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Utilization of Eco-Friendly Iron Oxide Nanoparticles for Wastewater Treatment

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Abstract

This study investigates the potential of green-synthesized Iron II Oxide (Fe_2O_3) nanoparticles for remediating wastewater from River Benue, Nigeria, focusing on removing lead (Ld) pollutants. The nanoparticles were synthesized using Annona squamosa leaf extract, offering an eco-friendly alternative to conventional remediation methods. The wastewater sample, collected from River Benue in Jimeta-Yola, was analyzed using FT-IR, SEM, and atomic absorption spectroscopy. FT-IR analysis identified functional groups like alcohol O-H and conjugated alkene C=C, confirming the role of plant metabolites in nanoparticle synthesis and stabilization. SEM imaging revealed cubical Fe_2O_3 nanoparticle sizes ranging from 25 to 34 nm, stabilized by the plant extract. The remediation process tested varying nanoparticle concentrations (100, 500, 1000, and 1500 ppm) and contact times (60, 120, and 180 minutes) at pH 8.90 and 21.33 K. The highest adsorption efficiency was observed at 1500 ppm over 180 minutes, reducing lead levels from 0.69 mg/L to 0.02 mg/L due to increased chelating sites. Lower concentrations (1000, 500, and 100 ppm) also demonstrated significant adsorption, with lead levels dropping to 0.21, 0.32, and 0.50 mg/L, respectively. The study highlights the advantages of Fe_2O_3 nanoparticles, including simplicity, rapid production, environmental safety, and a high surface area for effective pollutant adsorption. This green synthesis approach is more sustainable and operationally simpler than tradisional methods, avoiding the environmental risks associated with conventional techniques. The findings suggest that Fe_2O_3 nanoparticle is promise for efficient and sustainable wastewater purification, offering a viable alternative to existing water treatment technologies. This research underscores the potential of eco-friendly nanomaterials in addressing heavy metal pollution, particularly in developing regions.

Keywords: Remediation; Wastewater; Annona squamosa; Synthesis; Nanotechnology.

INTRODUCTION

Water is essential for life, development, and health, and the importance of clean drinking water in fostering well-being cannot be overstated (Rheeder et al., 2023). However, water pollution has become a global crisis, driven by industrialization, urbanization, and population growth. This pollution harms the environment and contributes to air pollution, posing severe risks to human health (Talema, 2023). Access to clean water and sanitation is a fundamental human right, yet increasing pollution, water scarcity, and inadequate treatment technologies have made achieving this goal a significant challenge (Angelakis et al., 2023; Hait et al., 2024).

Historically, conventional water purification methods, particularly chemical-based approaches, have been widely used despite their complexity and hazardous nature (Parvulescu et al., 2021). In contrast, nanotechnology has emerged as a transformative field, offering innovative solutions due to the unique properties of nanomaterials, such as small particle size, high surface area, and exceptional thermal, optical, and mechanical characteristics (Eddy et al., 2023). Nanotechnology can revolutionize various sectors, including healthcare, manufacturing, and environmental protection, by producing smaller, more efficient, cost-effective materials with reduced energy and resource consumption (Malik et al., 2023).

Metal-based and metal oxide nanoparticles, in particular, have gained attention as sustainable solutions for water remediation. Their small size, large surface area, and quantum effects make them highly effective. Green synthesis methods, which use biological materials like plant extracts, are preferred over chemical and physical methods due to their environmental friendliness, cost-effectiveness, and scalability (Mamuru et al., 2022; Gupta et al., 2023). For instance, iron oxide nanoparticles synthesized from *Prosopis Africana* leaf extract have shown promising results, with characterization techniques like UV-vis spectroscopy, SEM, and FT-IR confirming their structure and functionality (Elebo et al., 2024). Similarly, zinc oxide nanoparticles synthesized using *Mangifera indica* extract have demonstrated potential in degrading pollutants like Congo red dye (Mamuru et al., 2022).

Despite these advancements, there is limited research on the application of eco-friendly iron oxide nanoparticles (Fe_2O_3-NPs) for wastewater treatment in regions like Adamawa State, Nigeria. This study explores the potential of green-synthesized Fe_2O_3-NPs for sustainable wastewater management, addressing a critical gap in current research (Abd Elmohsen et al., 2024).

