



Biosynthesis of Magnetite Nanoparticles utilizing *Azadirachta indica* leaves for removal of Cadmium



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ABSTRACT

Cadmium toxicity occurs when the body is exposed to cadmium via industrial emissions, infected food, cigarette smoke, contaminated water etc. This affects the kidney, bones, lungs, liver etc. In this study, Magnetite nanoparticles were biosynthesized utilizing *Azadirachta indica* leaves, oven dried at 1200°C for 1 hr and calcined for 4 hr at 500°C in a muffle furnace. The synthesized material was characterized using Energy-Dispersive X-ray Spectroscopy (EDX), Fourier Transform Infrared Spectroscopy (FTIR) and Scanning Electron Microscopy (SEM) for the elemental composition, functional group and morphology. Adsorption studies were carried out and the percentage of degradation of cadmium were investigated under various conditions such as pH, contact time, temperature and initial concentration of the *Azadirachta indica* mediated iron oxide nanoparticle. The result showed that the percentage of Cadmium removal was 53.83% at 3.13mg/L, at contact time of 40min with highest removal percentage of 53.14%, at temperature of 65°C, 76.19% optimum removal of Cadmium was recorded and at pH 7, the optimum cadmium removal was 59.65%. The equilibrium data was best represented by Langmuir and Freundlich isotherm model and the Pseudo-first order kinetic model better explained the kinetic data for the Cadmium ion. The findings showed that *Azadirachta indica* leaf extract have proven to be an effective capping agent and a sustainable bio material for cadmium removal in polluted water.

Keywords:

Biosynthesis;
Magnetite
Nanoparticles;
Azadirachta indica
leaves;
Cadmium;
Aqueous Solution.

INTRODUCTION

In the expansive arena of environmental science, where the very survival of our planet hangs in the balance, the narrative of heavy metal contamination unfolds as a profound tale of caution and consequence (Alzohairy, 2016). The rapid surge of modern industry, propelling economic growth and technological marvels, has unwittingly sowed the seeds of environmental peril (Award *et al*, 2021). Cadmium (Cd), once mere elements confined to the periodic table, have metamorphosed into stealthy adversaries, infiltrating ecosystems insidiously and relentlessly (Balali-Mood *et al*, 2021). The toxic tendrils weave through soil, water, and air, permeating the core of life itself. In this delicate ecological equilibrium, the clamour of progress clashes with the whispers of environmental sustainability (Chen X, 2015, Joudeh *et al*, 2022, Rashid *et al*, 2023).

Amidst this tumultuous backdrop, nanotechnology emerges as a ray of scientific hope, challenging the conventional boundaries of environmental remediation (Bhuiyan *et al*, 2020).

Among the myriad nanomaterials, magnetite nanoparticles stand as formidable entities, armed with magnetic properties that empower them to attract, capture, and immobilize heavy metal ions (Bhuiyan *et al*, 2020). Their minute size, coupled with a surface area that defies their microscopic stature, provides an ideal platform for intricate interactions with heavy metal contaminants (Award *et al*, 2021). They transform into molecular sponges, absorbing the pollutants that imperil our ecosystems and livelihoods.

Yet, the synthesis of these nanoparticles poses ethical and environmental quandaries. Traditional methods, often involving harsh chemicals and energy-intensive processes, cast a shadow over the essence of sustainable solutions (Chen X, 2015). It is within this crucible of scientific dilemma that the concept of green synthesis emerges, illuminating a pathway toward environmental harmony (Das *et al*, 2023). At its core lies *Azadirachta indica*, the revered neem tree. Its leaves, adorned with nature's biochemical marvels, offer a sustainable source for nanoparticle synthesis.

This fusion of nature and technology, where ancient botanical wisdom converges with cutting-edge nanoscience, epitomizes the essence of eco-friendly innovation (Del Prado-Audelo *et al*, 2021).

This monumental scientific endeavour does not merely scratch the surface; it delves deep into uncharted territory. Its purpose is not just to optimize the green synthesis process but to immerse in a symphony of scientific exploration. X-ray diffraction (XRD) serve as lenses dissecting the synthesized nanoparticles, unveiling their crystalline secrets and morphological intricacies (Duttmann *et al*, 2018). Fourier-transform infrared spectroscopy (FTIR) deciphers the chemical dialogues, shedding light on the intimate bonds between the nanoparticles and their botanical origins (Ethaib *et al*, 2022). Scanning electron microscopy (SEM) (Duttmann *et al*, 2018).

