

Manuscript ID:
IJRSEAS-2026-030221



Quick Response Code:



Website: <https://eesrd.us>



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Volume: 3

Issue: 2(I)

Pp. 86-89

Month: April

Year: 2026

E-ISSN: 3066-0637

Submitted: 08 Mar. 2026

Revised: 18 Mar. 2026

Accepted: 09 Apr. 2026

Published: 30 Apr. 2026

Address for correspondence:

Purva A. Khadtar
D.G. Tatkare Mahavidyalay of Arts,
Science, Commerce, IT &
Management, Mangaon – Raigad

How to cite this article:

Purva A. Khadtar, Shubham
Yerunkar. (2026). A
Comprehensive study of Green
Synthesis of Iron Nanoparticles
using Justicia Adhatoda (adulsa)
and their characterization by FTIR
and X-ray diffraction method.
International Journal of Research
Studies on Environment, Earth, and
Allied Sciences, 3(2(I)), 86-89.

A Comprehensive study of Green Synthesis of Iron Nanoparticles using Justicia Adhatoda (adulsa) and their characterization by FTIR and X-ray diffraction method

Purva A. Khadtar¹, Shubham Yerunkar²

¹D.G. Tatkare Mahavidyalay of Arts, Science, Commerce, IT & Management,
Mangaon – Raigad

²Asst. Prof., D.G. Tatkare Mahavidyalay of Arts, Science, Commerce, IT &
Management, Mangaon – Raigad

Abstract:

The present study investigates a sustainable and eco-friendly approach for the synthesis of iron nanoparticles (FeNPs) using leaf extract of *Adhatoda vasica*, a well-known medicinal plant rich in bioactive phytochemicals. Nanotechnology has gained immense importance due to its diverse applications in biomedical, environmental, and industrial fields, with iron nanoparticles being particularly valuable because of their magnetic properties, catalytic efficiency, high surface area, and biocompatibility. However, conventional methods of nanoparticle synthesis involve toxic reagents, high energy consumption, and environmental hazards. This has led to the development of green synthesis methods that utilize plant-based materials as natural reducing and stabilizing agents.

In this study, aqueous leaf extract of *Adhatoda vasica* was prepared and employed for the reduction of ferric ions (Fe^{3+}) from ferric chloride solution into iron nanoparticles. The synthesis process was indicated by a distinct color change, confirming nanoparticle formation. The phytochemicals present in the plant extract, such as flavonoids, alkaloids, phenolic compounds, and terpenoids, played a dual role in reducing metal ions and stabilizing the formed nanoparticles, thus eliminating the need for additional chemical agents.

The synthesized iron nanoparticles were characterized using Fourier Transform Infrared Spectroscopy and X-ray Diffraction techniques. FTIR analysis revealed the presence of functional groups such as hydroxyl ($-\text{OH}$), carbonyl ($\text{C}=\text{O}$), and amine ($\text{N}-\text{H}$) groups, indicating the involvement of plant biomolecules in nanoparticle formation and capping. XRD analysis confirmed the crystalline nature of the iron nanoparticles and provided information about their phase structure. The average crystallite size was estimated using the Debye-Scherrer equation, demonstrating that the synthesized particles are in the nanoscale range.

The study was conducted with experiments performed in triplicates, and statistical analysis including mean and standard deviation was applied to ensure reliability of results. Despite some limitations such as possible nanoparticle aggregation and variation in particle size, the results confirm the successful synthesis of stable iron nanoparticles using a green route.

Overall, this research highlights the potential of *Adhatoda vasica* as an effective biological resource for nanoparticle synthesis and establishes a simple, cost-effective, and environmentally benign method. The synthesized iron nanoparticles show promising applications in areas such as drug delivery, environmental remediation, catalysis, and antimicrobial activity. This work contributes to the advancement of green nanotechnology and promotes the use of plant-based systems for sustainable material synthesis.

Keywords: Green Nanotechnology, *Adhatoda vasica*, Iron Nanoparticles (FeNPs), Plant-mediated synthesis, Fourier Transform Infrared Spectroscopy, X-ray Diffraction, Phytochemicals, Eco-friendly synthesis, Crystalline structure, Nanomaterials.

Introduction

Justicia Adhatoda commonly known in English as Malabar Nut, adulsa, *Adhatoda, vasa, vasaka* is native to Asia is a flowering plant that belongs to the Acanthaceae family. It is an important medicinal plant. It contains various bioactive compounds such as alkaloids, tannins, saponins, phenolics and flavonoids. The most important is vasicine, a quinazoline alkaloid. Green chemistry synthesis methods for NPs have positive aspects over chemical methods, for example being safe, eco-friendly, harmful compounds are not employed, and also cheap. Examples of green synthesis methods are as follows: firstly, the active biological component such as enzymes works as reducing and also as capping agents, and this is the reason why these small nanoparticles could be generated in the course of largescale production. Secondly, the plant extracts also reduce the metal ions in a short time, and it is widely used because of the presence of reducing and stabilizing agent in extracts. Nanotechnology has become one of the most significant scientific advancements due to its wide applications in medicine, agriculture, environmental remediation, and material science. Among different nanomaterials, iron nanoparticles (FeNPs) are particularly important because of their magnetic properties, catalytic efficiency, large surface area, and eco-friendly nature.

