

**EFFECT OF *PICRALIMA NITIDA* STEM BARK RESIDUE AS ADSORBENT IN
DOMESTIC WASTEWATER (SULLAGE) TREATMENT**

BY

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CERTIFICATION

This is to certify that this work was carried out by Ugbesia, Ruth Aladi, Mat. No. PG/SPE2415171, of Department of Environmental Standards, Faculty of SPESSE, University of Benin, Benin City, Edo State, Nigeria, in partial fulfilment of the requirements for the award of Master of Science (M.Sc) in Environmental Standards.

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Name:.....

Signature and Date:.....

DEDICATION

This work is dedicated to the Almighty God for his guidance and protection over my life, and to my ever supportive husband, Engr. Hector Ehi Ugbesia.

ACKNOWLEDMENT

I would like to express my appreciation to God Almighty for His faithfulness and blessings in my life, his mercy and grace that have brought me this far. I will forever be grateful.

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ABSTRACT

The rising issue of domestic wastewater pollution, mainly from heavy metals and organic pollutants, poses significant risks to public health and environmental sustainability in Nigeria. Traditional treatment methods are often costly and energy-intensive, emphasising the need for affordable, eco-friendly alternatives. This study investigated the use of *Picralima nitida* stem bark residue as a sustainable biosorbent for removing iron (Fe^{3+}), zinc (Zn^{2+}), chemical oxygen demand (COD), and total dissolved solids (TDS) from domestic wastewater. The research focused on evaluating the adsorption efficiency, identifying optimal conditions, modelling the underlying mechanisms, and comparing the cost-effectiveness to commercial activated carbon.

The study conducted an experimental investigation using batch adsorption tests in controlled laboratory conditions. *P. nitida* bark residue was washed, oven-dried, ground, and chemically activated before analysis. The adsorbent was characterised through Fourier Transform Infrared Spectroscopy (FTIR), Scanning Electron Microscopy (SEM), and Brunauer–Emmett–Teller (BET) surface analysis. Parameters such as dosage, contact time, temperature, and pH were systematically varied to evaluate performance. Additionally, Langmuir and the Freundlich isotherm models were employed to interpret the equilibrium data. A cost analysis was also performed to assess the economic viability of the adsorbent.

The results indicated that adsorption efficiency increased with longer contact time and stabilised after 90 minutes. The highest removal rates were 100% for Fe^{3+} , 99.60% for Zn^{2+} , 98.90% for COD, and 89.97% for TDS at near-neutral pH and ambient temperature. The equilibrium data conformed to the Langmuir isotherm ($R^2 = 0.9940$), indicating monolayer adsorption. Economically, the treatment cost was ₦19.50 per litre, which is roughly 30–40% lower than that of commercial activated carbon. The study concludes that *Picralima nitida* stem bark residue is an effective, cost-efficient, and sustainable biosorbent for domestic wastewater treatment, supporting Nigeria's initiatives in sustainable waste utilisation and SDG 6 (Clean Water and Sanitation).

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CHAPTER ONE

INTRODUCTION

1.1 Background of the Study

Water is an essential resource for sustaining life and is required by most living organisms (Shan *et al.*, 2017). Water covers approximately 71% of the Earth's surface. The water we use in our household can come from different sources, such as streams, rivers, wells, boreholes, and natural springs (Forster & Pinedo, 2016).

Rising pollution from point and non-point sources in agriculture, industry, and households, driven by the ever-increasing population, urbanisation, and climate change, has led to a significant increase in the volume of domestic wastewater globally. Domestic wastewater, particularly sullage, which includes effluents from kitchens, bathrooms, and laundry, is often discharged untreated or partially treated into the environment, especially in developing countries (UN-Habitat, 2020). This practice contributes to environmental degradation, contaminates groundwater and surface water sources, and poses serious public health risks due to the presence of organic pollutants, surfactants, suspended solids, and, in some cases, trace heavy metals (WHO, 2017; Oladejo *et al.*, 2022).