Yet, this scientific odyssey transcends the confines of the laboratory, venturing into the heart of contaminated environments where heavy metal ions pervade the natural order. Here, the synthesized magnetite nanoparticles emerge as ecological guardians, fighting pollution beyond the realm of laboratory glassware (Girish K, 2018, Guerra *et al* 2018). The research immerses itself in the intricate web of real-world scenarios, where pH, temperature, and diverse environmental matrices become variables in the complex equation of environmental remediation (Harmansah *et al* 2022).

In the annals of scientific significance, this research marks a paradigm shift, not only showcasing scientific prowess but emphasizing the moral imperative of responsible innovation. It advocates for a future where scientific inquiry intertwines with ecological ethics, measuring progress not just in technological leaps but in restored environmental harmony. Through the synthesis of magnetite nanoparticles, a humble leaf finds its place in the chronicles of environmental stewardship. As the world grapples with the spectre of heavy metal contamination, this research stands as a testament to human ingenuity, environmental mindfulness, and the audacious spirit to protect our planet. Through nanoparticle synthesis, humanity embarks on a transformative journey, not just to unveil the secrets of nature but to honour them, crafting a sustainable legacy for generations yet unborn.

Heavy metals like Cd is a harmful pollutant found in our environment due to industrial activities and farming. It doesn't break down naturally and can harm both nature and people (Jain *et al* 2015). Scientists have been trying to find effective ways to remove this pollutant (Jaishankar *et al*, 2014, Joudeh *et al*, 2022). Nanoparticles have shown promise in cleaning up heavy metals (Khanal. S. 2021). Among these, magnetite nanoparticles are particularly interesting because they can attract and trap heavy metals. However, making these nanoparticles often involves

using harmful chemicals and a lot of energy [Khayyun *et al*, 2019].

In recent years, researchers have explored an eco-friendlier approach called "green synthesis (Ethiab *et al*, 2022)." This method uses natural substances, like plant extracts, to create nanoparticles without harming the environment (Meador *et al*, 2005). *Azadirachta indica*, or neem, a tree known for its medicinal properties, has caught scientists' attention. Neem leaves are rich in special chemicals that could help create these eco-friendly nanoparticles (Mensah *et al*, 2021).

Despite the potential of neem-based magnetite nanoparticles, there's still a lot we don't know. We need to figure out the best way to make these particles, understand their properties, and see how well they can clean up heavy metals. This study aims to fill these gaps in our knowledge. By exploring the green synthesis of magnetite nanoparticles using neem leaves, we hope to find a safer and more sustainable solution for dealing with heavy metal pollution. This research could lead to eco-friendly methods for cleaning our environment, making it safer for everyone.

MATERIALS AND METHODS

Chemicals

Analytical grade iron (II) sulfate heptahydrate ($\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$) and sodium acetate were purchased from Sigma Aldrich. All the chemicals were used without further purification.

Collection and preparation of plant extract

Azadirachta indica leaves with voucher number 109 were harvested from Ifaki-Ekiti, Ekiti State, Nigeria, and the leaves were subsequently extracted. The leaves were meticulously cleaned using distilled water to eliminate any impurities, washed thoroughly, and then weighed. A total of 129.86 g of the leaves was measured and blended with 1298.6 mL of water. The resulting mixture underwent filtration, and the obtained filtrate was preserved for subsequent analysis. This process ensured the preparation of a solution suitable for further examination and investigation.

Green synthesis of iron oxide nanoparticles

The iron oxide nanoparticles were synthesized following an environmentally friendly procedure using *Azadirachta indica* Leaves, as described in a previously published method with slight adjustments (Morel *et al*, 2008). In a standard experiment, 9.6 g of iron (II) sulfate heptahydrate ($\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$) was dispersed in a beaker containing 120 mL of *Azadirachta indica* Leaves extract solution along with 4g of PVA, and the mixture was vigorously stirred for 2 hours at 100 °C. The resulting solution exhibited a homogeneous dark reddish-brown colour after 2 hours, indicating the formation of iron

oxide nanoparticles. Subsequently, the mixture was allowed to cool to room temperature. To purify the synthesized product, a centrifuge machine was employed, and the product was neutralized using distilled water/ethanol before being dried in a vacuum oven at 1200 °C for 1 hr. Further characterization was conducted after calcination for 4 hours at 500°C. This multi-step process ensured the successful synthesis and purification of iron oxide nanoparticles from *Azadirachta indica* Leaves, establishing a foundation for subsequent analyses and applications.

Adsorption Study

The adsorption studies were carried out as described by Ifijen *et al.* (2020) with some modifications. In the adsorption studies, 0.2 grams of *Azadirachta indica*-mediated iron oxide nanoparticles were meticulously combined with 50 mL of 0.1 M solutions containing cadmium (II) sulfide each treated separately. This amalgamation occurred at room temperature, and the duration of the interaction was maintained for a period of 10 minutes. The purpose of this experimental setup was to investigate the adsorption capacity and efficiency of the synthesized nanoparticles for cadmium ions under specific conditions. The controlled environment allowed for a systematic examination of the adsorption process, shedding light on the affinity and performance of the adsorbent material toward each heavy metal ion individually.

