

RESEARCH ARTICLE

Green Synthesis of Iron Oxide Nanoparticles using the Leaf Extract of *Phyllanthus Niruri*

N. Gowrimanohari^{1*}, Rosemary Michael¹**ABSTRACT**

In recent years, nanotechnology has emerged as a start-of-the-art. with multifarious applications in a wide array of fields. Studies on green synthesis of nanoparticles moves forward these days. The present work involves the green method of synthesizing Iron oxide nanoparticles [Fe₂O₃] by *Phyllanthus Niruri* leaf extract and NaOH which acts as a precipitating agent. Furthermore, the green synthesized Iron oxide nanoparticles were characterized and its antibacterial activity was investigated. As this plant extract is more beneficial. it is energy efficient, low cost and environmentally friendly process than the bio- hazardous chemical synthesis. Iron oxide nanoparticles are gaining importance for their uses in environmental remediation technologies. The characterization of nanoparticles includes the IR, UV-Vis, and Size determination using SEM and XRD. The average crystalline size of the iron oxide nanoparticles was calculated by Debye's Scherrer formula. $d = 12.34\text{nm}$. The analytical studies revealed that the synthesized iron oxide nanoparticles almost have the identical size and morphology. Thus, the above studies concluded. the synthesized material was iron oxide nanoparticles.

Keywords: Iron oxide, *Phyllanthus Niruri*, Antibacterial, nanoparticle, XRD.

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1. Introduction

Nanotechnology is a science centered on molecules, atoms, supramolecular molecules aiming to create nano structures, which has an enhanced functionalities, and the term nanoparticle describes particulate matter which has the size ranging from 1-100 nm,^[1-2] the advantage of the nanoscale size offers significantly large surface area to volume ratio.^[3] Over the past few years, the synthesis in these nanoparticles is an important research in the modern field of material science on account of their distinctive potential applications in the field of magnetic optoelectronic, electronic and information storage. Nanomaterials can provide solutions to many technological and environmental challenges from the conversion in the field of solar energy, medicine and waste water treatment.^[4] Over the past few decades, there is an increase in the emphasis on the synthesis of metal nanoparticles and quantum dots.^[5,6] Since, they have the application of unique optical and electrical property. Green approach is a technique for the controllable synthesis of

nanoparticles of well-defined size and shape. Medical nanotechnology is an important application in the field of detecting the basic struggle with cancer, which is the core of research activities world- wide.^[7] Polysaccharide, tolensis, irradiation, polyoxometalate and biological methods are the various methods for the synthesis of green nanoparticles. Nanotechnology is a science of designing, making of nanostructures, application of Nanomaterials, and investigation of a various properties of materials, with their nano meter dimensions.^[8]

Some of the distinct advantages that biological synthesis protocols have conventionally used physically and chemically are

- Clean and eco-friendly method, as toxic chemicals are not used.
- Even during the large-scale production, the small nanoparticles can be produced.
- No use of the external experimental conditions like high energy and high pressure.

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1.1 Role of nano in Green synthesis

Green synthesis, characterization and applications of nanoparticles shows how eco-friendly nanoparticles are engineered and used. Bio-synthesis techniques employing plant extracts can be used to reduce and stabilize the metallic nanoparticles. The use of agricultural wastes, or plants and their parts has emerged as an alternative to chemical synthetic procedures as it does not require exaggerated process such as intracellular synthesis and multiple purification steps or the maintenance of microbial cell cultures. Here in this present work, I have used Ammonium iron (II) Sulfate which is an inorganic compound as this salt is the preferred source of ferrous ions, and the solid has a long shelf life, being resistant to oxidation.

2. EXPERIMENTAL PROCEDURE

2.1 Phyllanthus Niruri Leaf

Phyllanthus Niruri is a weed found in coastal areas. It is commonly known as gale of the wind or "stone breaker". And it is known for protecting the Liver. It is having high potential anticancer and antioxidant agents^[9] to cure viral hepatitis and increased vin-blastine cytotoxicity towards multi drug resistant cancer cells. It has been studied in the human population and has shown efficacy in preventing stone recurrence. It can be a remedy for HIV- AIDS.^[10]

2.2 Preparation of Phyllanthus Niruri Leaf Extract

Freshly collected leaves of Phyllanthus Niruri, were cleansed under the running water, and later they are allowed to wash with the distilled water. Then the leaves are taken separately from the stem. As they have, tiny fruits under their leaves, they are also removed cautiously. Then about 50 grams of cleaned leaves are set aside to boil with 100 ml of distilled water in a water bath for 3 hours, heated at 60°C. And the prepared solution was allowed to bring back to the room temperature. The leaf extract is then filtered through a filter paper. And the extract used for the further experiments.