Domestic wastewater contains a variety of pollutants, including organic matter, nutrients, and pathogens, which can harm human health and the Environment. Contaminant removal from wastewater has become a significant challenge in tackling water pollution. The treatment of wastewater has become essential due to diminishing water resources, increasing wastewater disposal costs and stricter discharge regulations that have lowered permissible contaminant levels in waste streams. The diversity of water pollutants necessitates a wide range of treatment methods that are both effective and economically feasible (Crini *et al.*, 2019).

In recent years, the development of sustainable, low-cost, and environmentally friendly treatment options has gained momentum. One of the most promising methods is adsorption, a physicochemical process that removes contaminants by adhering them to the surface of solid materials. Although activated carbon is widely used as an adsorbent due to its high surface area and adsorption efficiency, it remains cost-prohibitive for widespread use, particularly in low-income settings (Ali *et al.*, 2022). This has spurred interest in natural and agricultural waste materials as alternative adsorbents. Biosorbents such as fruit peels, nut shells, sawdust, and plant barks have been extensively studied for their potential to remove pollutants due to the presence of functional groups like hydroxyl, carboxyl, and phenolic groups that interact with contaminants in water (Haleem *et al.*, 2020, Ahmad *et al.*, 2021). These materials are not only abundant and renewable but also reduce solid waste when repurposed for water treatment.

The need for scalable, affordable technologies and investigating the potential of *P. nitida* bark as a biosorbent for treating domestic wastewater is timely. This research provides valuable insights into its adsorption efficiency, optimal operational parameters, and economic viability.

1.2 Statement of the Problem

Picralima nitida, commonly referred to as "Akuamma", is a tropical plant found extensively in West and Central Africa. Its bark, seed, and fruit have been used in traditional medicine for their antimalarial, analgesic, and anti-inflammatory properties (Akinmoladun *et al.*, 2021). Despite its widespread use in ethnomedicine, the stem bark residue, which is typically discarded after extraction processes, has not been widely explored for environmental applications. Preliminary phytochemical and structural analyses suggest that the bark contains lignocellulosic compounds and alkaloids, making it a viable candidate for use in adsorption-based treatment technologies (Olabemiwo *et al.*, 2016). Utilising *Picralima nitida* stem bark residue as an adsorbent not only provides a low-cost alternative to commercial

adsorbents but also aligns with the principles of waste valorisation and circular economy, whereby organic residues are repurposed for environmental remediation. Additionally, it offers an eco-friendly solution that could be adapted for decentralised water treatment systems in rural and semi-urban areas. Given the urgency of improving water quality, especially in underserved

The persistent discharge of untreated or inadequately treated domestic wastewater, also known as sullage, into the environment poses a significant threat to public health and ecological balance, particularly in developing countries like Nigeria. Sullage contains a complex mixture of organic and inorganic pollutants, including detergents, oils, pathogens, and sometimes heavy metals, all of which contribute to the degradation of water quality and the spread of waterborne diseases (Oladejo *et al.*, 2022; WHO, 2017).

Conventional wastewater treatment systems, such as activated carbon filtration, membrane separation, and advanced oxidation processes, are effective but often prohibitively expensive and technologically demanding. Their operation requires skilled labour, constant power supply, and maintenance, conditions that are rarely met in rural and peri-urban communities where the burden of wastewater pollution is most severe (Gupta & Nayak, 2019; Wang *et al.*, 2020).

While low-cost, natural adsorbents derived from agricultural or plant waste have emerged as promising alternatives, most research and applications have focused on commonly available materials like coconut shells, banana peels, and sawdust. These materials, though beneficial, may not be readily available or sustainable in all regions, highlighting the need to explore region-specific biomass resources that are both abundant and underutilised. *Picralima nitida* is a medicinal plant widely distributed in West Africa, and its stem bark is often discarded as waste after medicinal extraction. Preliminary evidence suggests that the bark contains lignocellulosic materials and bioactive compounds that may enhance its capacity to adsorb

contaminants (Olabemiwo *et al.*, 2016; Akinmoladun *et al.*, 2021). However, there is a lack of empirical data on its potential application as an adsorbent for treating domestic wastewater. Moreover, critical parameters such as optimal pH, contact time, dosage, and temperature for its effective use remain undefined. Additionally, while environmental sustainability is a key concern, economic viability is equally crucial for the adoption of any treatment method. There is limited information on the cost-benefit analysis of using *P. nitida* stem bark residue compared to commercial adsorbents, which hinders its potential scale-up and implementation in resource-constrained settings