The percentage (%) removal of Cd were noted by using the equation below

$$\% \text{ removal} = \frac{C_o - C_e}{C_o} \times 100$$

The amount of heavy metal per gram of the iron oxide nanoparticle was calculated using this equation

$$q_e = \frac{C_o - C_e}{W_{nfe}} \times V$$

where

C_o is the initial concentration of heavy metal

C_e is the equilibrium concentration of heavy metal

W_{nfe} is mass of iron oxide nanoparticles

V is the volume of heavy metal solution

RESULTS AND DISCUSSION

Elemental Composition of Iron Oxide Nanoparticles Mediated by *Azadirachta indica*

Figure 1 shows the elemental composition of Iron Oxide nanoparticles mediated by *Azadirachta indica*. The weight concentration of oxygen was 31.95%, 63.96% for iron and 4.09% for carbon. The presence of carbon was

as a result of the calcination. This is similar to the report of Neto *et al.*, 2023. The observed elemental composition is significant as it influences the properties and potential applications of the iron oxide nanoparticles. The high concentration of oxygen suggests the presence of oxygen-rich functional groups or surface coatings on the nanoparticles, which can impact their surface chemistry and reactivity. The significant concentration of iron is consistent with the formation of iron oxide nanoparticles, and its presence is crucial for the magnetic properties associated with these materials. Understanding the elemental composition of *Azadirachta indica*-mediated iron oxide nanoparticles through such analysis provides valuable information for studying their structure, properties, and potential uses in various fields, including biomedicine, catalysis, and environmental remediation.

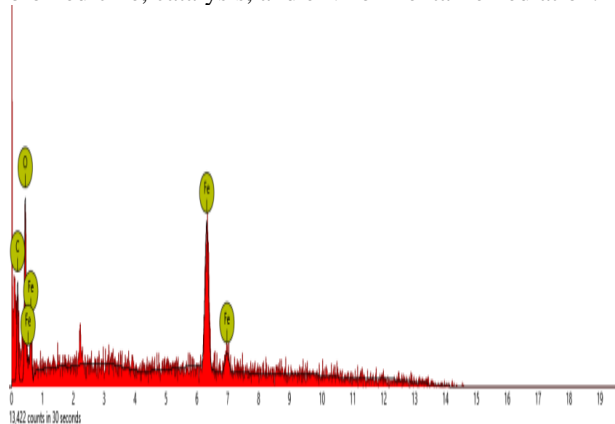


Figure 1: Energy-Dispersive X-ray spectroscopy (EDX) analysis of the elemental composition of *Azadirachta indica*-mediated iron oxide nanoparticles

Functional Group Analysis of *Azadirachta indica*-mediated iron oxide nanoparticles

Figure 2 displays the FTIR spectra of *Azadirachta indica*-mediated iron oxide nanoparticles which provides crucial insights into the molecular composition and interactions involved in the synthesis process. Notably, the broad absorption bands at 3294 cm^{-1} are attributed to the stretching vibrations of hydroxyl groups (-OH) (Rajendra *et al.*, 2023). Moreover, absorption peaks around 1084 cm^{-1} indicate the presence of carbonyl groups (C=O), suggesting the involvement of organic compounds such as polysaccharides and proteins from the extract (Yesiltas *et al.*, 2021). These organic components likely contributed to the formation and stabilization of the iron oxide nanoparticles. The peaks observed around 693 and 998 cm^{-1} in the finger print region confirms the presence of Fe-O, unequivocally establishing the synthesized nanoparticles as iron oxide (Morel *et al.*, 2008; Rajendra *et al.*, 2023).