2.3 Preparation of Fe³⁺ and Fe²⁺ Mixture

9.65 gram of ammonium iron (III) sulphate dodecahydrate was made to dissolve in 100ml of distilled water. 3.92 gram of ammonium Iron (II) sulphate hexahydrate was allowed to dissolve in 100 ml, of distilled water separately. Both the solutions were allowed to dissolved completely, So as to get 0.3 M solution of Fe (III) and 0.1 M solution of Fe (II). 5ml of

each solutions were mixed together by stirring, using the magnetic stirrer to get the iron salt mixture in 1:3 ratio.

2.4 Exploring Iron Oxide Nanoparticles

Optimum synthesis was done by adding together, the 1.2 ml of Phyllanthus Niruri leaf extract to 10ml of iron salt mixtures. [Mixture of 0.3M (NH₄)₂ Fe (SO₄)₂·6H₂O] and [0.1M (NH₄)₂ Fe (SO₄)₂·12H₂O] in 1:3 ratio. The reaction was made to stir under magnetic stirrer for 30 min at 30°C, for the conversion of iron species into Fe₂O₃, In NaOH was added drop wise and the stirring continued for 15 minutes. Thus, the conversion of reddish yellow colour extract to black indicates the formation of iron oxide nanoparticles. And the solution is kept undisturbed for the couple of days, for ageing and then the solution is centrifuged for 5 mins. The obtained precipitate is placed in oven to dry for 5 hours at 100°C, and a black powdered form is obtained by grinding it in a mortal pester.

2.5 Enactment of iron oxide nanoparticles

Iron oxide nanoparticles have an important application in the various fields. Synthesis of iron oxide nanoparticles has been extensively reviewed.^[1-5] The most important iron oxides were goethite, akageneite, lepidocrocite, magnetite, and hematite. Iron oxide nanoparticles consist of maghemite or magnetite particles with the diameter, ranging from 1 and 100 nano meters.

3. RESULTS AND DISCUSSION

3.1 X Ray Diffraction Analysis

The characterization of the synthesized iron oxide nanoparticles from Phyllanthus Niruri leaf extract was subjected to the XRD studies, and the XRD pattern of the iron oxide nanoparticles were shown in (Fig. 1) and their concentration has been determined using the X-ray diffractometer for the conformation of the nanoparticles. The mean crystalline size of iron oxide was found to be 12.34 nm, calculated by using Debye Scherer's Formula. The strongest peaks appeared at 2θ value of (35.4059), (62.5800), (13.6000). The positions and relative intensities of the reflection peak of Fe₂O₃, nanoparticles coated with XRD diffraction peaks of standard Fe₂O₃, samples indicating that the black coloured powders are nanoparticles.^[11] The structural parameters of iron oxide is given in Table 1.

$$D = (k\lambda / \beta \cos\theta)$$

Table 1: Structural Parameters of Iron Oxide nanoparticles

2θ (deg)	FWHM	Crystalline size D (nm)	Average crystalline size (nm)	Dislocation density (6x10 ¹⁵)	Microstrain (ε ₁₀ ³)
35.40	0.731	11.90	12.34	6.174	4.7485
62.58	0.760	12.78			
13.60	1.933	4.32			

3.2 Fourier Transform Infrared Spectroscopy Analysis

The presence of functional groups and biomolecules in the leaf extracts and within the Fe₂O₃ nanoparticles after synthesis are carefully weighted quantity of the synthesis nanoparticles are subjected to FTIR analysis. The FTIR spectra of Fe₂O₃ nanoparticles are synthesized by green method are recorded in the range of 500-4000 cm⁻¹ is shown in Fig. 2. The peaks at 1741.72 cm⁻¹ confirms the presence of C=O (strong) stretching, the presence of medium O-H bending at 1327.03cm⁻¹, the peaks at 3419.79 cm⁻¹ confirms the presence of N-H stretching and so on. The following Table 2 shows the wave number and their corresponding functional groups involved in Fe₂O₃ nanoparticles.