1.3 Aims and Objectives of the Study

The aim of this research is to evaluate the potential of *Picralima nitida* stem bark residue as an effective, low-cost adsorbent for the treatment of domestic wastewater (sullage).

The specific objectives are:

- i. To analyse and refine the stem bark residue of *Picralima nitida* to evaluate its effectiveness as an adsorbent.
- ii. To determine the ideal operating conditions, including pH, contact time, dosage, and temperature, to enhance the efficiency of the adsorbent in domestic wastewater treatment.
- iii. To evaluate the cost-effectiveness of *Picralima nitida* stem bark residue as a potential alternative to traditional adsorbents.

1.4 Scope of the Study

This study focuses on the use of *Picralima nitida* stem bark residue as an adsorbent for the treatment of domestic wastewater (sullage). It involves:

- i. Collection and preparation of *P. nitida* stem bark residue,
- ii. Laboratory-scale treatment of domestic wastewater,

- iii. Analysis of key water quality parameters (e.g., turbidity, pH, BOD, COD, TSS, heavy metals),
- iv. Optimisation of operating conditions (pH, contact time, dosage, temperature),
- v. Cost comparison with conventional adsorbents such as activated carbon.

The study is limited to domestic (non-industrial) wastewater and does not cover microbial pathogen removal or large-scale pilot testing.

1.5 Justification of Study

Wastewater pollution, particularly from industrial effluents and domestic discharges, presents a growing environmental challenge globally. The presence of heavy metals, dyes, and other toxic contaminants poses significant threats to aquatic life, public health, and ecosystem integrity (Ali *et al.*, 2012). Conventional treatment methods such as chemical precipitation, membrane filtration, and ion exchange are often expensive, energy-intensive, and sometimes inefficient at removing low concentrations of pollutants (Babel & Kurniawan, 2003).

Recently, attention has shifted toward low-cost, eco-friendly, and locally available materials for use in adsorption, a highly effective technique for pollutant removal. Agricultural and plant-based waste materials, especially barks, peels, and leaves, have gained popularity due to their abundance and sorption capacity (Demirbas, 2008). In this context, *Picralima nitida* (commonly known as Akuamma) bark represents a promising adsorbent candidate.

Picralima nitida is a tropical medicinal plant whose bark contains several functional groups (e.g., hydroxyl, carboxyl, and amine) associated with the binding of metal ions and organic pollutants (Kone *et al.*, 2004). Its high cellulose and lignin content provides an extensive surface area and active sites favourable for adsorption processes (Njoku *et al.*, 2013).

Picralima nitida, a commonly discarded plant material, as a low-cost sustainable biosorbent, if proven effective, could offer an environmentally friendly and affordable option for

wastewater treatment, particularly in regions where conventional treatment systems are inaccessible or unaffordable and by identifying the optimal operational conditions for its application, the research contributes to the practical scalability of using *P. nitida* stem bark in decentralised water treatment setups. The cost-benefit analysis of this natural adsorbent provides critical data that may support policy formulation, encourage local entrepreneurship in environmental management, and promote circular economy practices in waste valorisation.

Using *Picralima nitida* bark for wastewater treatment aligns with green chemistry principles by valorising plant-based waste and reducing reliance on synthetic materials. It offers a dual benefit of environmental remediation and waste minimisation. Moreover, its availability as a by-product of herbal medicine use in many African communities supports its cost-effectiveness and sustainability.

Despite its recognised pharmacological properties, the application of its bark in environmental clean-up remains underexplored, creating a critical research gap that this study seeks to address.