Traditional physical and chemical synthesis methods of nanoparticles often involve toxic chemicals, high temperatures, and expensive equipment, which can lead to environmental pollution and health hazards. Therefore, green synthesis has emerged as a sustainable alternative. Green synthesis uses biological systems such as plants, bacteria, fungi, and algae to produce nanoparticles in an eco-friendly and cost-effective manner.

The plant *Adhatoda vasica* is widely used in traditional medicine and contains bioactive compounds like alkaloids (vasicine), flavonoids, phenolics, and terpenoids. These phytochemicals play a crucial role as reducing, stabilizing, and capping agents in nanoparticle synthesis, making it an excellent candidate for green synthesis.

Characterization techniques such as Fourier Transform Infrared Spectroscopy and X-ray Diffraction are essential to confirm nanoparticle formation, identify functional groups, and determine crystalline structure.

Literature Review

The synthesis of metal nanoparticles through green chemistry approaches has garnered significant attention due to their eco-friendly, cost-effective, and scalable nature. Among various nanomaterials, iron nanoparticles (FeNPs) have emerged as a promising class of materials with applications in biomedicine, catalysis, wastewater treatment, and environmental remediation. Traditional physical and chemical synthesis methods, though effective, often require high energy input and involve hazardous reducing agents like sodium borohydride (NaBH_4), hydrazine, and organic solvents, which pose environmental and health risks. In recent years, plant-mediated synthesis of nanoparticles has become an attractive alternative due to the presence of natural bioactive compounds such as alkaloids, flavonoids, phenolics, tannins, terpenoids, and saponins. These phytochemicals function as both reducing and capping agents, stabilizing the nanoparticles while also influencing their morphology and size. The synthesis of FeNPs using plant extracts thus aligns with the principles of green chemistry by eliminating toxic reagents, reducing energy usage, and ensuring biocompatibility. The plant *Justicia adhatoda*, also known as Malabar Nut (*Adulasa*), is a widely known medicinal herb rich in secondary metabolites such as vasicine (a quinazoline alkaloid), flavonoids, and tannins. These compounds have demonstrated antioxidant and antimicrobial activities, making the plant suitable for nanoparticle synthesis. In the current project, the aqueous extract of Malabar Nut leaves has been used for the green synthesis of FeNPs, where color change from orange to dark brown confirmed the reduction of Fe^{3+} ions to Fe^0 nanoparticles. Several studies support the efficacy of plant extracts in synthesizing iron and other metal nanoparticles. For example, Shah et al. demonstrated the successful synthesis of iron nanoparticles using leaf extracts of various plants and confirmed their photocatalytic and antibacterial properties. Roy et al. showed that silver and iron nanoparticles synthesized using medicinal plants were effective in dye degradation and microbial inhibition. Similarly, Naseem and Farrukh [10] synthesized FeNPs using *Lawsonia inermis* and *Gardenia jasminoides* leaves, reporting high antibacterial activity against *E. coli*. *Justicia Adhatoda* is well known for its medicinal properties, including antimicrobial, anti-inflammatory, and antioxidant activities. Previous research has shown that extracts of *A. vasica* contain vasicine, vasicinone, and other bioactive compounds capable of reducing metal ions. However, limited studies are available on the use of *Justicia Adhatoda* for iron nanoparticle synthesis, indicating a research gap that this study aims to address.

Research Methodology

This research focuses on the green synthesis of iron nanoparticles (FeNPs) using *Justicia Adhatoda* (*Adulsa* leaves) extract, their characterization, and evaluation of catalytic applications.

1 Laboratory Requirement :-

100 Conical flask, Measuring Cylinder 100 ml, Volumetric flask 100 ml, Burette 25 ml, Beaker 100 ml, Glass rod, Suction pump, Sonicator, Hot plate with magnetic stirrer etc., 0.1 N FeCl_3 solution, Leaf Extract, 0.5 % ammonia solution

2 Collection of Plant Materials :-

Fresh leaves of Malabar Nut were collected from village Kharavali, Mangaon, Raigad. *Justicia Adhatoda* is commonly known in English as Malabar Nut, *adulsa*, *Ashatada*, *vasa*, *vasaka* is native to Asia is a flowering plant that belongs to the *Acanthaceae* family. It is an important medicinal plant.

3 Green Synthesis Method :-

Green synthesis method is proved as beneficial over other methods which are implemented for the synthesis of nanoparticles. Green synthesis method is eco-friendly approach and compatible for pharmaceutical & other biomedical applications. For all experimental study pure and analytical grade chemicals were used.