MATERIALS AND METHODS

Collection of Plant Materials

According to Nahari *et al.,* (2022), fresh leaves of *A. squamosa* were hand-picked from the Chemistry Department block of the Faculty of Physical Sciences, Modibbo Adama University Yola, which was authenticated in the Plant Science Department and a voucher specimen was saved in the herbarium (No. MAU/PLS/0067).

Preparation of Plant Leaf Extract

Fresh *A. squamosa* leaves were used to extract the active constituents. The leaves were thoroughly washed with tap water and then rinsed with double-distilled water to remove dust particles. Approximately 20 grams of the fresh leaves were finely chopped, mixed with 100 ml of distilled water, and boiled at 60°C in a water bath for 10 minutes. The mixture was then cooled and filtered using a muslin cloth, followed by filtration through Whatman No. 1 filter paper. The resulting pale yellow solution was collected, stored in screw-capped bottles at 4–8°C, and utilized as a reducing agent for the synthesis of Fe_2O_3 nanoparticles.

Preparation and Synthesis of Fe_2O_3-NPs

A 0.01 M Ferrous sulfate solution was prepared. 20 mL of the plant extracts was added dropwise with constant stirring to about 100 mL of the solution for one hour till the colour changed to dark green. The product was centrifuged at 3000 rpm for 15 minutes, washed with ethanol and distilled water, and allowed to dry at room temperature (Mamuru *et al.,* 2022).

Solution and Surface Characterization of Fe_2O_3-NPs

The synthesized metal nanoparticles were confirmed by analyzing the aqueous component. The biomolecules responsible for the reduction of ferrous oxide were identified using a Perkin Elmer Fourier Transform Infrared (FT-IR) Spectrophotometer, which provided insights into the functional groups involved in the process. Additionally, the size and morphology of the nanoparticles were determined using Scanning Electron Microscopy (SEM), which revealed detailed images and dimensions of the synthesized nanoparticles. These analytical techniques collectively validated the successful formation and characteristics of the nanoparticles.

Batch Experiment for Waste Water Remediation of Heavy Metal

In a controlled experimental setup, distinct concentrations of Fe_2O_3 nanoparticles (100, 500, 1000, and 1500 ppm) were prepared and dispersed into 100 mL of wastewater within a 250 mL Erlenmeyer flask. The mixture was intermittently shaken at room temperature (30°C) and maintained at a neutral pH. Samples of approximately 10 mL were collected from the flask at regular contact time intervals (60, 120, and 180 minutes) using variable doses (0.2 and 0.5 g). To ensure accuracy, the experiment was repeated twice under identical conditions. The percentage removal of heavy metals was determined using the Environmental Protection Agency (EPA) method, and the removal efficiency (R%) was calculated to evaluate the effectiveness of the Fe_2O_3 nanoparticles in treating the wastewater (Yadav et al., 2023).

RESULTS AND DISCUSSIONS

Optical Property

Adding 20 mL of extract to the flask containing 0.01 M ferrous sulfate did not immediately change the colour (Fig. 1B), however at the end of one hour the colour of the solution become dark-green (Fig. 1C) which indicates the formation of nanoparticles (Fig. 1D). The colour change is likely due to the excitation of surface plasmon vibrations in FeO nanoparticles (Waqas *et al.,* 2022).

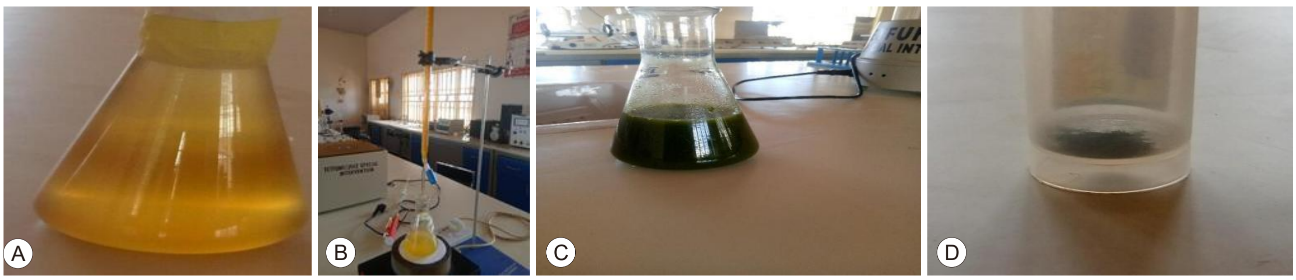


Figure 1. Leaf extract of *Annona squamosa* (A), Ferrous sulfate solution (B), Metal nanoparticles liquid (C), and the Metal nanoparticles solid (D).