3.3 Scanning Electron Microscope (SEM) analysis

The SEM analysis has been performed to study the morphology and the size distribution for the synthesized

Table 2: Functional Groups in IR Spectra of Fe₂O₃

Wave number (cm ⁻¹)	Band Assignment
569	C-Br Stretching
2879.72	C-H Stretching
1327.03	O-H Bending
3672.47	O-H Stretching
1060.85	S=O Stretching
1741.72	C=O Stretching

iron oxide nanoparticles. The SEM image of iron oxide nanoparticles were taken in different resolution it is shown in Fig. 3. From the image it has been observed that the

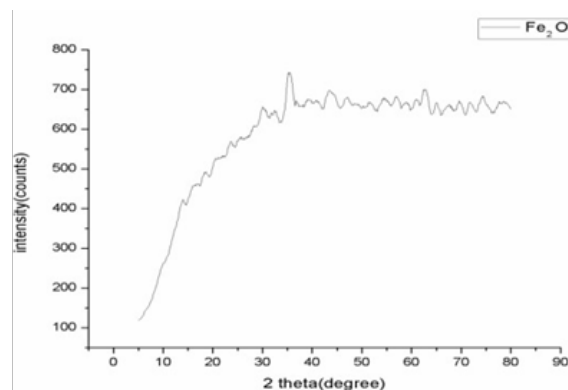


Fig. 1: XRD Pattern of Iron Oxide nanoparticles

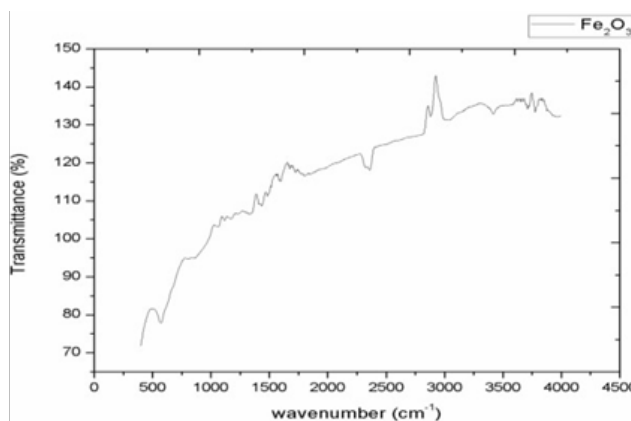


Fig. 2: FTIR Spectrum of Fe₂O₃ nanoparticles

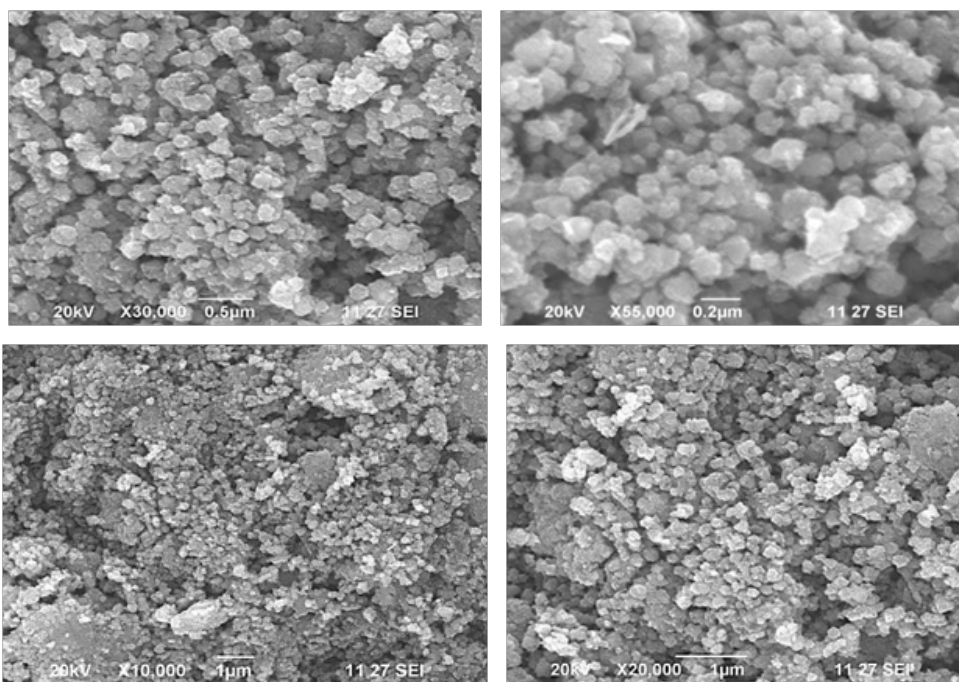


Fig.3 SEM images of Iron Oxide nanoparticles

shape of the prepared nanoparticles was tiny spherical like structure.

3.4 UV-Vis Analysis

Absorbance and transmission studies of iron oxide have been absorbed by using UV - visible spectrometer, and results are shown in Fig. 4. The colour change from reddish yellow colour indicated the formation of iron oxide nanoparticles UV spectroscopic analysis was done in the range of 190-1100nm. The absorbance spectrum of iron oxide nanoparticles in UV analysis is at 558 nm. Hence the band gap energy was found to be 2.2 e V by using the formula,

