Jurnal Inovasi Teknologi dan Edukasi Teknik, 5(4), 2025

GREEN SYNTHESIS AND CHARACTERIZATION OF IRON NANOPARTICLES USING PHYLLANTHUS AMARUS LEAF EXTRACT AND THEIR APPLICATION IN CRYSTAL VIOLET DYE DEGRADATION

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doi: 10.17977/um068.v5.i4.2025.1

Keywords
Green synthesis
Phyllanthus amarus
Iron oxide nanoparticles
Crystal violet
Photocatalysis
Environmental remediation

Abstract

The green synthesis of nanoparticles using plant extracts offers a sustainable and eco-friendly alternative to conventional chemical methods that often involve toxic reagents and high energy consumption. In this study, iron oxide nanoparticles (FeNPs) were synthesized using aqueous leaf extract of *Phyllanthus amarus*, a medicinal plant known for its rich phytochemical content. The biosynthesized FeNPs (PaFeNPs) were characterized using Fourier Transform Infrared Spectroscopy (FTIR), Thermogravimetric Analysis (TGA), UV-Vis and Energy Dispersive X-ray (EDX) spectroscopy to confirm their formation, functionalization, and elemental composition. Phytochemical screening of the leaf extract revealed the presence of key biomolecules such as flavonoids, terpenoids, saponins, proteins, carbohydrates, and reducing sugars, which played critical roles in the reduction and stabilization of the nanoparticles. The photocatalytic efficiency of the synthesized PaFeNPs was evaluated by degrading crystal violet (CV) dye under both ultraviolet (UV) light and dark conditions. Under UV exposure, significant dye degradation was observed, attributed to the generation of hydroxyl and superoxide radicals, while in the dark, dye removal was primarily due to adsorption. The color change from purple to nearly colorless confirmed effective CV removal in both conditions. These findings demonstrate that *P. amarus*-mediated FeNPs possess excellent photocatalytic and adsorptive properties, highlighting their potential application in environmental remediation. This study not only confirms the feasibility of using *Phyllanthus amarus* for the green synthesis of FeNPs but also suggests their potential for wastewater treatment applications, thereby contributing to sustainable nanotechnology and environmental protection.

1. Introduction

In recent years, nanotechnology has emerged as a state-of-the-art and cutting-edge field with various applications across various sectors. It encompasses the study and manipulation of materials at the nanoscale and includes nanomaterials, nanotools, and nanodevices (Ebrahiminezhad et.al, 2016). Among these, nanoparticles have attracted significant research attention due to their ease of synthesis and manipulation (Gupta and Xieh, 2018). Traditionally, nanoparticles have been synthesized using physical and chemical methods. However, these conventional techniques often involve toxic reagents, high energy consumption, and adverse environmental effects. As a result, research has increasingly focused on developing green and eco-friendly synthesis protocols (Pattanayak et.al, 2013).

Nanotechnology has become a cornerstone of industrial advancement and innovation, with notable impacts across diverse sectors. It plays a key role in developing diagnostic biosensors, drug delivery systems, and imaging probes in the medical field. Nanomaterials enhance production processes, extend shelf life, and improve bioavailability in the food and cosmetics industries. Applications of nanoparticles also extend to drug delivery, hyperthermia, gene therapy, food preservation, antimicrobial activity, bioseparation, ferrofluids, environmental remediation, lithium-ion batteries, and pigments (Laurent et. al., 2011).

Currently, three areas of nanotechnology are considered top priorities: nano-medicine, nano-energy, nano-environment, and nano-information and communication technology (Busolo et al., 2012). Nano-medicine encompasses nano-imaging, the development of nanodevices, novel therapeutics, theranostics, drug delivery systems, and considerations around clinical applications and toxicology. Nano-energy includes technologies for solar energy, hydrogen production and storage, fuel cells, and environmental protection via catalytic applications. The nano-environment focuses on developing eco-friendly materials such as biodegradable plastics, low-toxicity rechargeable batteries, self-cleaning, and nano-coated glass surfaces.