Fourier transform infrared spectroscopy analysis

The Fourier transform infrared analysis was done to identify the likely biomolecules responsible for reducing the metal salt to its nanoparticles and capping the reduced metal nanoparticles (Fe_2O_3-NPs) synthesized using *Annona squamosa* leaf extract.

The FT-IR analysis for the iron oxide nanoparticles displayed that there are strong, broad, and medium bands at 3253.53 cm^{-1}, 1623.24 cm^{-1}, 1057.89 cm^{-1}, 804.95 cm^{-1}, 766.54 cm^{-1}, 685.58 cm^{-1}, and 591.45 cm^{-1}. The bands which appeared at 3253.53 cm^{-1} and 1623.24 cm^{-1} correspond to alcohol O-H stretching and conjugated alkene; C=C stretching, respectively, the bands at 1057.89 cm^{-1} and 804.95 cm^{-1} are due to primary alcohol C-O stretching; tri-substituted C=C bending, respectively. The FT-IR bands observed at 766.54 cm^{-1} may be attributed to mono-substituted C-H bending, whereas bands at 685.58 cm-1 and 591.45 cm-1 were apportioned to C-Br stretching and C-I stretching vibrations due to alcohol arising from the flavonoid constituent of the plant metabolites (Das *et al*., 2023).

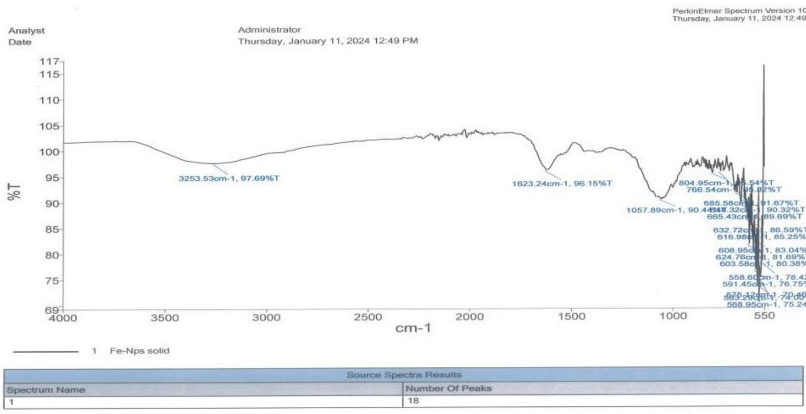


Figure 2. FT-IR absorption spectra of Fe_2O_3 nanoparticles.

The SEM analysis of Fe_2O_3 nanoparticles

The SEM image of the Fe_2O_3 NPs exposed understanding into their morphology and size. The agglomeration of particles designates a likely weak capping effect from the plant extract, resulting in adequate stabilization of the nanoparticles. The particles displayed a uniform distribution and mostly cubic shape, with sizes varying between 25 and 34 nm, as noted by (Nyabadza *et al*., 2023).

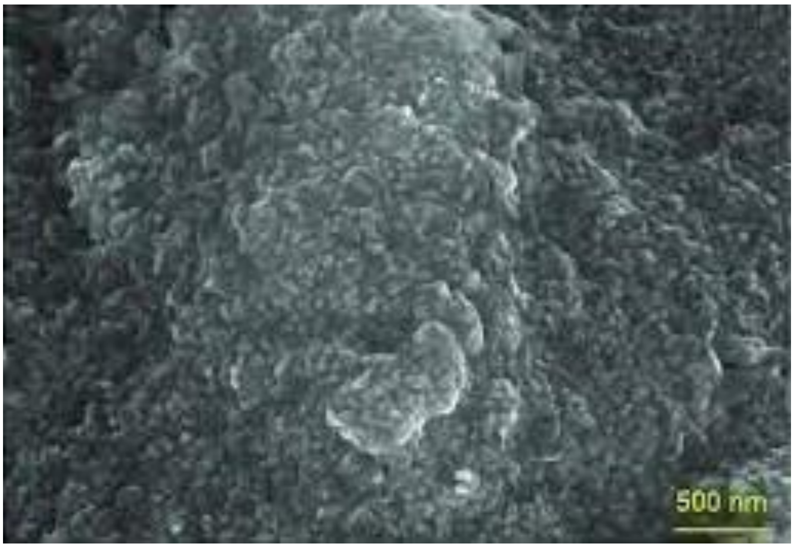


Figure 3. SEM Micrograph of Fe_2O_3-NPs.

