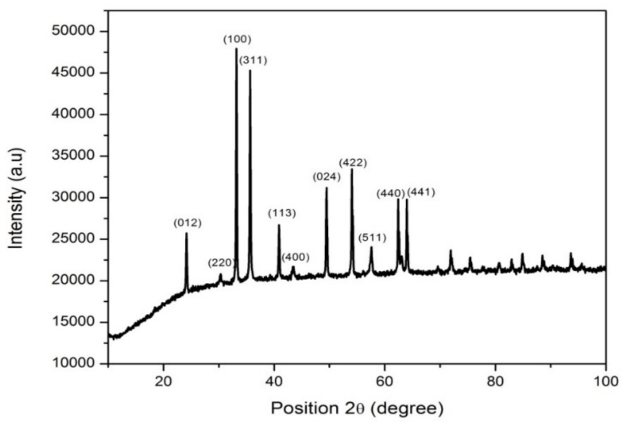

Fig 1: XRD Pattern of $NiFe_2O_4$ nanoparticles

It shows that the synthesized nano particles has a spinel shaped nano particles and has the structure with face center cubic system. From the XRD pattern, It is confirmed that the final product obtained is $NiFe_2O_4$ alone and no other phase or impurities is detected in the obtained pattern. The average crystallite size of the $NiFe_2O_4$ nanoparticles are around **53.116 nm** which are calculated from the full width at half maximum of the strongest peaks obtained from the XRD spectra and was determined by the Debye scherrer equation. Micro strain is a strain expressed in terms of parts per million and it is calculated around 0.3575. The dislocation density is a measure of the number of dislocations in a unit volume and it is around 0.0006562 nm^{-2} for the prepared nanoparticles.

UV- VIS Analysis

To study the optical properties of the $NiFe_2O_4$ nanoparticles, optical absorption spectrum is analysed. From the absorption spectrum, the energy band gap and the type of transitions can be determined. The room temperature absorption spectra of $NiFe_2O_4$ nanoparticles in the range of 200 to 700 nm are shown in the fig :2 from the absorption spectra, the absorption peak is in the range of around 606 nm.

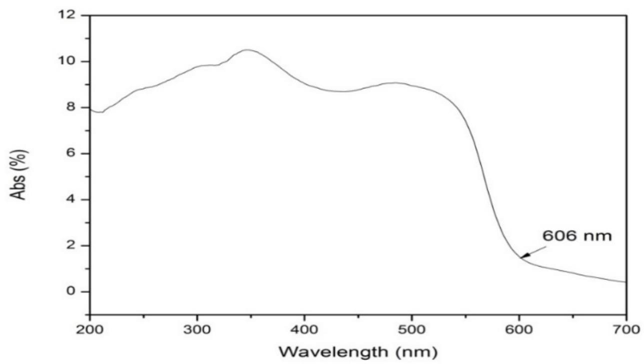

Figure 2: Absorption spectra of $NiFe_2O$ nanoparticles

The optical band gap of the nanoparticles can be calculated from the absorption spectra using Tauc's relation which is, $\alpha h\nu = (h\nu - E_g)^n$, where $h\nu$ is the photon energy, n is exponent factor which determines the type of transition causing the absorption and take the value $1/2$ and 2 depending whether transition is direct or indirect. $NiFe_2O_4$ nanoparticles are indirect band gap materials and the transition is indirect.

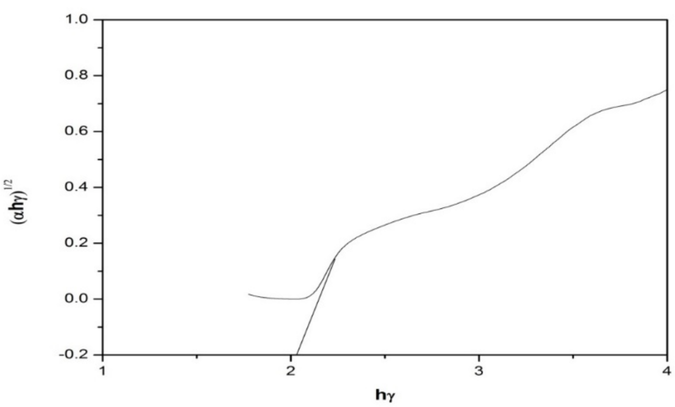

Fig 3: Tauc's plot of $NiFe_2O_4$

The bandgap energies Eg of $NiFe_2O_4$ nanoparticles are calculated cut off wavelength obtained from the absorption corresponding to the intercept point of the straight line a=0. Tauc's plots is shown in fig :3 From the Tauc's plot, the calculated band gap energies of $NiFe_2O_4$ nanoparticles is 2.1 eV.

