International Journal of Advanced Research in Biological Sciences
ISSN: 2348-8069
www.ijarbs.com
(A Peer Reviewed, Referred, Indexed and Open Access Journal)
DOI: 10.22192/ijarbs Coden: IJARQG (USA) Volume 9, Issue 8 -2022

Research Article

DOI: http://dx.doi.org/10.22192/ijarbs.2022.09.08.013

Green synthesis, characterization and antibacterial activity of iron oxide nanoparticles derived from *Solanum nigrum* leaf extract

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Abstract

In order to create iron nanoparticles, extract from *Solanum nigrum* was used. By using UV/Vis absorption spectroscopy, X-ray diffraction spectroscopy, and SEM examination, the produced iron nanoparticles were identified. Iron oxide nanoparticles exhibit a distinctive absorption peak in the UV/Vis spectrum between 200 and 300 nm. The average particle size of magnetite nanoparticles, as determined by the X-ray diffraction method, was found to be 24.1 nm. By using the well diffusion method, it was discovered that the synthetic iron nanoparticles have antibacterial action against harmful bacteria such *Staphylococcus aureus*, *Streptococcus pyogenes*, *Escherichia coli*, and *Klebsiella pneumoniae*. This biosynthetic method has been discovered to be economical, environmentally benign, and promising for use in a variety of fields.

Keywords: Iron nanoparticles, *Solanum nigrum*, antibacterial activity

Introduction

Utilizing materials at the nanoscale, or with a dimension smaller than 100 nm, is the subject of nanotechnology. The produced nanoparticles have particular features and are used in a variety of applications, including biological labels, sensors, health care devices, data storage, catalysis, pigment, and ion exchangers [1,2]. These nanoparticles are used in a variety of fields, such as food and agriculture, environment, medicine, and so on[3-5]. They have special physical, optical, chemical, electrical, and medical properties. Due to their superparamagnetic properties and employment as contrast agents in nuclear magnetic resonance, magnetic nanoparticles have recently become a major concern in the healthcare sector. They are also utilised for cancer hyperthermia treatment, sustained and targeted medication administration, etc. Utilizing materials at the nanoscale, or with a dimension smaller than 100 nm, is the subject of nanotechnology. They are also employed in cancer hyperthermia therapy, sustained and targeted drug delivery, and other applications. The two main requirements for a substance to be used in medicine are that it must be non-toxic and biocompatible. This section is very concerned with iron oxide nanoparticles, which are an inorganic transition metal oxide. Iron oxide nanoparticles have been created using a variety of techniques. This covers both physical and chemical procedures such as Co-Precipitation, Thermal Decomposition, Microemulsion, Hydro-thermal Synthesis, Sonochemical Synthesis, Microwave Method, etc. [6,8]

However, these techniques have a number of drawbacks, including high costs, slow production rates, hazardous chemicals, and by product contamination. A straightforward, effective, and environmentally friendly solution is needed to address this. This is made possible by the new "Green Chemistry" approach, which uses a safe and non-toxic process to create nanoparticles. Microbes and plant extracts are employed for this [9–11]. Plant extracts are chosen over microorganisms for the creation of nanoparticles because they may be produced more quickly and require less upkeep of cell cultures [12]. In the current study, iron oxide is synthesized in a green manner using a leaf extract from *Solanum nigrum* plants.

Materials and Methods

Synthesis of Fe_2O_3 nanoparticle

Preparation of *Solanum nigrum* leaf extract

Solanum nigrum leaves weighing about 25g were obtained from the market in Chidambaram, Tamil Nadu. They were properly cleaned with distilled water, chopped into little pieces, and then cooked in 100 mL of the same water until the water turned golden yellow. The Whattman filter paper was used to filter the extract.

Preparation of ferric chloride solution

100 ml of 1mM Ferric chloride ($FeCl_3$) solution was prepared using distilled water.

Synthesis of iron oxide nanoparticle

In a beaker, a 1:1 mixture of the produced ferric chloride solution and *Solanum nigrum* leaf extract was added. The *Solanum nigrum* leaf extract is combined with ferric chloride solution to create a black, particle-filled solution. For three hours, a magnetic stirrer was used to continuously agitate the liquid. The generated solid product was extracted using centrifugation, cleaned three to four times with distilled water, and then allowed to air dry. After the dry product was calcined at 750°C for three hours, a powder with a reddish brown colour was produced. Karthikamurganantham *et al* (2015).