One primary concern is the contamination of freshwater resources, primarily due to the improper disposal of industrial, agricultural, and municipal wastes (Adewoye and Lateef, 2004). This has significantly reduced the availability of safe water for domestic and agricultural use (Adewoye et al., 2013). Nanotechnology offers promising solutions through nanoparticle-based filters for water purification and nano-sensors for pollution detection. Nanotechnology enhances processing speeds, memory capacity, and power efficiency in the information and communication technology sector.

Magnetic nanoparticles have garnered attention due to their unique properties and broad applications, including the immobilization of proteins and enzymes, bioseparation, immunoassays, drug delivery, and biosensing (Chen et al., 2002). Ferromagnetic nanoparticles are especially valuable due to their small sizes and the ability to form single magnetic domains. Among these, iron oxide nanoparticles (FeNPs) have shown great potential for environmental remediation because of their high surface area-to-volume ratio and enhanced reactivity (Reguyal et al., 2017).

FeNPs are now used in various environmental clean-up technologies, including detoxifying and removing carbon monoxide, organic dyes, arsenic, chromium, and mercury (Ali et al., 2016). They are also applied in wastewater treatment using adsorption, membrane filtration, photocatalysis, and dematerialization methods (Xu et al., 2012). Table 1 summarizes the diverse applications of FeNPs across multiple disciplines.

Table 1: Applications of Iron Oxide Nanoparticles Across Various Fields

Discipline	Application
Environmental Remediation	Wastewater treatment, pollutant sensing, energy distribution, pollution prevention
Biomedical	Magnetic hyperthermia, controlled drug release, MRI contrast agents, biosensors, tissue repair
Defense and Aerospace	Fuel additives, energy devices, sensors, nano coatings, electronics
Construction	Coloring materials, nanoscale sensors, smart coatings, nanocomposites
Electronics	Nanoscale memory, spintronics, printed electronics, nanowires, NEMS
Healthcare	Nanoscale biosensors, antimicrobial surfaces, nano photothermolysis, implants, vaccination platforms
Automotive/Textiles	Catalysts, lubricants, sensors, nanofibers, coatings, fuel cells, smart materials
Agriculture and Food	Nano-pesticides, nano-fungicides, nano-fertilizers, nanofood, gene transfer, encapsulation, packaging

Due to their antimicrobial, antioxidant, and environmental remediation potential, FeNPs have been investigated to combat various issues from microbial infections to pollution. The present study proposes a green synthesis route for producing iron oxide nanoparticles using plant extracts—a cost-effective and environmentally friendly method.

Phyllanthus amarus, a small annual herb from the family Euphorbiaceae, is an ideal candidate for green synthesis due to its rich phytochemical profile. Known for its wide range of medicinal uses, P. amarus is referred to locally as *Carry me go seed* (English), *Eyin Olobe* (Yoruba), *Geeron-Tsuntsaayee* (Hausa), and *Enyikwonwa* (Igbo). The plant thrives in moist, shaded environments and is traditionally used to treat malaria, gastrointestinal disturbances, skin diseases, and sexually transmitted infections. Several studies have documented its antibacterial effects on Escherichia coli, Klebsiella pneumoniae, and Proteus mirabilis (Contreras and Gamara, 2012), as well as its effectiveness against hepatitis B and HIV-1 reverse transcriptase (Oguta, 2019; Xin et al., 2017).

Phyllanthus amarus contains a range of bioactive compounds, including lignans, terpenes, flavonoids, alkaloids, tannins, and saponins, contributing to its antiviral, antibacterial, anti-inflammatory, antidiabetic, anticancer, hypolipidemic, hepatoprotective, neuroprotective, and antioxidant properties (Patel et al., 2011; Bankole et al., 2011).

In light of this, the current research focused on extracting the phytochemicals from P. amarus leaves for use in the green synthesis of iron oxide nanoparticles (FeNPs). The synthesized nanoparticles were characterized using various analytical techniques, including Fourier Transform Infrared (FTIR) spectroscopy, Thermogravimetric Analysis (TGA), UV-Vis spectroscopy, and Energy Dispersive X-ray (EDX) analysis. Furthermore, the photocatalytic degradation of crystal violet dye was evaluated using both bare FeNPs and P. amarus-mediated FeNPs, demonstrating their potential application in environmental remediation.