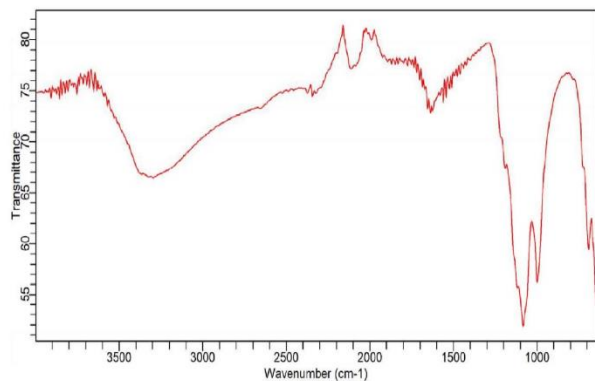
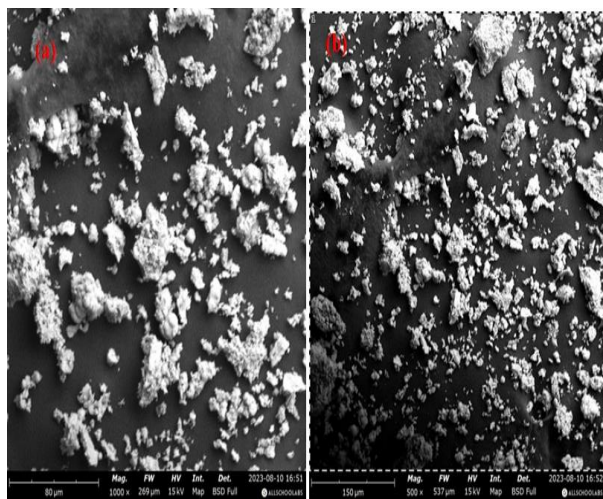


Figure 2: FTIR spectra of *Azadirachta indica*-mediated iron oxide nanoparticles

Scanning Electron Morphology of the *Azadirachta Indica*-Mediated Iron Oxide Nanoparticles

The morphology of *Azadirachta Indica*-mediated iron oxide nanoparticles at 80 and 150 μ m revealed that particles are shapeless and this suggests a certain level of irregularity and complexity in the structure of *Azadirachta indica*-mediated iron oxide nanoparticles, **agglomeration, nanoscale size and sample preparation (Samrot et al, 2018).**



Adsorption of Cadmium

Effect of initial concentration on the adsorption of *Azadirachta indica*-mediated iron oxide nanoparticle

Figure 4 shows the influence of concentration on the removal efficiency of cadmium from contaminated water using *Azadirachta indica*-mediated iron oxide nanoparticles as an adsorbent. The result in Figure 4 shows that percentage removal of cadmium decreases from 40.4% to 39.9% as the concentration reduces from 12.5 mg/L to 6.25 mg/L. This implies that higher removal can be achieved at lower concentrations. This can be related to the work of Aremu et al, 2023.

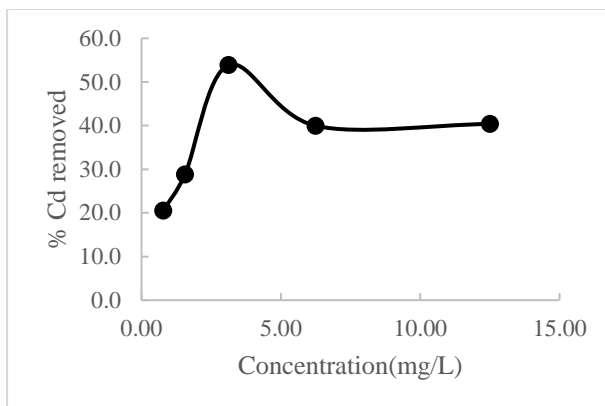


Figure 4: Effect of concentration on the removal of cadmium from contaminated water using *Azadirachta indica*-mediated iron oxide nanoparticles as an adsorbent.

Effect of contact time on the adsorption of cadmium using *Azadirachta indica*-mediated iron oxide nanoparticles

Figure 5 shows the effect of time on the removal of cadmium ion from contaminated water using *Azadirachta indica*-mediated iron oxide nanoparticles as an adsorbent. The results as the contact time increases from 10 min to 40 min, the percentage removal of cadmium increases from 4.38% to 53.14%. This implies that there are still active sites available for the removal process of cadmium in *Azadirachta indica*-mediated iron oxide nanoparticles. This is similar to the work of Sirsath et al. 2015.

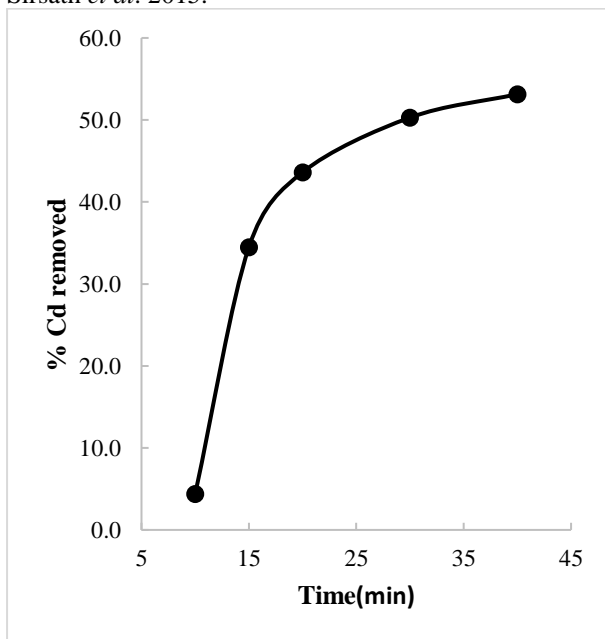


Figure 5: Effect of contact time on the removal of heavy metals of cadmium from contaminated water using *Azadirachta indica*-mediated iron oxide nanoparticles as an adsorbent.