Sample Characterization

The synthesized Fe_2O_3 NPs were exposed to various characterization methods to identify their specific properties. For optical properties, UV-Vis spectrometer was used to record the absorption spectrum. SEM was used to determine the shape of Fe_2O_3 NPs. The functional groups were identified by recording the FTIR spectrum. And particle size of the Fe_2O_3 NPs were characterized by using XRD.

Antibacterial Activity

Bacteria strains

The Iron nanoparticle synthesis from the extracts (SN-NPs) were tested for antimicrobial activity against two Gram-positive bacterial strain and two Gram-negative-bacterial strain.

Agar well diffusion assay

Fe_2O_3 NPs tested for their antibiotic sensitivity pattern against different types of Gram-positive and negative pathogens, which are known to develop antibiotic resistance, such as *Staphylococcus aureus*, *Streptococcus pyogenes*, *Klebsiella pneumonia,* and *Escherichia coli*. Colonies from different types of bacteria were inoculated in L.B. broth and incubated for 24 hours at 37°C in an incubator. The bacterial cultures were then diluted to 1: 100 (equivalent to a cfu of 10^8). A 50–100 μL of the bacteria was taken, and a streak was made on nutrient agar (L. B. agar) medium using a sterile spreader in all directions. The antibiotic disks were applied with aseptic precautions. The disks were soaked in different samples of iron oxide nanoparticles and extracted and placed with centers at least 30 mm apart. The plate was incubated at 37°C in an incubator for 24 hrs. After incubation, the zone of inhibition around the disks was observed and measure.

Results and Discussion

Visual Observation

The addition of Ferric Chloride(B) solution to the *Solanum nigrum* leaf extract(A) produces a brown colour solution(C) suspended with particles. Upon calcination at 750°C for three hours, brownish-black colour powder(D) was obtained.