3.4 Preparation of Powder:-

Fresh leaves of Malabar Nut Plants were washed two to three times in distilled water by which the dust particles were removed and plant material was then placed to dry under sunlight for 3-4 days. All of the dried leaves of plants were grind using pestle & mortar. After the process of grinding the leaves powder went through sieving to get very fine particles of uniform size. Nanoparticles were synthesized by using sieved powder.

5 Preparation of Leaf Extract :-

Take 20 gm of Powder in a 250 ml conical flask. Add 100 ml distilled water to it. Keep this conical flask in sonicator for 25 min. at temperature 50-60°C. Then filter the solution by using Whatmann Filter paper No. 1 (Filter 3-4 times) and collect the filtrate as a plant extract.

6 Synthesis of Fe Nanoparticles :-

A conical flask containing a 0.1 M FeCl₃ solution (1.622 gm FeCl₃ (anhydrous) dilute in 100 ml distilled water) was subjected to sonication without heating for 25 minutes. Add 10 ml 0.5 % ammonia solution to it. Ultrasound irradiation offers an alternative energy source which is usually accomplished by heating. Keep these conical flask in sonicator for 10 minutes. After that add 25 ml plant extract, 2-3 ml per minute to it by using burette. Keep the flask in sonicator for 25 minutes more. The development of FeNPs is indicated by a specific time-related color change from orange to dark brown. Allow the mixture at room temperature for 24 hours. Adulsa plant extract contains phytochemicals which changed iron ions into iron nanoparticles. Centrifuge the remedy to settle down the solution. Collect & dry the compound at room temperature. Grind the comp. using crystal pastel & mortal.

7 Characterization of FeNPs :-

In this study, we aimed synthesize Iron nanoparticles using the green synthesis method and characterize their structural and magnetic properties. • The characterization of nanoparticles is a branch of nanometrology that deals with the characterization, or measurement, of the physical and chemical properties of nanoparticles. Nanoparticles are characterized for various purposes, including nanotoxicology studies. • Once the NPs are synthesized, Nanoparticles differ in their physical properties such as size, shape, and dispersion, which must be measured to fully describe them by using various technique. The common techniques of characterizing NPs are X-Ray diffraction (XRD), Fourier Transmission Infrared (FTIR) spectroscopy.

FTIR spectroscopy is performed to identify functional groups present on the surface of iron nanoparticles and to confirm the role of phytochemicals in reduction and stabilization.

XRD analysis is conducted to determine the crystalline nature and phase identification of iron nanoparticles. The average crystallite size is calculated using the Debye-Scherrer equation.

Expected Outcomes

Successful green synthesis of iron nanoparticles using Adhatoda vasica extract

Observable color change confirming nanoparticle formation

FTIR spectra indicating functional groups involved in reduction and capping

XRD patterns confirming crystalline structure and nanoscale size

Development of a simple, cost-effective, and eco-friendly synthesis method

Additionally, the synthesized nanoparticles are expected to show potential applications in:

Biomedical fields (drug delivery, antimicrobial activity)

Environmental remediation (wastewater treatment)

Catalysis and industrial applications

Understanding of the role of medicinal plant biomolecules in nanoparticle formation. Data supporting use of green FeNPs in catalytic and environmental applications. Development of a sustainable approach for producing catalytically active nanoparticles. Potential application in pollutant removal, dye degradation, and green chemical processes.

Conclusion

Green synthesis of iron nanoparticles using Adhatoda vasica represents an environmentally friendly and sustainable approach in nanotechnology. The presence of phytochemicals enables efficient reduction and stabilization of nanoparticles without the need for toxic chemicals.

Characterization using Fourier Transform Infrared Spectroscopy and X-ray Diffraction confirms the formation, functionalization, and crystalline nature of the nanoparticles.

Although challenges such as particle aggregation and size variation exist, the overall method is simple, cost-effective, and scalable. This study contributes to the advancement of green nanotechnology and promotes the use of medicinal plants in nanoparticle synthesis.

Acknowledgement

I would like to express my sincere gratitude to all those who contributed to the successful completion of this study entitled "A Comprehensive Study of Green Synthesis of Iron Nanoparticles Using Justicia adhatoda (Adulsa) and Their Characterization by FTIR and X-Ray Diffraction Method."

First and foremost, I express my heartfelt thanks to my guide/supervisor for their invaluable guidance, continuous encouragement, and constructive suggestions throughout the course of this work. Their expertise and support greatly helped in the successful completion of this study.

I also extend my sincere appreciation to the faculty members and laboratory staff of the department for providing the necessary facilities, technical assistance, and academic support during the experimental work.

I am grateful to my institution for providing access to laboratory equipment, chemicals, analytical instruments, and research materials required for carrying out this study successfully.

Special thanks are due to my friends and colleagues for their cooperation, motivation, and helpful discussions during the preparation of this project.

Finally, I would like to express my deepest gratitude to my family for their constant encouragement, patience, and unwavering support throughout my academic journey.

Financial support and sponsorship

Nil.

Conflicts of interest

The authors declare that there are no conflicts of interest regarding the publication of this paper.

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