Fourier Transformation Infrared Spectroscopy

In order to understand the chemical and Structural nature of the synthesized samples and the effect of leaf extract during synthesis $NiFe_2O_4$ nanoparticles, carried out by the FTIR analysis. Figure 3.6 shows the FTIR spectrum of $NiFe_2O_4$ nanoparticles in the range of 400 to 4000 cm^{-1}. The bands obtained at 518 cm^{-1} indicates Fe-O refer to intrinsic stretching vibrations of the metal at the tetrahedral site, M_{tetra} MO, were as the lowest band, usually observed at 430cm^{-1} is indicates Ni –O bond , is assigned to octahedral metal stretching M_{octa} MO. However, clear peak due to the broadening of this peak attributed to very small particles spinel ferrites.

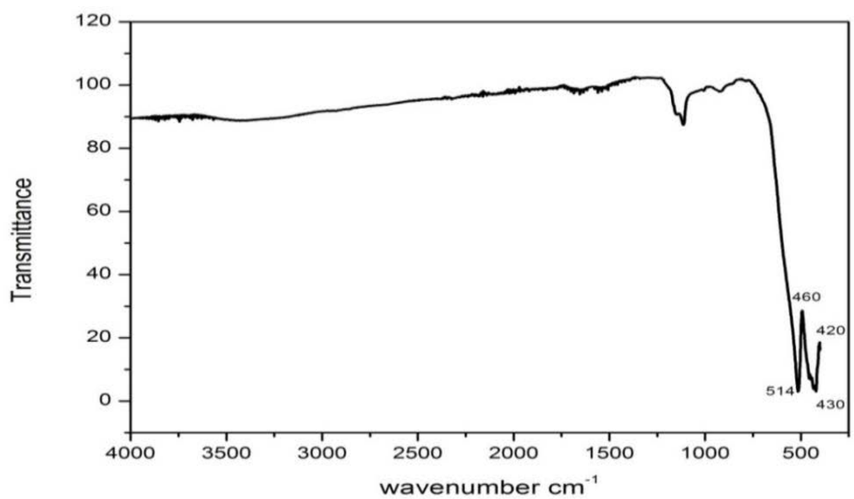

Figure 4: FTIR spectrum of $NiFe_2O_4$ nanoparticles

The bands obtained at 518 cm^{-1} indicates Fe-O refer to intrinsic stretching vibrations of the metal at the tetrahedral site, M_{tetra} MO, were as the lowest band, usually observed at 430cm^{-1} is indicates Ni –O bond , is assigned to octahedral metal stretching M_{octa} MO. However, clear peak due to the broadening of this peak attributed to very small particles spinel ferrites.

Scanning Electron Microscopy

The surface morphology of the synthesized nanoparticles is analyzed by the scanning electron microscopy analysis. Fig:4 shows the scanning electron micrograph of synthesized $NiFe_2O_4$ nanoparticles scanned by 15Kv electron beam in the range of 3 μm.

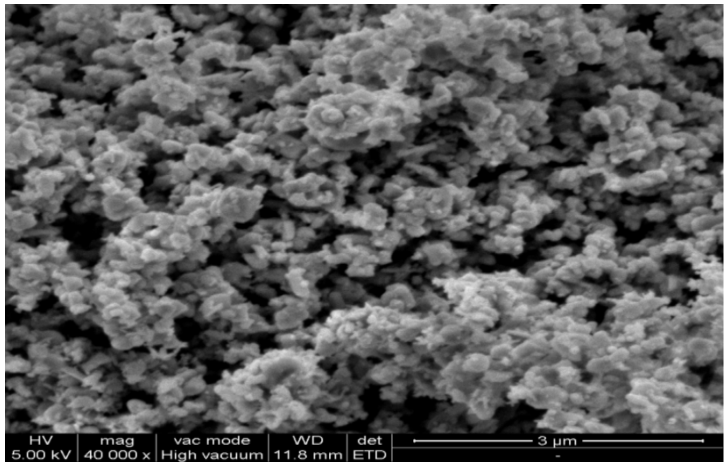

Fig 5: SEM image of $NiFe_2O_4$ nanoparticles

The SEM image shows that the equal distributions of nanoparticles are observed throughout the sample and mostly the particles are spherical shape at 3 μm range. The Overall morphology shows the uniform thickness and smoothness interface on the surface having prefect regular shape. SEM image shows the purity of the prepared nano sample.