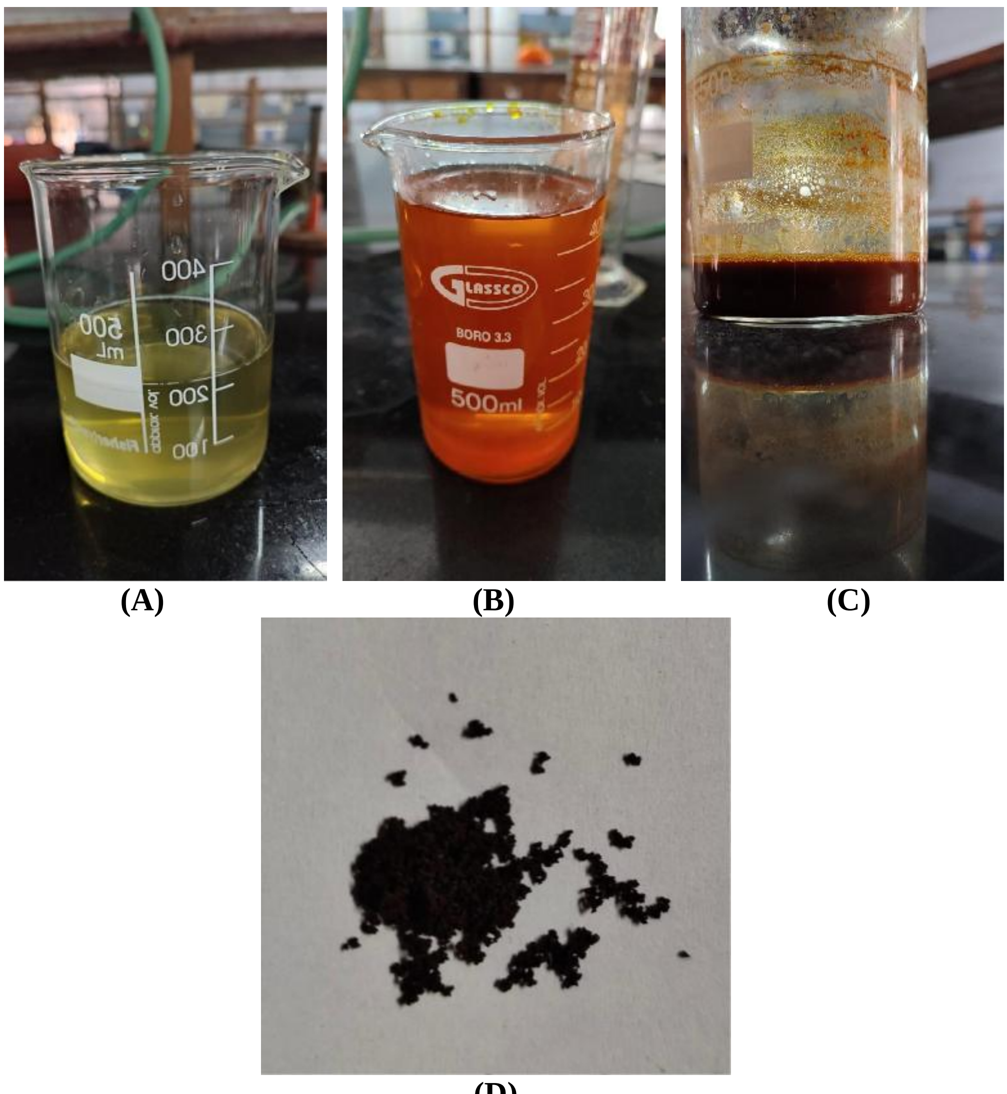


Fig 1: (A) *Solanum nigrum* leaf extract (yellow colour); (B) Ferric Chloride solution (reddish yellow colour); (C) Mixture of leaf extract and Ferric Chloride solution (brown colour);(D) Synthesized Iron Oxide compound (brownish black colour)

X Ray Diffraction (XRD)

Figure 2 shows the X-ray diffraction spectrum of the synthesized Fe_2O_3 NPs using the *Solanum nigrum* extract. The spectrum was recorded at a speed scan of 1 degree per minute in a 2theta/theta range of 10–70 degrees at an X-ray wavelength of 1.54 nm. The indexed diffraction peaks shown in the figure represent the crystalline phase of Fe_2O_3NPs. The match of the peaks with JCPDS card number 019-0629for Fe_2O_3NPs confirms the synthesis of hematite α-Fe_2O_3 nanocrystals. The average crystallite size D was calculated by the Debye Scherrer equation:

$$D = 0.9\lambda/\beta\ (\cos\theta)$$

Where,
λ is the wavelength of X-ray,
θ is the Braggs angle in radians,
β is the full width at half maximum of the peak.

The crystallite size D was found to be an average size of 24.1 nm. The results of the XRD analysis supported the tetragonal structure of α-Fe_2O_3 nanoparticles. By comparing the SEM and XRD size values, one concludes that there are multiple crystals in one particle in the case of single-crystal nanoparticles; the crystallite size and particle size are the same.