Effect of temperature on the adsorption of cadmium using *Azadirachta indica*-mediated iron oxide nanoparticles
 The result in figure 9 shows that increasing the temperature from 25°C to 65°C increases the percentage

removal of cadmium from 33.3% to 76.2%. This suggests that increased temperatures results in increased kinetic energy which invariably increases the removal efficiency of cadmium (Al-Naimi & Al-Ghouti, 2020).

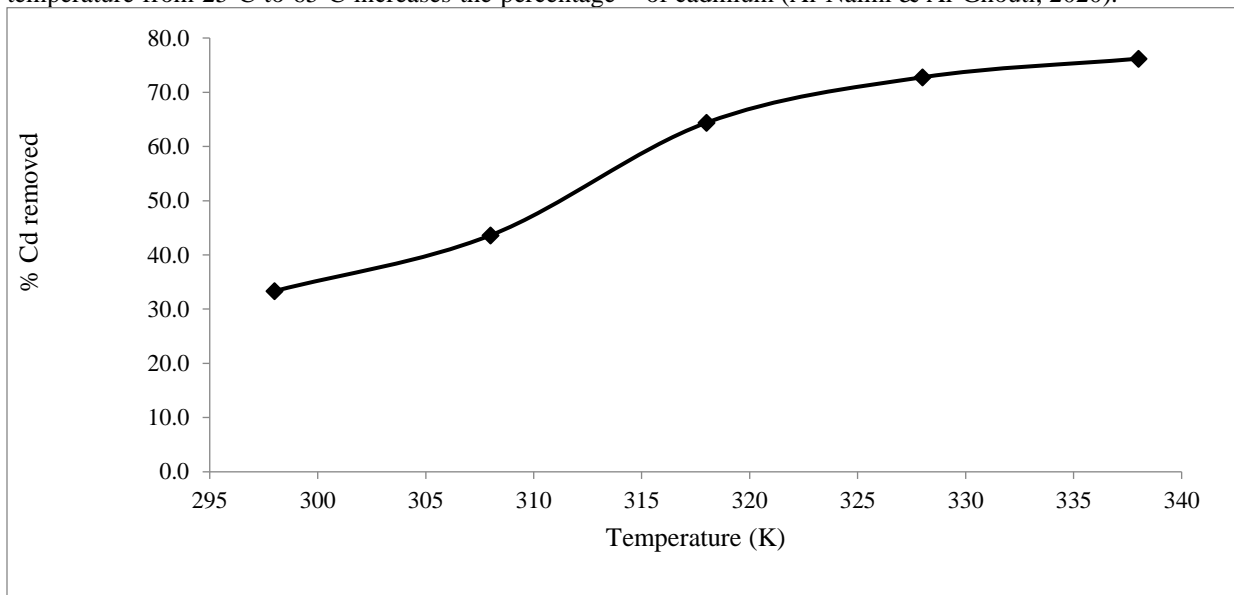


Figure 6: Effect of temperature on the removal of cadmium ions from contaminated water using *Azadirachta indica*-mediated iron oxide nanoparticles as an adsorbent.

Table 1: Data showing the thermodynamic factors for Cd

T(K)	Final Ce(mg/l)	q _e	K	InK	Del S=intercept x 8.314	Del H= Slope x 8.314	Del G= del H – TdelS	1/T	Log Kc for Cd
298	8.3333	0.3333	0.04	- 3.2188	58.3319	- 18102.9	- 35485.80	0.0034	- 0.3010
308	7.0476	0.4362	0.06892	- 2.7838	58.3319	- 18102.9	- 36069.13	0.0032	- 0.1115
318	4.4524	0.6438	0.14459	- 1.9338	58.3319	- 18102.9	- 36652.44	0.0031	0.2591
328	3.4048	0.7276	0.21371	- 1.5432	58.3319	- 18102.9	- 37235.76	0.0030	0.4267
338	2.9763	0.7619	0.256	- 1.3625	58.3319	- 18102.9	- 37819.08	0.0029	0.5051