Batch Experiment for Waste Water Remediation of Heavy Metal

Table 1 highlights the significant impact of additional ions on adsorption effectiveness, as they compete with the primary ions for chelation sites on the adsorbent. At low concentrations of toxic metals, the availability of ions is limited, reducing scavenging efficiency. However, at higher concentrations, the increased availability of ions enhances removal efficiency, as demonstrated by reduced lead concentration in wastewater from 0.69

mg/L to 0.02 mg/L after remediation. Temperature also plays a crucial role in adsorption processes. Higher temperatures increase the adsorption rate for endothermic adsorption while for exothermic adsorption, the rate decreases. In this study, raising the temperature from 10°C to 21°C improved adsorption, but beyond this point, the process declined due to increased kinetic energy, which caused desorption of metal ions from the adsorbent sites (Priya et al., 2022).

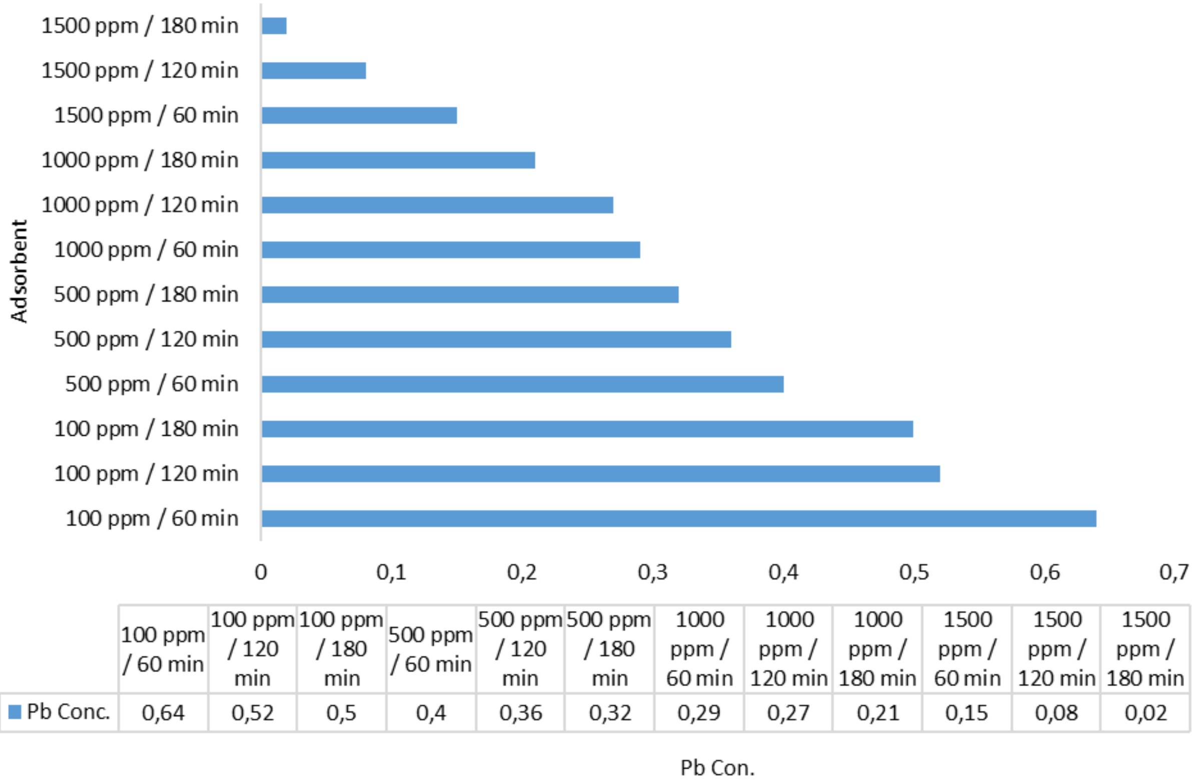
The effectiveness of wastewater remediation from heavy metals also depends on the contact time between the Nano sorbent and metal ions. Increasing the contact time from 60 to 180 minutes significantly enhances adsorption efficiency, as more time allows for greater interaction between the pollutants and the adsorbent's active chelation sites. Initially, adsorption occurs rapidly due to the availability of open active sites, but as these sites become occupied, the process slows down. This trend was similarly observed in the adsorption of divalent mercury ions on CANPs, where adsorption increased significantly as contact time rose from 0 to 90 minutes (Tiwari et al., 2023).