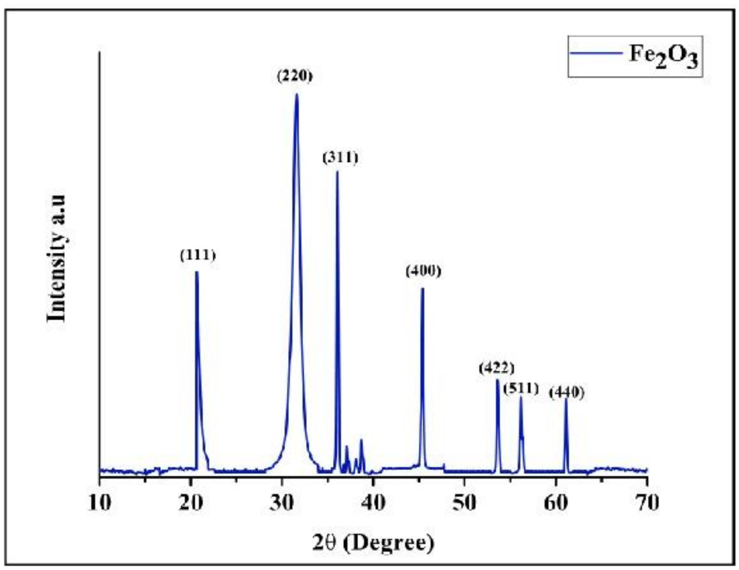
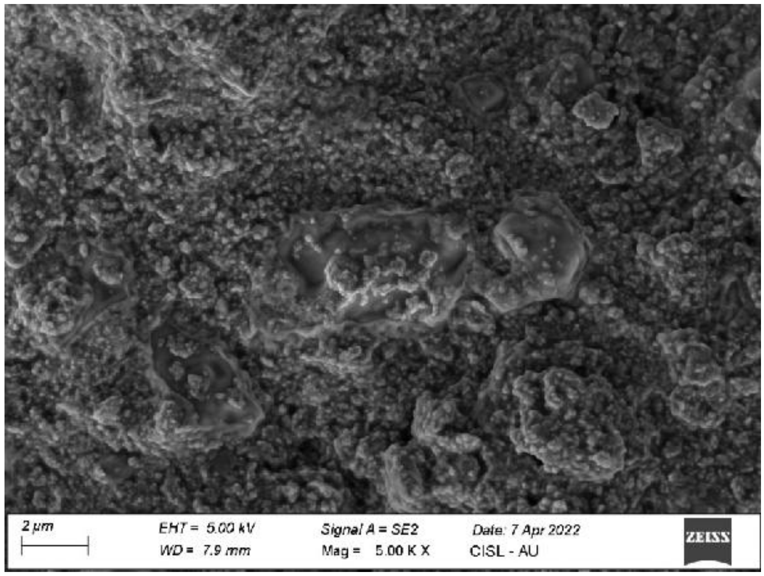


Fig 2: Xray Diffraction of Fe_2O_3 NPs synthesized using *Solanum nigrum* leaves extract.

Scanning electron microscopy (SEM) and energy dispersive X-ray spectroscopy (EDS)

SEM and EDS the morphologies of Fe_2O_3 NPs synthesized using *Solanum nigrum* leaves extract samples were examined using the SEM technique. The surface of the material was significantly magnified in the micrographs produced by scanning electron microscopy (SEM). Fig 3 (a) shows greater magnification micrographs of Fe_2O_3 NPs samples taken at different magnifications.

The synthesized Fe_2O_3 NPs had a uniform shape and a smooth surface, and this surface demonstrated the Hexagonal structural characterization of Fe_2O_3 NPs. Figure 3 (b) represents the EDS analysis showing firm Fe,O peaks in Fe_2O_3 NPs, as shown in Table 1.



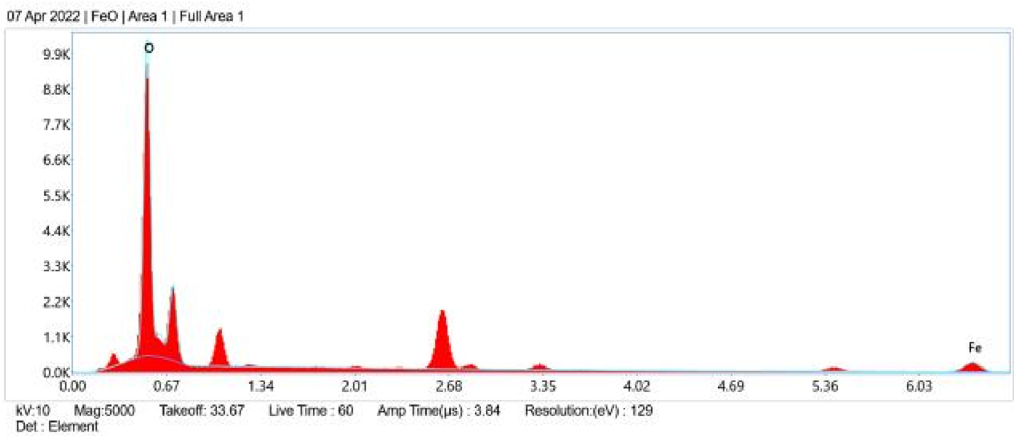


Fig 3 (a) SEM image and (b) EDS graph of Fe_2O_3 NPs synthesized using *Solanum nigrum* leaves extract.