2. Methods

2.1. Materials

Fresh *Phyllanthus amarus* leaves were collected from the surroundings of the Science Laboratory Technology (SLT) building within the Federal University of Technology, Owerri, Nigeria. The plant was taxonomically identified and authenticated at the Herbarium of the Department of Pharmacognosy, Nnamdi Azikiwe University, Agulu Campus.

The following reagents and equipment were used: Iron (III) Chloride Hexahydrate ($FeCl_3 \cdot 6H_2O$, 98%), ethanol, Whatman No. 1 filter paper, watch glass, measuring cylinders, standard flasks, refrigerator, mechanical grinder, sterile sample bottles, beakers, and distilled-deionized water.

2.2. Preparation of Leaf Extract for Phytochemical Screening

The *Phyllanthus amarus* leaves were detached from the stems, thoroughly washed with deionized water to remove dust and contaminants, and air-dried at room temperature for two weeks. After drying, the leaves were pulverized using a mechanical grinder. Approximately 100 g of the powdered leaves were boiled in 100 mL of deionized water and then filtered upon cooling using Whatman No. 1 filter paper (Xin-Hua et al., 2017). The filtrates were measured and stored in sterile, airtight containers at 4°C for subsequent use. Phytochemical screening was conducted using standard protocols described by Pattanayak and Nayak (2013).

2.3. Green Synthesis of Iron Oxide Nanoparticles Using *Phyllanthus amarus*

Iron oxide nanoparticles (FeNPs) were synthesized using a modified procedure from Kubde and Meenal (2013). A 0.01 M aqueous solution of $FeCl_3 \cdot 6H_2O$ was mixed with *P. amarus* leaf extract in a 1:1 volume ratio. The reduction reaction was initiated immediately, indicated by a change in the solution's color to black, suggesting the formation of FeNPs. The mixture was centrifuged at 4000 rpm for 30 minutes, and UV–Vis analysis was immediately performed to confirm nanoparticle synthesis. The nanoparticles were freeze-dried and stored under refrigeration for characterization.

2.4. Characterization of Iron Oxide Nanoparticles

2.4.1. Fourier Transform Infrared Spectroscopy (FTIR)

FTIR spectroscopy was used to identify functional groups responsible for the reduction and capping of FeNPs. Dried PaFeNP powder was analyzed using a Perkin-Elmer 100 Series FTIR spectrometer at a scan rate of 4 ms^1 over a wavenumber range of 4000–400 cm^1, with a resolution of 4 cm^1. The analysis was conducted in diffuse reflectance mode using KBr pellets.

2.4.2. Thermogravimetric Analysis (TGA)

TGA was used to study the thermal stability and decomposition profile of the synthesized PaFeNPs. Mass changes in the sample as a function of temperature were analyzed to assess the presence of volatile compounds and organic capping agents.

2.4.3. Energy Dispersive X-ray Spectroscopy (EDX)

EDX analysis was performed to determine the elemental composition of the synthesized nanoparticles. The presence and proportion of iron, oxygen, and other elements were quantified to confirm the formation of iron oxide.

2.5. Photodegradation of Crystal Violet Dye Using PaFeNPs

The photodegradation efficiency of the synthesized PaFeNPs was evaluated using crystal violet (CV) dye under both UV and dark conditions. A mixture of 50 mg of PaFeNPs and 100 mL of 10 mg/L CV solution was stirred at room temperature and exposed to UV light (λ = 395 nm) for 30, 50, 70, and 90 minutes. After each interval, the solution was analyzed using a UV-Vis spectrophotometer at 582 nm to determine the final dye concentration. The dye removal percentage was calculated using:

Removal Percentage (%)=Co−CfCo×100\text{Removal Percentage (\%)} = \frac{C_o - C_f}{C_o} \times 100Removal Percentage (%)=CoCo−Cf×100 where CoC_oCo = initial dye concentration and CfC_fCf = final concentration.

The experiment was repeated without light to assess the nanoparticles' adsorption capacity in dark conditions.

3. Results and Discussion

3.1. Phytochemical Analysis

Phytochemical screening of *P. amarus* leaf extract (Table 1) revealed the presence of flavonoids, terpenoids, saponins, proteins, carbohydrates, and reducing sugars. These bioactive compounds play a critical role in the bioreduction of iron ions and stabilization (capping) of FeNPs. The presence of bio-reducing agents such as flavonoids, ascorbic acid, and enzymes like reductase and dehydrogenase significantly contributes to the formation and stability of the nanoparticles (Devatha et al., 2016).