Anti-Bacterial and Anti-Fungal Studies

The anti-microbial activities of the synthesized nickel ferrite nano particles were tested in the selected microbes.The bacteria's were cultured on agar plates with $NiFe_2O_4$ nano particles. Agar well diffusion method was implemented for the antimicrobial activity of nickel ferrite nano particles. The choice of the bacterium is based on the common infections affected in the community resulted from their various intakes present in the polluted water. Since the presence of these bacterium in the water are in larger proportion, there is a need to develop nano based incubators based on nano particles to control their growth and development in the hydro system. For the analysis of anti-bacterial effect, thee control used for this study is Amikacin. The anti-bacterial activities of the developed nickel ferrite nano particles were tested on the selected microbe ssuch as E.coli, Pseudomonas aeruginosa, Enterobacter, Bacillus cereus and Staph aureus in the agar based medium.

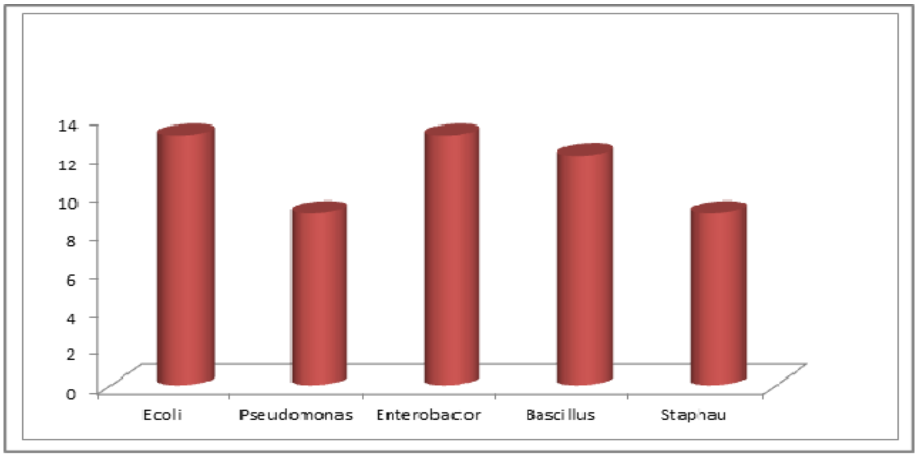

Fig 6:Anti – Bacterial analysis of $NiFe_2O_4$ nanoparticles

E.coli, is a gram negative bacteria and it is a facultative anaetobic, rod shaped bacterium that is commonly found in the lower intestine of warm blooded organism and it causes diarrhea and nausea (Tenaillon 2010). Enterobacter is gram positive rod shaped pathogenic bacteria (Adeolu 2016). They are non-encapsulated and facultative anaerobes that do not ferment lactose or do so slowly. Bacillus cereus is a Gram-positive, rod-shaped, facultative anaerobic, motile, beta-hemolytic, spore forming bacterium commonly found in soil and food (Felis 2009). Staph aureus is gram negative bacteria (Masalha2001). It is spherical shaped bacteria it is growing in the presence of 15% Nacl or 40% bile and is mainly associated with skin glands as mucus membranes of warm blooded animals. The results of the antibacterial effects were presented in the form of area of inhibition is shown in Fig 5,6.

The present investigation shows that the anti- bacterial effects of as $NiFe_2O_4$ nano particles E.coli, Pseudomonas aeruginosa, Enterobacter, Bacillus cereus and Staph aureus which has the inhibition diameter of 13mm, 9mm, 13mm, 12mm, 9mm respectively. the control is used in this study was amikacin. Amikan is used for infection of the membrances, spinal cord, urinary tract, blood, abdomen, bones, skin, joints, and lungs. The anti-bacterial activity of the E.coli, Enterobacter and Bacillus Pseudomonas are reported to be more observable effect than compared with that of pseudomonas and Staph aureus. This excellent antibacterial effect observed on these microbes clearly shows that the prepared nickel ferrite nano particles provides an effective source that enhace the microbial inhibition.

Anti-Fungal Studies

Candida albicans is a unicellular budding fungi which is principle pathogenic species causing candidiasis. It is often observed in HIV-infected patients (Gow 2017). Candida albicans is usually a harmless commensal of human beings but it can give rise to superficial infections of the oral or vaginal thrush. Sometimes healthy individuals are infected in skin or nail. . Candidiasis is the fourth most frequent hospital acquired infection worldwide it leads to immense financial implications. It will become causing life – threatening systemic and blood stream infections and mortality rate can be as 50%. There are few drugs such as amphotericin B, echinocandin, or fluconazole for systemic infections, nystatin for oral and esophageal infections, clotrimazole for skin and genital yeast infections. It affects a disease called "black mold" on fruits and vegetables such as grapes, onions, peanuts, apricots and is a common contaminant of food (Samson 2001). Recent evidence suggests that some A. niger strains do produce ochratoxin A. Aspergillus flavus is find worldwide as a saprophyte in soils and causes infection on many agriculture crops.