Table 1: EDS measurements of Fe_2O_3 NPs synthesized using *Solanum nigrum* leaves extract.

Element	Weight %	Atomic %
O	45.27	74.28
Fe	54.73	25.72

UV-Vis spectroscopy

Ultraviolet-visible spectroscopy was used to monitor the degree of oxidation of Fe_2O_3 NPs. Fe_2O_3 NPs spectrum produced is illustrated in Fig 4. The absorption peak was discovered at 222 nm.

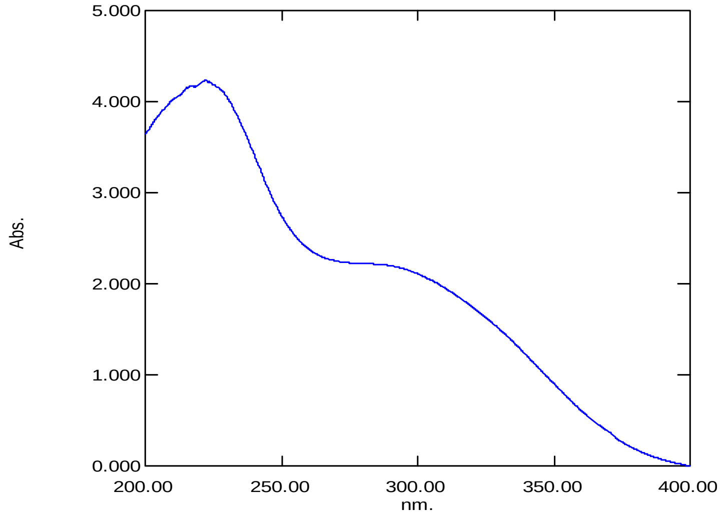


Fig 4: UV-Visible spectroscopy of Fe_2O_3 NPs synthesized using *Solanum nigrum* leaves extract.

Antibacterial activity

The antibacterial activity of Fe_2O_3 against bacterial pathogens was tested by a well diffusion method. The antibacterial potential of the biosynthesized Fe_2O_3 NPs using *Solanum nigrum* leaf extract is tested against *Escherichia coli*, *Staphylococcus aureus*, *Streptococcus pyogenes Klebsiella pneumonia* and shown in Fig.5, 6and table 2. The biosynthesized Fe_2O_3 NPs showed antibacterial effect on all the tested bacterial strains. The bactericidal effect of Fe_2O_3 NPs was found higher for Gram-negative bacteria than Gram-positive bacteria and was based on the difference in the structural composition of Gram-positive and Gram-negative bacteria. *Solanum nigrum* leaf extract-derived Fe_2O_3 NPs showed the maximum zone of inhibition was observed against *Klebsiella pneumoniae* (24 mm) followed by *Escherichia coli* (20 mm), *Staphylococcus aureus* (18 mm) and *Streptococcus pyogenes* (16 mm) at 100 µl concentration. Similar results on antibacterial effect for *Klebsiella pneumonia* and *E. coli* by Fe_2O_3 NPs were reported previously in the literature.

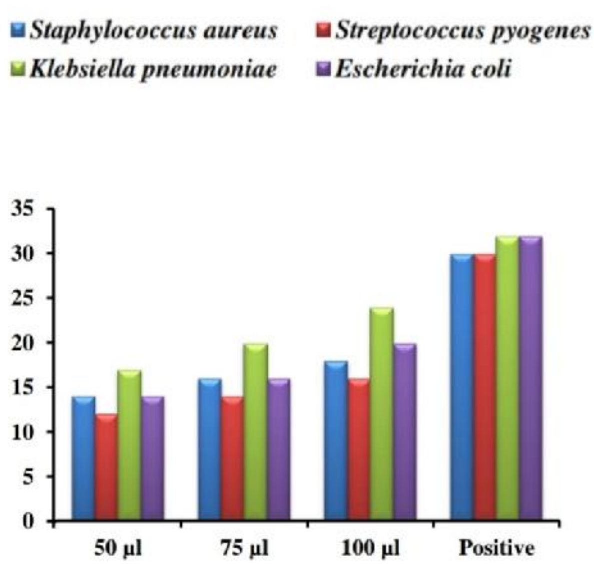


Fig 5: Graphical representation of zone of inhibition of Fe_2O_3 NPs synthesized using *Solanum nigrum* leaves extract.