$$E = (hc/\lambda) \text{ eV}$$

The range of iron oxide is between (2.2 -2.7) eV.^[12] The Table.3 shows the various wavelengths and their corresponding absorbance.

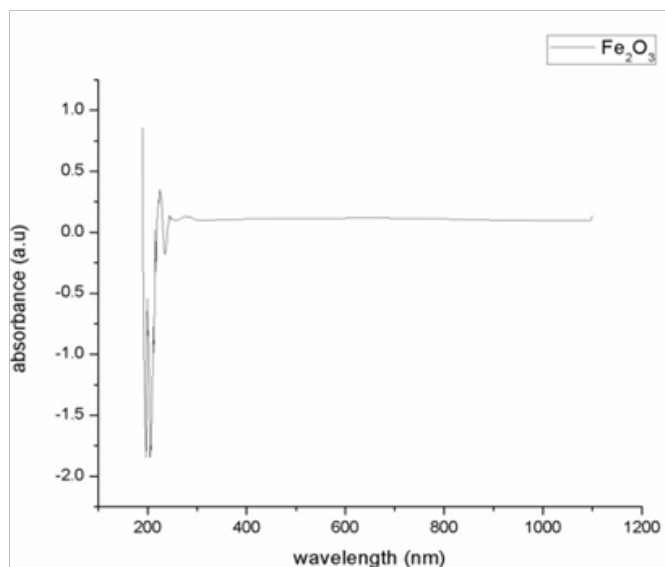


Fig. 4: UV-Vis spectra of iron oxide nanoparticles

Table 3: Optical parameter of Iron Oxide Nanoparticles

Wavelength (nm)	Absorbance (a.u)
279.00	0.149
310.00	0.097
256.00	0.098

3.5 Energy Dispersive X-ray Analysis (EDX) Analysis

The composition of elements of the explored iron oxide nano powder was examined by the Energy Dispersive X-ray Spectroscopy (EDX), a micro analytical technique used in association with SEM.^[13, 14] The EDX examine the X-ray emitted from the synthesized sample, when electrons bombarded on the surface of the sample. By measuring the intensity and energy of the signal, data about the

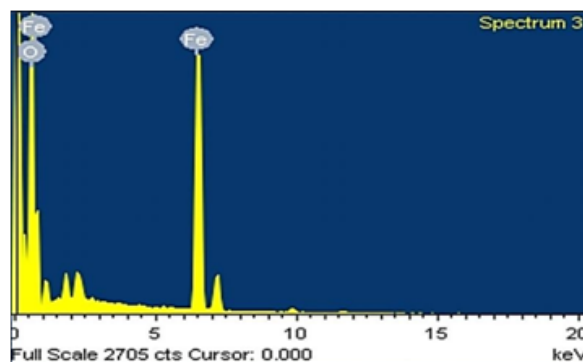


Fig. 5: EDX graphical representation of Iron Oxide nanoparticles

chemical composition was detected. The EDX spectrum shows frequency of X-rays in count of each energy-level. The peak's intensity determines the amount of the element in the sample. Since the EDX, demonstrated that explored Fe₂O₃ Nano-powders consists of 77.38 % of O- content and 22.62% of Fe-content without the presence of any other type of elements in Fig. 5.