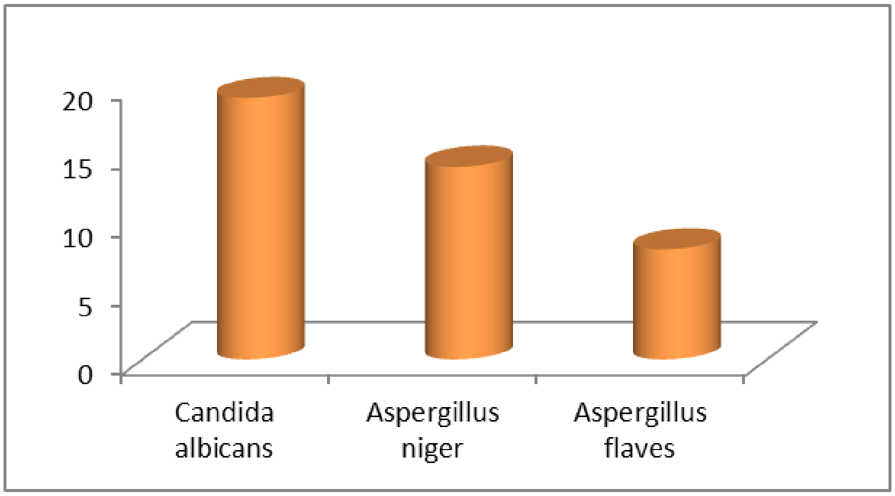

Fig 7 : Anti – fungal analysis of $NiFe_2O_4$ nanoparticles

Common hosts of the pathogen are cereal grains, legumes, and tree nuts. Specifically, A. flavus infection causes ear rot in corn and yellow mold in peanuts either before or after harvest (Amaike 2011). Contagion can be present in the agricultural field, pre-harvest, post-harvest, during storage, and during transport. A. flavus has the possible to infect seedlings by sporulation on injured seeds. Pathogen can attack seed embryos and cause infection in grains. It has decreases germination and lead to infected seeds planted in the agricultural field. The pathogen can also discolor embryos, damage seedlings, and kill seedlings, which reduce grade and price of the grains.

The anti-fungal activities of the nickel ferrite nano particles were tested on the selected microbes Candida albicans, Aspergillus niger and Aspergillus flaves. The control used in this study was nystatin. It is used for fungal inside of mouth and stomach. In the present investigation, the anti-fungal activity of the Candida albicans, Aspergillus niger and Aspergillus flaves was reported with more observable effect. The anti- fungal effects on as Candida albicans , Aspergillus niger and Aspergillus flaves increases with inhibition diameter from 19mm, 14mm and 8mm respectively shows in Fig: 6,8 Antifungal activity of nickel ferrite nano particles suggests that it can be a good alternative inhibition of treating the microbes based on fungal caused infections and the infected area.

Conclusion

$NiFe_2O_4$ nano particles are synthesized using the leaf extract of Azadirachta Indica and the crystalline structure of the nanoparticles is determined by the Powder X-ray diffraction studies. From the XRD pattern it is confirmed that the synthesized nanoparticles are of Cubic structure and average Crystallite size is around 53 nm . The room temperature absorption spectra of $NiFe_2O_4$ nanoparticles in the range of 200 to 700 nm. From the absorption spectra, the absorption peak is in the range of around 606 nm. Tauc's plot, the calculated band gap energies of $NiFe_2O_4$ nanoparticles is 2.1 eV. The bands obtained at 518 cm^{-1} indicates Fe-O refer to intrinsic stretching vibrations of the metal at the tetrahedral site, M_{tetra} MO, were as the lowest band, usually observed at 430cm^{-1} is indicates Ni –O bond , is assigned to octahedral metal stretching M_{octa} $\rightarrow$ MO. However, clear peak due to the broadening of this peak attributed to very small particles spinel ferrites.

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The surface morphology of synthesized nanoparticles are analyzed using SEM. The surface morphology of $NiFe_2O_4$ nano particles is spherical in shape. Investigation of anti-bacterial effect shows that the anti-bacterial activity of the E.coli and Enterobacter was reported with more observable effect than the control amikacin. The anti-fungal activity of the candida albicans and Aspergillus niger was reported with more observable effect than the control nystain. $NiFe_2O_4$ is the best alternative for the treatment of two fungi Candida albicans, and Aspergillus niger.

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