Therefore, this study justifies the exploration of *Picralima nitida* bark as a viable, natural adsorbent for wastewater treatment, based on its chemical characteristics, local abundance, and potential environmental benefits.

CHAPTER TWO

LITERATURE REVIEW

2.1 Conceptual Review

2.1.1 Domestic Wastewater (Sullage)

Domestic wastewater, also known as sullage, is primarily generated from daily human activities within households, including bathing, cooking, cleaning, and sanitation. It contains a complex mixture of organic and inorganic substances, such as food residues, detergents, oils, fats, nutrients, and microorganisms (UN-Habitat, 2020). Typical parameters used to assess the pollution load in domestic wastewater include biochemical oxygen demand (BOD), chemical oxygen demand (COD), total suspended solids (TSS), nutrients (nitrogen and phosphorus), and pathogenic microorganisms (WHO, 2017).

Studies have shown that untreated domestic wastewater significantly contributes to the degradation of water bodies, leading to eutrophication. In this process, excess nutrients promote algal blooms that deplete oxygen, killing aquatic life (Oladejo *et al.*, 2022). In many developing regions, inadequate sanitation infrastructure means that domestic wastewater is often discharged directly into water bodies without any treatment, posing health risks like cholera, typhoid, and other waterborne diseases (Eze *et al.*, 2020). Consequently, there is an urgent need to develop practical, affordable, and sustainable treatment methods for sullage before its release into the environment.

2.1.2 Adsorption and Biosorption

2.1.2.1 Adsorption

Adsorption is a mass transfer process whereby molecules from a fluid phase (liquid or gas) adhere to the surface of a solid adsorbent. It is widely applied in water and wastewater treatment due to its ability to remove a broad spectrum of contaminants, including heavy metals, dyes, and organic pollutants (Ali *et al.*, 2022). Adsorption is basically a process in which removable species are transported from a liquid phase onto the surface of a solid phase. Depending on the type of attractions between adsorbate and adsorbent, the adsorption process can be divided into two types: Physisorption (Physical adsorption) and Chemisorption (chemical adsorption)c

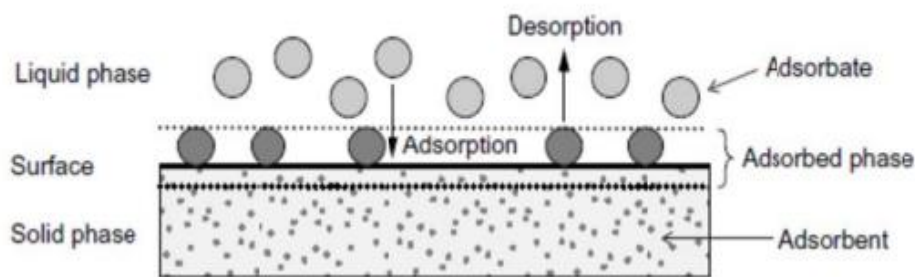


Figure 2.1: schematic diagram of the adsorption process (source: Worch, 2012)

Adsorbed species are bound to the solid surface by physicochemical interactions; Adsorption is usually considered a cost-effective and reliable method for wastewater treatment (Liu *et al.*, 2020). The removal efficiency of adsorption can range up to 99.9%. The United States Environmental Protection Agency (USEPA) has recognised the adsorption process as one of the most effective wastewater treatment techniques, among others (Akhtar *et al.*, 2025). The effectiveness of adsorption depends on several factors, including adsorbent surface area,

porosity, presence of functional groups, pH, temperature, and contact time (Ahmad *et al.*, 2021)