3.6 Antibacterial Activity

Determination of antimicrobial activity by agar well diffusion method

The given sample was tested for antimicrobial activity by well diffusion method. Liquid Mueller Hinton agar media and the Petri plates were sterilized by autoclaving at 121 °C for about 30 minutes at 15 lbs pressure. Under aseptic conditions in the laminar air flow chamber, about 20 ml of the agar medium was dispensed into each Petri plate to yield a uniform depth of 4 mm. After solidification of the media, 18 hrs culture of Gram positive microorganisms such as Bacillus cereus (MTCC 430), Staphylococcus aureus (MTCC 3160), Gram negative micro organisms such as E. Coli (MTCC 1698)

Table. 4: Antimicrobial activity Iron Oxide Nanoparticles

S. No	Micro organisms	Zone of inhibition in Diameter (mm)			Std. Antibiotic (Gentamycin) 30 mcg/disc
		Control (100 µl)	50 µl	100 µl	
					30mcg/disc
1	Bacillus cereus	Nil	0	0	28
2	Staphylococcus aureus	Nil	0	0	29
3	Escherichia coli	Nil	0	0	27
4	Klebisella pneumoniae	Nil	0	0	27

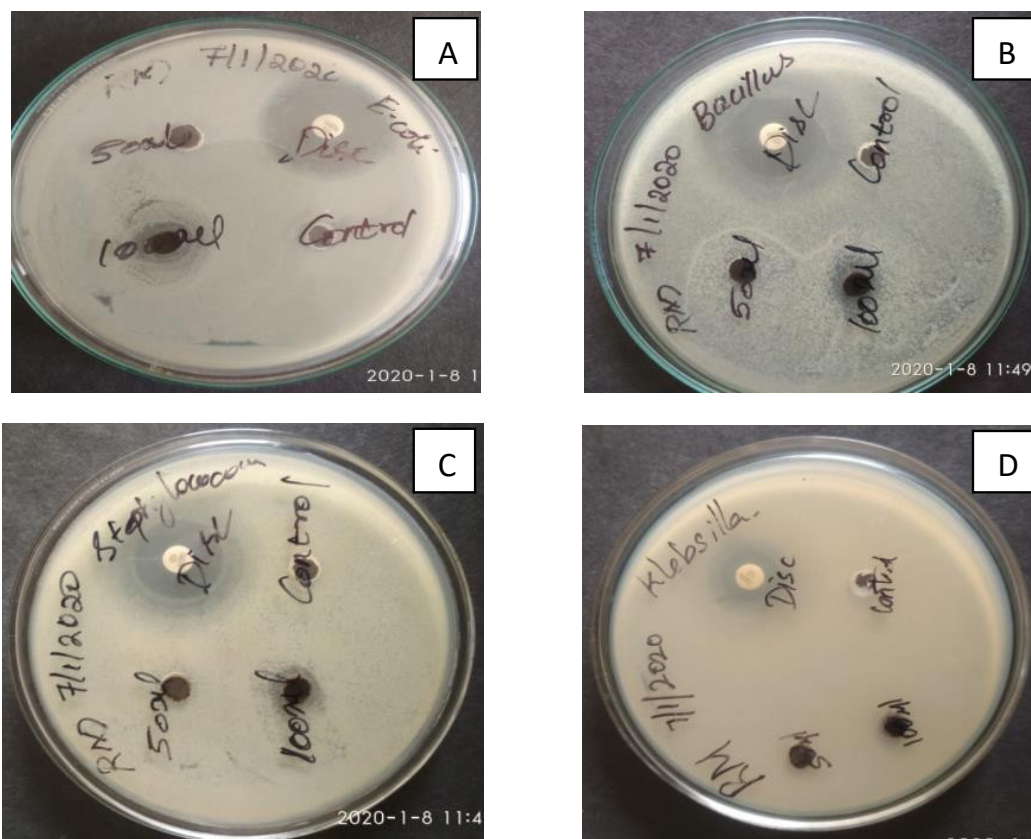


Fig. 6: Zone of Inhibition against Antibacterial Agents

and *Klebsiella pneumoniae* (MTCC10309) obtained from IMTECH, Chandigarh were swabbed on the surface of the agar plates. Well was prepared by using cork borer followed with loading of 50 pi and 100 pi of each sample to the distinct well with sterile distilled water as negative control and gentamycin (30mcg/disc) as positive control. The sample loaded plates were then incubated at 37°C for 24 hours to observe the zone of inhibition. Hence, for iron oxide nanoparticles the antibacterial assays are NIL.