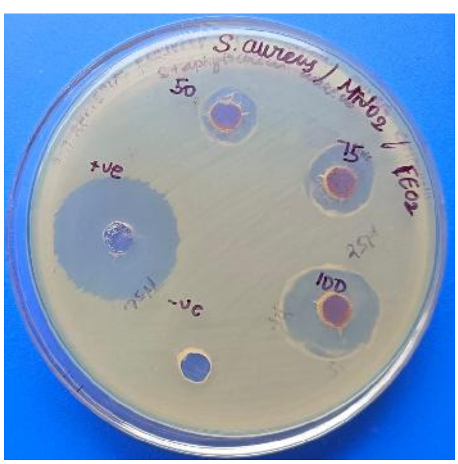
The mechanism of antibacterial activity of Fe_2O_3 NPs may be attributed to the penetration and disintegration of the membrane by smaller sized NPs which lead to cell lysis. The release of H_2O_2 from the surface of Fe_2O_3 NPs also reported as the possible mechanism for bactericidal activity. The generation of H_2O_2 is highly depended on the surface area of Fe_2O_3 and the generatedH_2O_2 penetrates the cell membrane and cause damage to kill the bacteria. Due to the presence of alkaloids, terpenoids, flavonoids, tannins, carbohydrates, sterols, saponins, proteins, and amino acids in *Solanum nigrum* leaf extract showed potential bioreducing activity and also bactericidal activity against the tested bacteria which could be useful for biomedical applications.

Table.2. Zone of inhibition of Fe_2O_3 NPs synthesized using *Solanum nigrum* leaves extract.

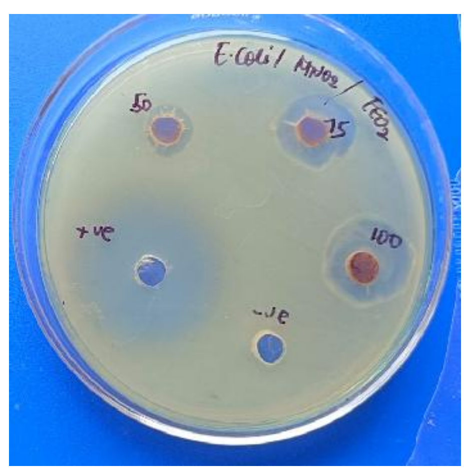
Bacterial pathogens	Zone of inhibition mm				
	50µl	**75 µl**	**100 µl**	**Positive**	**Negative**
Staphylococcus aureus	14	16	18	30	-
Streptococcus pyogenes	12	14	16	30	-
Klebsiella pneumoniae	17	20	24	32	-
Escherichia coli	14	16	20	32	-

Streptococcus pyogenes

Klebsiella pneumonia



Staphylococcus aureus



Escherichia coli

Fig 6: Antibacterial activity of Fe_2O_3 NPs synthesized using *Solanum nigrum* leaves extract.

Conclusion

Iron oxide nanoparticles were successfully synthesized by biogenic route using *Solanum nigrum* leaf extract. UV–Visible spectroscopy analysis confirmed the synthesis of Fe_2O_3 by the existence of band at 222 nm. SEM analysis revealed that the synthesized Fe_2O_3were in Hexagonal structure. Crystal size (24.1) of the Fe_2O_3 was confirmed by XRD analysis. EDS explained that the Fe,O peaks are present inFe_2O_3NPs synthesized using *Solanum nigrum* leaves extract. Fe_2O_3 showed better antibacterial activity against Gram-negative bacteria than Gram-positive bacteria. Fe_2O_3 NPs synthesized *Solanum nigrum* leaf extract could be utilized as antibacterial agent in biomedical, textile, and food industries.

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How to cite this article:
M. Rashmi Sulthana and M. Shenbagam Madhavan. (2022). Green synthesis, characterization, and antibacterial activity of iron oxide nanoparticles derived from *Solanum nigrum* leaf extract. Int. J. Adv. Res. Biol. Sci. 9(8): 131-139.
DOI: http://dx.doi.org/10.22192/ijarbs.2022.09.08.013