2.1.2.2 Conventional and non-conventional adsorbents for wastewater treatment

Conventional Adsorbents

For several years, conventional adsorbents have gained popularity in liquid-solid adsorption for pollutant removal. Activated carbon has been the standard in industrial water treatment due to its high efficiency. However, the high cost of these materials has driven research into alternative, low-cost adsorbents derived from natural, agricultural, and industrial waste sources (Crini *et al.*, 2018). It has also been known for its highly effective removal of contaminants from drinking water sources such as groundwater, rivers, lakes and reservoirs. Activated carbon remains the most commonly used industrial adsorbent due to its high surface area, porosity, and strong affinity for a wide variety of pollutants. It is highly effective in removing organic contaminants, colour, and odour from water. Key limitations, including high production costs, regeneration expenses, limited selectivity, and the necessity for frequent replacements, have driven interest in alternative materials. (Singh *et al.*, 2018). Other conventional materials include ion-exchange resins, silica gel, molecular sieves, and zeolites. These materials are favoured in industries for their high adsorption capacity and reproducibility, though their cost and energy-intensive production pose environmental and economic challenges (Singh *et al.*, 2018).

Non-conventional adsorbents

Unconventional adsorbents have been under investigation in the past three decades to create more affordable and efficient adsorbents for the removal of contaminants at trace levels (Akhtar *et al.*, 2025).

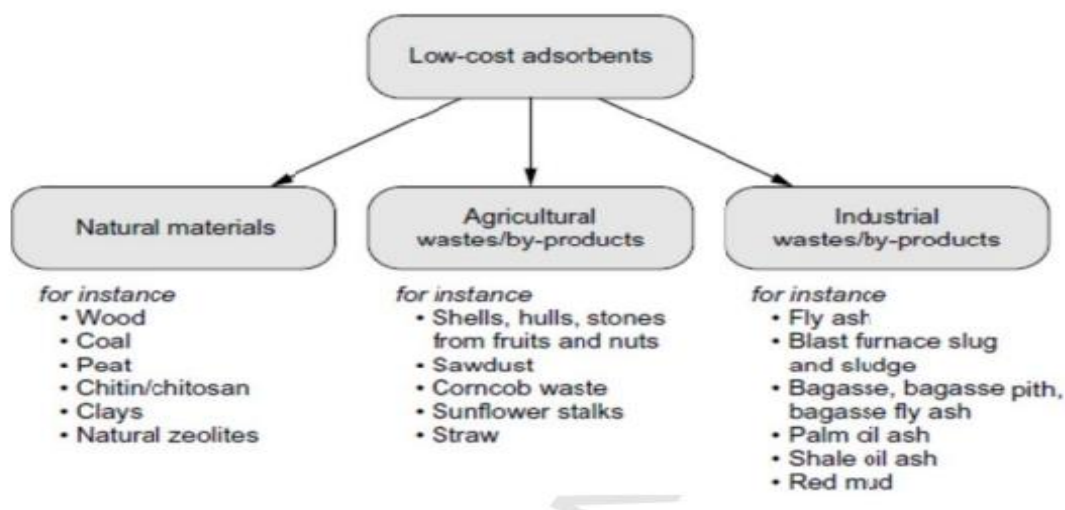


Figure 2.2: Schematic diagram of a low-cost adsorbent (Source: Worch, 2012)

2.1.2.3 Natural Adsorbents for Water Purification

Natural adsorbents are gaining increasing attention for water and wastewater treatment due to their abundance, environmental friendliness, low cost, and high efficiency in removing a wide range of pollutants. Singh *et al.* (2018) highlight that natural adsorbents include materials such as chitin/chitosan, clay minerals, zeolites, peat, and wood, which possess inherent adsorption capabilities due to their surface chemistry and structural features.

Chitin and Chitosan

Chitin, a biopolymer extracted from crustacean shells, and its derivative chitosan are among the most studied natural adsorbents. Chitosan, formed through the deacetylation of chitin, exhibits enhanced adsorption due to the presence of amino and hydroxyl functional groups. These groups facilitate interactions with heavy metals, dyes, and other pollutants through mechanisms such as ion exchange and chelation (Singh *et al.*, 2018). Chitosan has been reported to effectively remove various organic and inorganic contaminants, including dyes like acid blue nine and food yellow 3, under optimised conditions of pH and contact time. Chemically modified chitosan showed higher adsorption capacities (up to 375.94 mg/g for

food yellow 3) compared to its unmodified form, demonstrating the benefits of functional enhancement (Singh *et al.*, 2018).