Additionally, adsorbent dosage plays a critical role in adsorption capacity. Increasing the dosage from 100 to 1500 ppm improved adsorption efficiency to 97% by providing more active sites for metal chelation. However, further increases in dosage led to a buildup of the adsorbent, reducing surface area and adsorption capacity. The pH of the reaction medium also significantly influences adsorption reactions and capacity, as it affects the availability of active sites and the interaction between the adsorbent and metal ions (Fei et al., 2022).

Figure 4. Formation of nanoparticle bits (A), Different concentrations of nanoparticle bits in waste water (B), and remediated water with different concentrations of nanoparticles and contact time (C).

Table 1. Adsorption process.

Metal Ions	Adsorbent (ppm)	Contact Time (min)	Initial Metal Conc. (mg/L)	Dosage of Adsorbent (mg/L)	Final Metal Conc. (mg/L)	pH	Temp. (K)
Fe (II)	FeONPs 100	60	0.69	0.2	0.64	8.90	21.33
"	FeONPs 500	60	"	"	0.52	"	"
"	FeONPs 1000	60	"	"	0.50	"	"
"	FeONPs 100	120	"	"	0.40	"	"
"	FeONPs 500	120	"	"	0.36	"	"
"	FeONPs 1000	120	"	"	0.32	"	"
"	FeONPs 100	180	"	"	0.29	"	"
"	FeONPs 500	180	"	"	0.27	"	"
"	FeONPs 1000	180	"	"	0.21	"	"
"	FeONPs 1500	60	"	0.5	0.15	"	"
"	FeONPs 1500	120	"	0.5	0.08	"	"
"	FeONPs 1500	180	"	0.5	0.02	"	"



	100 ppm / 60 min	100 ppm / 120 min	100 ppm / 180 min	500 ppm / 60 min	500 ppm / 120 min	500 ppm / 180 min	1000 ppm / 60 min	1000 ppm / 120 min	1000 ppm / 180 min	1500 ppm / 60 min	1500 ppm / 120 min	1500 ppm / 180 min
Pb Conc.	0,64	0,52	0,5	0,4	0,36	0,32	0,29	0,27	0,21	0,15	0,08	0,02

Figure 5. Adsorption chart of wastewater.

CONCLUSION

The study showcased the successful application of eco-friendly Fe_2O_3 nanoparticles, synthesized from *Annona squamosa* leaf extract, for wastewater remediation, particularly in removing heavy metals like lead. These nanoparticles, characterized by their simplicity, rapid production, and environmental safety, provided a larger surface area for reactions than conventional methods, significantly enhancing their effectiveness. This green synthesis approach offers a sustainable and straightforward alternative to traditional wastewater treatment techniques, which are often complex and pose environmental risks.

The efficacy of Fe_2O_3 nanoparticles in treating wastewater from the River Benue was confirmed through various analyses. FT-IR spectroscopy identified flavonoids in the leaf extract as key agents in the synthesis and stabilization of the nanoparticles. SEM imaging revealed that the nanoparticles were cubical, well-stabilized, and ranged in size from 25 to 34 nm. Atomic absorption spectroscopy demonstrated a remarkable reduction in lead contamination levels, from 0.69 mg/L to 0.02 mg/L, achieving a 97% removal efficiency. These findings highlight the potential of green-synthesized Fe_2O_3 nanoparticles as a highly effective and environmentally friendly solution for wastewater treatment.

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***Authors Contributions*:** Saminu Hamman Barau designed the study, carried out data collection, performed laboratory work, and drafted the manuscript. Abdulazeez Mumsiri Abaka, Jameelah Bakari, and Suleiman Alhaji Saidu reviewed and proofread the manuscript. All authors read and approved the final version of the manuscript.

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Competing Interest: All authors declare no competing interest.

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