Table 1: Qualitative Phytochemical Screening of *P. amarus* Extract

Phytochemical	Presence
Alkaloids	-
Steroids	+
Flavonoids	+
Tannins	-
Terpenoids	+
Anthraquinones	-
Phlobatannins	-
Cardiac glycosides	-
Reducing sugars	+
Carbohydrates	+
Saponins	+
Coumarins	-
Proteins	+
Phenols	-
Amino acids	-
Phytosterols	-

3.2. UV-Vis Properties of Iron Oxide Nanoparticles (FeNPs)

A rapid color change in the reaction mixture first indicated the successful synthesis of iron oxide nanoparticles (PaFeNPs). Immediately upon mixing the *P. amarus* extract with $FeCl_3 \cdot 6H_2O$ solution, the color transitioned from light brown to black within seconds (Figure 4.1). This observable transformation indicates FeNPs formation and is attributed to the excitation of surface plasmon vibrations, a typical phenomenon associated with metallic nanoparticles (Devatha et al., 2016). The dark coloration is further explained by the complexation of iron salts and subsequent capping by phenolic compounds present in the plant extract (Badmapriya & Asharani, 2016; Vadivel et al., 2012).

UV-Visible spectrophotometric analysis was carried out in the 200–900 nm range to confirm nanoparticle synthesis further. A prominent absorption peak was observed at 286 nm (Figure 1), corresponding to the iron oxide nanoparticles' surface plasmon resonance (SPR). This absorption confirms the excitation of SPR due to nanoparticle formation and is consistent with previously reported values for biologically synthesized FeNPs (Pattanayak & Nayak, 2013; Enshirah et al., 2018).

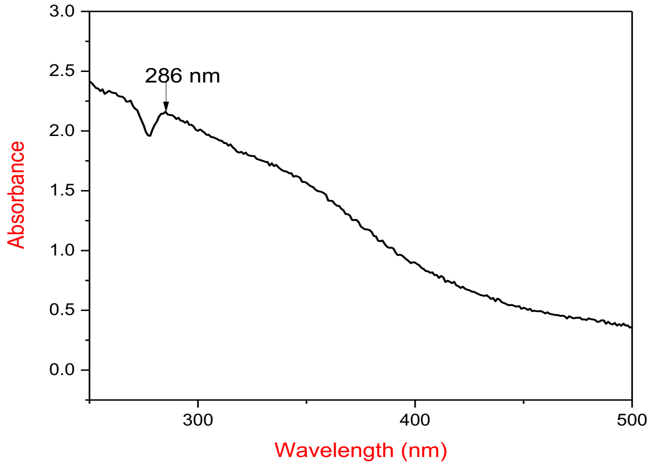

Figure 1: UV-Vis spectra of PaFeNPs

3.3. FTIR Analysis

3.3.1. FTIR spectroscopy of PaFeNPs

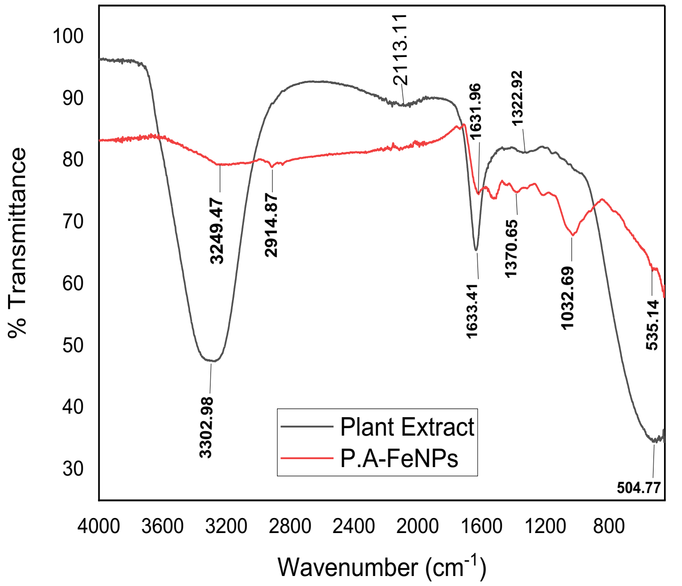

Figure 2: FTIR spectra of *P. amarus* leaf extract and FeNPs synthesized using *P. amarus leaves* extract