Zeolites

Natural zeolites, especially clinoptilolite, are microporous aluminosilicates that have high cation exchange capacities and molecular sieving properties. Their crystalline structure and large surface area make them effective for adsorbing metal ions such as Zn^{2+} , Cu^{2+} , Pb^{2+} , Cd^{2+} , and Mn^{2+} . Several studies have shown that natural and modified zeolites achieved adsorption efficiencies ranging from 5.89 to 65.75 mg/g depending on the pollutant and conditions (Singh *et al.*, 2018). Additionally, modified zeolites, such as hexamethylenediamine-modified and surfactant-modified zeolites, demonstrated improved removal of dyes and organic compounds due to the introduction of functional groups or enhanced hydrophobic interactions.

Wood and Bark

Various types of wood and bark are employed as biosorbents due to their porous nature and chemical composition. Eucalyptus bark, for example, has been used to remove dyes and chromium (Cr^{6+}) from wastewater without pretreatment (Singh *et al.* (2018) report an adsorption capacity of 45 mg/g for Cr^{6+} using eucalyptus bark at pH 2. Similarly, papaya wood showed significant efficiency in removing Cu^{2+} , Cd^{2+} , and Zn^{2+} , achieving removal rates of 97.8%, 94.9%, and 66.8%, respectively, at pH 5.

2.1.2.4 Nature and Composition of Agricultural Waste Adsorbents

Agricultural waste materials such as rice husks, wheat straw, corn cobs, fruit peels, coconut shells, and nut husks are rich in lignocellulosic compounds, mainly cellulose, hemicellulose, and lignin. These biopolymers contain functional groups such as hydroxyl, carboxyl, and phenolic groups that promote metal ion binding through complexation, ion exchange, and electrostatic interactions (Karić *et al.*, 2022; Mo *et al.*, 2018). The physicochemical properties of these biosorbents, such as high surface area, porosity, and functional group availability, can be further improved through modification techniques including acid/base treatment, carbonisation, and functionalisation with sulphur or amine groups (Mo *et al.*, 2018). For example, sulphur-functionalised rice husk demonstrated a high adsorption capacity of up to 138.85 mg/g for cadmium under optimal conditions (Mo *et al.*, 2018).

Effectiveness and Applications

- i. **High Removal Efficiency:** Agricultural waste adsorbents can achieve high removal rates for dyes (up to 98% for Rhodamine B), heavy metals (e.g., chromium, cadmium), and phenols (Al-Gheecti *et al.*, 2021; Karic *et al.*, 2021)
- ii. **Versatility:** Effective across a wide pH range and for various wastewater types (domestic, industrial, agricultural) (Al-Gheecti *et al.*, 2021; Karic *et al.*, 2021)
- iii. **Sustainability:** Utilisation reduces environmental pollution, supports the circular economy, and offers a renewable, biodegradable alternative to synthetic adsorbents (Karic *et al.*, 2021; Dai *et al.*, 2018)

2.1.2.5 Biosorption

Biosorption refers to the passive uptake of contaminants by biological materials. Various studies have documented the efficiency of biosorbents in removing pollutants.

Biosorption specifically utilises biological materials like plant biomass, microbial biomass, and agricultural residues as adsorbents. These biosorbents possess functional groups such as hydroxyl (-OH), carboxyl (-COOH), amino (-NH₂), and phenolic (-C₆H₅OH) that can bind pollutants through mechanisms such as ion exchange, complexation, and physical adsorption (Ahmad *et al.*, 2021). Biosorption is advantageous because biosorbents are renewable, biodegradable, inexpensive, and abundant, making them especially attractive in low-resource settings (Haleem *et al.*, 2020). According to Ali *et al.* (2022), biosorption processes are not only environmentally benign but also require less energy and produce less sludge compared to conventional chemical treatments, reducing operational costs and environmental footprints.

2.1.3 Plant-Based Adsorbents

Plant-based adsorbents have gained significant attention for wastewater treatment due to their abundance, low cost, and eco-friendliness. These adsorbents typically derive from lignocellulosic biomass composed mainly of cellulose, hemicellulose, and lignin, which contribute to their structural integrity and provide adsorption sites (Gautam *et al.*, 2020).