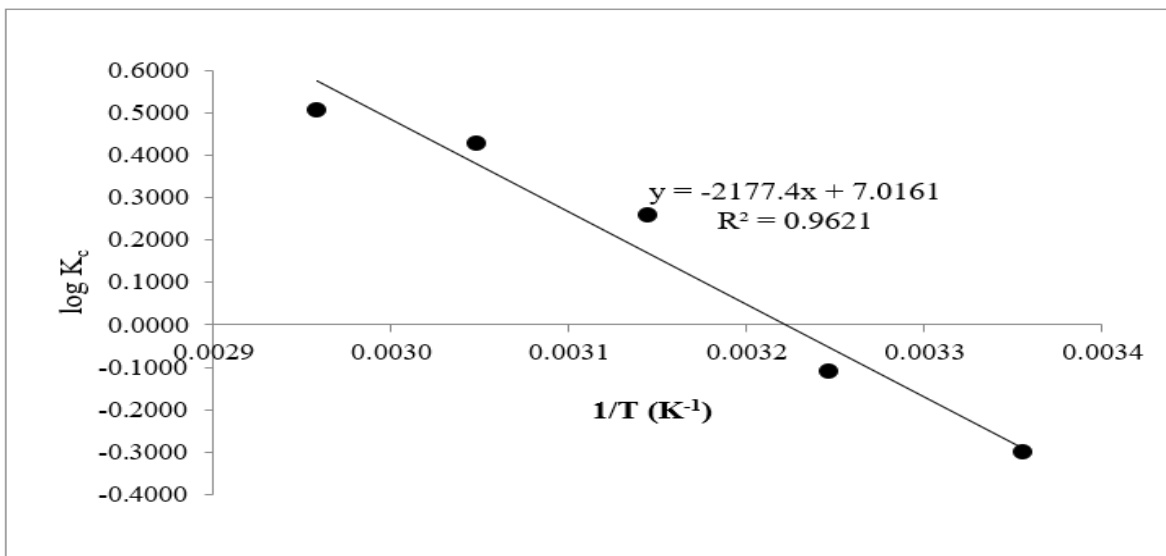


Figure 7: Graphical representation of thermodynamic factors for Cd ion

Effect of pH on the adsorption of cadmium using *Azadirachta indica*-mediated iron oxide nanoparticle

Figure 8 the effect of pH on the removal efficiency of cadmium from contaminated water using *Azadirachta indica*-mediated iron oxide nanoparticles as an adsorbent. The results shows that as the pH reduces in the acidic medium, the removal efficiency of cadmium from 42.3% to 7.62% which implies that lower acidic medium favours higher remover efficiency ().

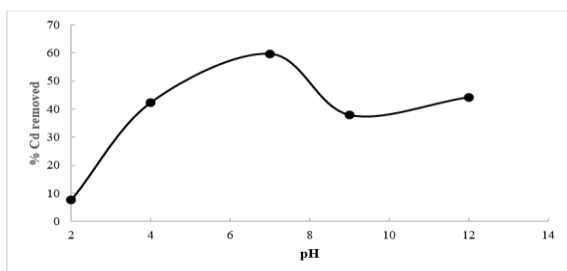


Figure 8: Effect of pH on the removal of cadmium ions from contaminated water using *Azadirachta indica*-mediated iron oxide nanoparticles as an adsorbent.

Adsorption Isotherms: Langmuir and Freundlich Models for cadmium ions

The Langmuir and Freundlich isotherms for cadmium ions adsorption is presented in Figure 9 and 10. The results showed that Langmuir model best fit the adsorption of cadmium ions with R² value of 0.7019 as against Freundlich model with lower R² value of 0.3498. This suggest a good monolayer adsorption on a homogeneous surface for Langmuir model (Jain *et al*, 2015; Chen, 2015) while it shows a multilayer adsorption mechanism on heterogeneous surfaces for Freundlich model (Khayyun *et al*, 2019; Tonk *et al*, 2022).

Table 2: Adsorption Isotherm for Cadmium ions

Initial (mg/L)	Final Ce(mg/L)	1/Ce	LogCe	LnCe	Qe	1/qe	Logqe
12.50	7.4524	0.134185	0.872295	2.008534	0.4038	2.476415	-0.39382
6.25	3.7525	0.26649	0.574319	1.322418	0.3996	2.502488	-0.39837
3.13	1.4416	0.693653	0.158858	0.365784	0.5387	1.85641	-0.26867
1.56	1.1123	0.899059	0.046212	0.106407	0.2884	3.467742	-0.54005
0.78	0.6207	1.611112	-0.20713	-0.47692	0.2053	4.871795	-0.68769