Figure 2 shows the FTIR spectra of the *P. amarus* leaf extract and PaFeNPs. Shifts in peak positions indicated interactions between bioactive compounds and iron ions. Notable peaks included O–H and N–H stretching at ~3300 cm^{-1}, C=O stretching at ~1631 cm^{-1}, and Fe–O vibrations at ~535 cm^{-1}, confirming nanoparticle formation and stabilization by plant phytochemicals (Ebrahiminezhad et al., 2014; Radini et al., 2018).

3.3.2. Thermogravimetric Analysis (TGA)

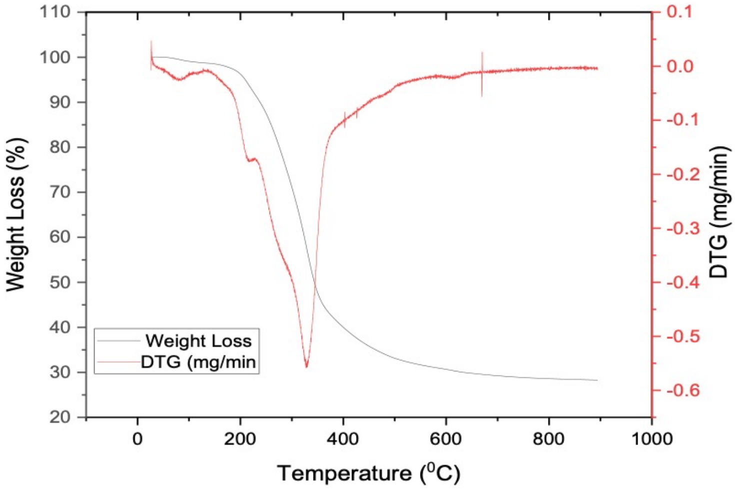

Figure 3: TGA and differential TGA (DTG) curves PaFeNPs

As shown in Figure 3, PaFeNPs exhibited a significant weight loss below 200 °C, attributed to water loss and decomposition of organic capping agents. Total weight loss was about 30%, confirming the presence of biomolecules on the nanoparticle surface. Decomposition peaks observed in DTG curves aligned with previously reported values (Juríková, 2012).