4. CONCLUSION

The present synthesis is capable of producing Iron oxide nanoparticles via green method to demonstrate the importance of environmentally friendly preparation of nanoparticles. The IR, UV-VIS, SEM, XRD studies, revealed that the synthesized iron oxide nanoparticles have almost identical size and morphology. The FTIR spectra of Fe_2O_3 nanoparticles are synthesized by green method and are recorded in the range of 500-4000 cm. The XRD studies revealed that the mean crystalline size of iron oxide nanoparticles was found to be 12.34 nm. From the SEM image it has been observed that the shape of the prepared nanoparticles was spherical like structure. These studies concluded that the synthesis of iron oxide nanoparticles using plant extracts is more beneficial. The present investigation may be a definite contribution to green chemistry in general.

REFERENCES

1. D. Tiwari, J. Behari, P. Sen, Application of nanoparticles in waste water treatment, *World applied science Journal*, 3 (2008) 417-433.
2. M.S. Mehata, M. Manjumder, B. Mallik, N. Ohta, External electric field effects on optical property and excitation dynamics of capped cds Quantum dots (QDS) embedded in a polymer film, *J.Phy.Chem.C*, 114(37) (2010) 15594-15601.
3. M.S. Mehata Enhancement of charge transfer and quenching of photoluminescence of capped cds Quantum dots, *Sci. Rep.*, 5 (2015) 12056.
4. P.V. Kumar, S. Pammi, P. Kollu, K. Satyanarayana, U. Shameem, Green synthesis and characterization of Silver nanoparticles using *Boerhaavia diffusa* plant extract and their antibacterial activity, *Ind.crop.prod.*, 52 (2014) 562-566
5. K.S. Siddiqi, A. Husen, Recent advances in plant mediated engineered gold nanoparticles and their application in biological system, *J.Trace Elem. Med. Bio.*, 40 (2017) 10-23.
6. S. Senapati, A. Ahmad, M.I. Khan, M. Sastry, R. Kumar, Extracellular biosynthesis of bimetallic Au-Ag alloy nanoparticles, *Small*, 1(2005) 517-520.
7. T. Klaus, R. Joerger, E. Olsson, C.G. Granqvist, Silver -based crystalline nanoparticles, Microbially fabricated, *Proceedings of the National Academy of Science of the USA*, 96 (1999) 13611-13614.
8. M. Mahmoudi , S. Sant , B. Wang, S. Laurent , T. Sen, Superparamagnetic iron oxide (SPIONs): development, surface modification and applications in chemotherapy, *Advanced Drug delivery Reviews*, 63 (2011) 24-26.

9. A.L. Udupa , Diuretic activity of phyllanthus niruri (Linn) in rats, *J. Health.*, 2 (2010) 511–512.
10. Udo Schwetmann, M. Cornell Rochelle, *Iron Oxide in the laboratory: Preparation and characterization*, John Wiley and Sons, (2000).
11. Rochelle M. Cornell, Uolo Schwertzmann, *The iron Oxides structure properties, reactions, occurances and uses*, 14. John wiley and sons, (2003).
12. David. C. Fernandez–Remolar, *Iron Oxide, Hydroxides and oxy -hydroxides*, *Encyclopedia of Astrobiology*, (2014) 1268–1270.
13. A.M. Awwad, N.M Salem, *A green and facile approach for synthesis of magnetic nanoparticles*, *Nanoscience and Nanotechnology*, 2 (2012) 208–213.
14. Rahdar, M. Aliahmad, Y. Azizi, *NiO Nanoparticles: Synthesis and Characterization*, *Journal of Nanostructure*, 5 (2015) 145–151.