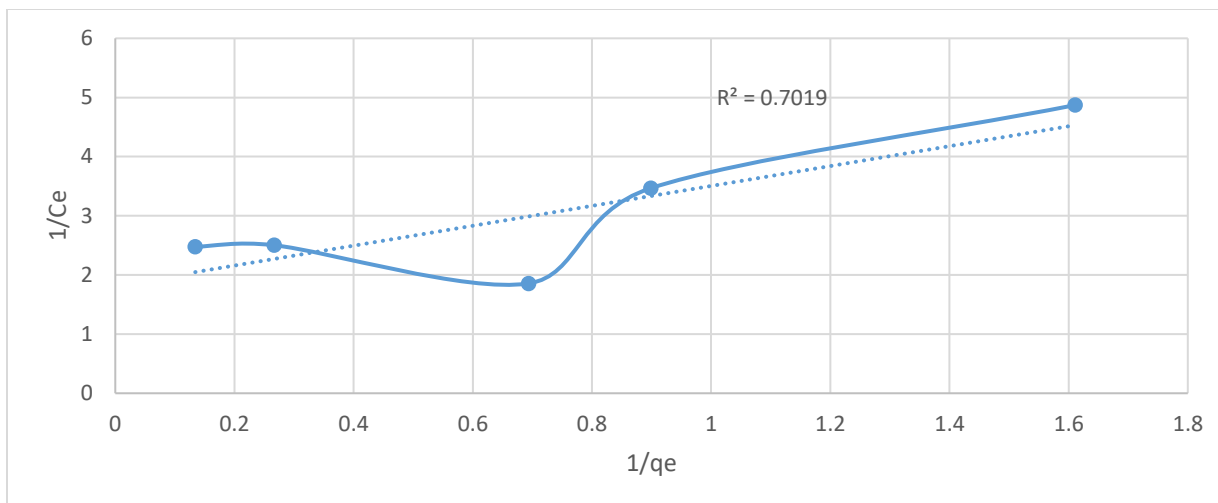


Figure 9: Adsorption isotherm (Langmuir) for Cadmium ions

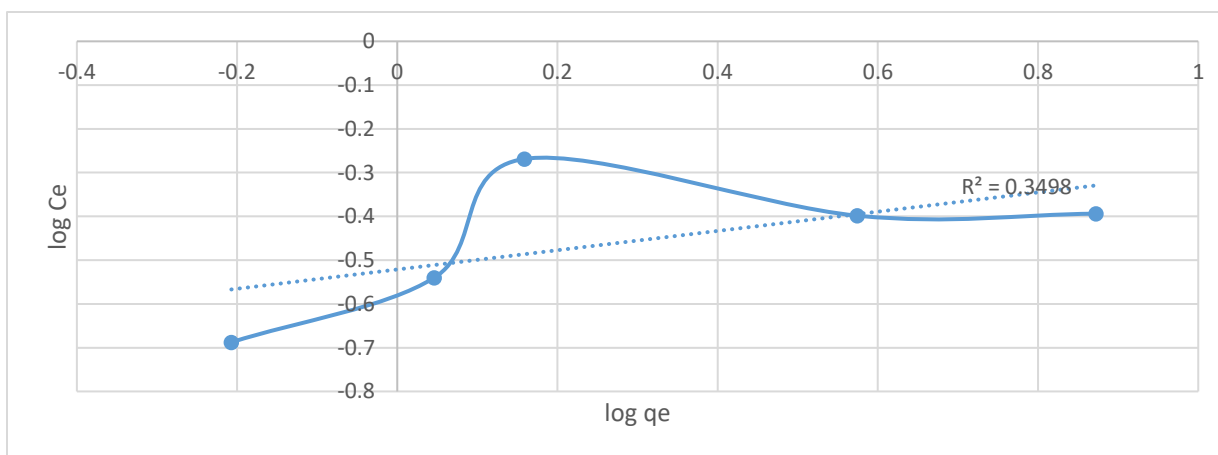


Figure 10: Adsorption isotherm (Freundlich) for Cadmium ions

Kinetic studies

The kinetics studies presented in Figure 11 and 12 for the adsorption of cadmium ions from contaminated water using *Azadirachta indica* mediated iron oxide nanoparticles. The results showed that pseudo-first-order kinetics best fits the removal of cadmium ions with R² value of 0.8129. This suggests that the rate of adsorption

is directly proportional to the number of unoccupied sites on the adsorbent (Islam *et al.*, 2021; Daset *et al.*, 2010; Daset *et al.*, 2023). The pseudo-second-order kinetic with a lower R² value of 0.2182 suggests that the adsorption kinetics may not be better represented by this model (Das *et al.*, 2010; Das *et al.*, 2023).

Table 3: Adsorption Kinetic data for the adsorption of cadmium ion from contaminated water using *Azadirachta indica* mediated- iron oxide nanoparticles.