3.4. EDX spectroscopy of PaFeNPs

3.4.1. EDX Analysis

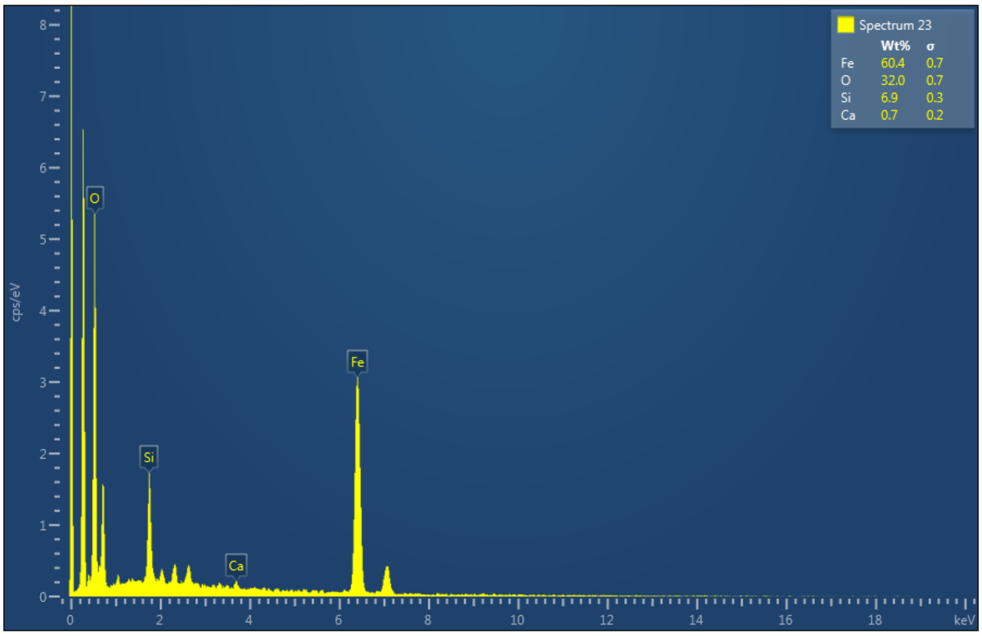

Figure 4: EDX analysis of PaFeNPs

EDX results (Figure 4) confirmed the presence of iron (Fe – 60.4%) and oxygen (O – 32.0%), with minor impurities such as silicon (6.9%) and calcium (0.7%) arising from the sample grid. This confirmed the elemental composition of iron oxide nanoparticles (Devatha et al., 2016).

3.5. Photodegradation of Crystal Violet

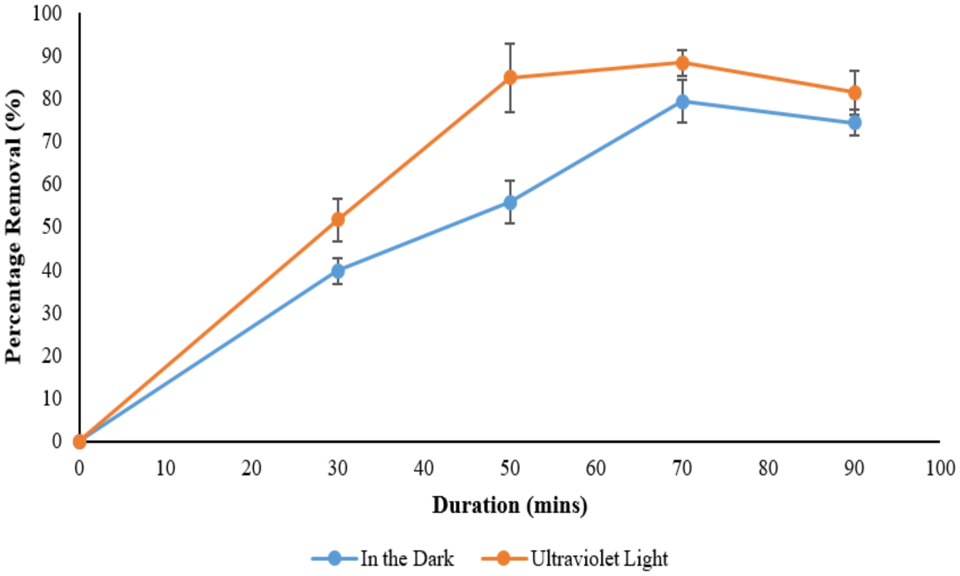

Figure 5. The effect of time on the percentage removal of CV dye in the absence and presence of ultraviolet light.

Figure 5 illustrates the removal efficiency of crystal violet by PaFeNPs under UV and dark conditions. Under UV light, hydroxyl and superoxide radicals were generated, enhancing photodegradation. In the absence of light, the removal was mainly due to adsorption. Rapid dye removal occurred within the first 70 minutes in both cases. The decline in efficiency after 70 minutes may be due to desorption or nanoparticle saturation. The color change from purple to nearly colorless indicated significant degradation.

Bhuiyan et al. (2018) explain that UV-irradiated FeNPs generate electron-hole pairs that drive oxidation reactions, converting CV into harmless byproducts. The synergistic effect of adsorption and photocatalysis was evident in UV conditions, while adsorption was the dominant mechanism in the dark.

4. Conclusion

This study successfully demonstrated the green synthesis of iron oxide nanoparticles (PaFeNPs) using *Phyllanthus amarus* leaf extract. The plant extract was a reducing and stabilizing agent, enabling eco-friendly, low-cost, and scalable nanoparticle production. Characterization via FTIR, TGA, and EDX confirmed the formation and functionalization of PaFeNPs. These nanoparticles exhibited excellent performance in degrading crystal violet dye under UV light and were efficient adsorbents in the dark. Thus, *P. amarus*-mediated FeNPs present promising potential for environmental remediation applications.

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