T	C _i	C _e	1/C _e	LogC _e	InC _e	q _e	1/q _e	Inq _e	t/qt	In(qe-qt)
10	12.50	0.4038	2.4765	-0.3938	-0.9068	0.0548	18.2482	-2.9041	182.6	-0.0245
15	12.50	0.3448	2.900	-0.4624	-1.0648	0.4310	2.3202	-0.8416	34.81	-0.2449
20	12.50	0.4362	2.2925	-0.3603	-0.8297	0.5452	1.8342	-0.6066	36.68	-0.3422
30	12.50	0.5029	1.9885	-0.2985	-0.6874	0.6286	1.5908	-0.4643	47.73	-
40	12.50	0.5314	1.8818	-0.2746	-0.6322	0.6643	1.5053	-0.4090	60.23	-0.4740

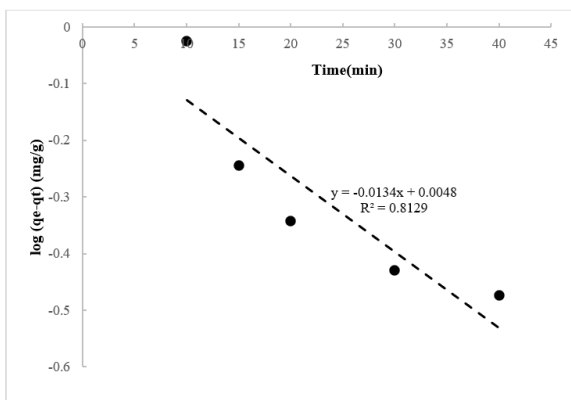


Figure 11: Pseudo first order adsorption kinetic isotherm of Cadmium ion using *Azadirachta indica* mediated-iron oxide nanoparticle.

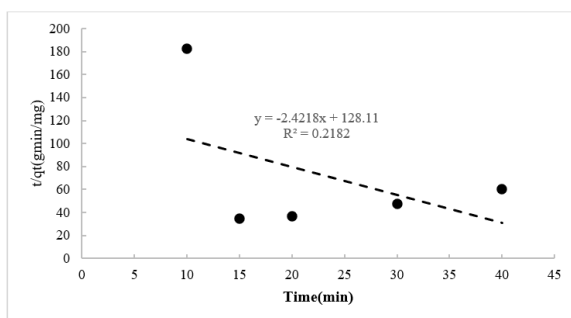


Figure 12: Pseudo-second order adsorption kinetic isotherm of cadmium ion using *Azadirachta indica* mediated- iron oxide nanoparticles.

In this study, *Azadirachta indica* leaf extract was used to synthesize iron oxide nanoparticles and characterized using SEM for morphological studies, FTIR for functional group investigation and EDX for elemental composition. The morphological studies showed that the particles were shapes as a result of the method of synthesis, the observed peaks around 693 cm^{-1} and 998 cm^{-1} indicates Fe-O band in the fingerprint region of the spectra. The percentage weights of Fe, O and C were 63.96%, 31.95% and 4.09% which indicates that FeO nanoparticles were actually synthesized. The different influential parameters considered were effects of contact time, initial concentration, temperature and pH. The removal efficiency of cadmium was achieved at 3.13 mg/L initial concentration, temperature of 65 $^{\circ}\text{C}$, pH 7 and 40 min contact time. The kinetic studies of cadmium removal from wastewater using *Azadirachta indica*-mediated iron oxide nanoparticles followed pseudo-first-order while Langmuir model best fits the removal efficiency of cadmium. *Azadirachta indica*-mediated iron oxide nanoparticles have proven to be a good adsorbent for cadmium removal in wastewater.

CONCLUSION

In this study, *Azadirachta indica* leaf extract was used to synthesize iron oxide nanoparticles and characterized using SEM for morphological studies, FTIR for functional group investigation and EDX for elemental composition. The morphological studies showed that the particles were shapes as a result of the method of synthesis, the observed peaks around 693 cm^{-1} and 998 cm^{-1} indicates Fe-O band in the fingerprint region of the spectra. The percentage weights of Fe, O and C were 63.96%, 31.95% and 4.09% which indicates that FeO nanoparticles were actually synthesized. The different influential parameters considered were effects of contact time, initial concentration, temperature and pH. The removal efficiency of cadmium was achieved at 3.13 mg/L initial concentration, temperature of 65 $^{\circ}\text{C}$, pH 7 and 40 min contact time. The kinetic studies of cadmium removal from wastewater using *Azadirachta indica*-mediated iron oxide nanoparticles followed pseudo-first-order while Langmuir model best fits the removal efficiency of cadmium. *Azadirachta indica*-mediated iron oxide nanoparticles have proven to be a good adsorbent for cadmium removal in wastewater.

Conflict of Interest: No conflict of interest to declare

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