Several agricultural wastes have been studied extensively:

- i. Banana peel powder has been demonstrated to remove heavy metals like lead (Pb) and cadmium (Cd) with adsorption efficiencies exceeding 90% under optimised conditions (Tetteh *et al.*, 2021).
- ii. Coconut husks and shells have been used to adsorb dyes and heavy metals effectively due to their high surface area and porosity (Gautam *et al.*, 2020).

- iii. Orange peels have also been shown to remove contaminants through ion exchange and complexation mechanisms (Verma *et al.*, 2019).

Chemical or physical modifications such as acid/base treatment, carbonisation, or impregnation with metals can enhance adsorption capacity by increasing surface area and functional group availability (Verma *et al.*, 2019). These materials are often utilised in batch or fixed-bed column systems and provide sustainable alternatives to expensive commercial adsorbents such as activated carbon (Haleem *et al.*, 2020).

2.1.4 *Picralima nitida* Stem Bark as an Adsorbent

Picralima nitida, belonging to the Apocynaceae family, is a medicinal plant native to West and Central Africa. Its stem bark has traditionally been used for treating ailments such as malaria, fever, and gastrointestinal diseases due to its rich content of bioactive compounds, including alkaloids, tannins, flavonoids, saponins, and phenolic compounds (Akinmoladun *et al.*, 2021; Olabemiwo *et al.*, 2016). Phytochemical analyses reveal that the stem bark contains lignocellulosic fibres that provide a porous matrix capable of adsorbing pollutants (Olabemiwo *et al.*, 2016). The functional groups present in these compounds (e.g., hydroxyl, carboxyl) can participate in the biosorption of heavy metals and organic pollutants from aqueous solutions (Akinyemi & Oloruntoba, 2023).

Though research on the use of *P. nitida* stem bark for wastewater treatment is limited, early studies indicate its potential as a low-cost and efficient biosorbent. Akinyemi and Oloruntoba (2023) highlighted its high surface area and porosity compared to other plant biomass adsorbents. Additionally, its availability as an agricultural waste in many parts of Nigeria and West Africa makes it a sustainable resource for wastewater remediation. Given these characteristics, further investigation into the adsorption efficiency, optimal operational

conditions, and economic feasibility of *Picralima nitida* stem bark residue is warranted to establish its viability as an alternative adsorbent in domestic wastewater treatment.

2.2 Theoretical Framework

The theoretical framework underpinning this study is based on adsorption theory and biosorption mechanisms, which explain how contaminants in domestic wastewater interact with adsorbent materials such as *Picralima nitida* stem bark residue.

2.2.1 Adsorption Theory

Adsorption is a surface phenomenon where molecules from a fluid phase (liquid or gas) accumulate on the surface of a solid adsorbent. The process is governed by physicochemical interactions such as van der Waals forces, electrostatic attraction, ion exchange, and chemical bonding (Foo & Hameed, 2010). Adsorption can be classified into two types:

- i. **Physisorption** (physical adsorption) involves weak van der Waals forces and reversible binding, usually occurring at lower temperatures.
- ii. **Chemisorption** (chemical adsorption) involves stronger covalent or ionic bonding and often irreversible adsorption (Foo & Hameed, 2010).

The efficiency of adsorption depends on adsorbent characteristics (surface area, pore size, functional groups), adsorbate properties (molecular size, polarity), and operational parameters (pH, temperature, contact time, dosage).

2.2.2 Biosorption Mechanism

Bio-sorption is an adsorptive method for potentially removing pollutants, metal ions, and organic dyes present in aqueous solutions. The name suggests that bio-sorbents are derived from biological sources or biological means. These bio-sorbents tend to attract or bind to pollutants due to particular functional groups present on their surface (Chojnacka, 2010)

Biosorption refers to the adsorption of pollutants by biological materials such as plant biomass, microorganisms, or agricultural residues. The process is driven by various mechanisms, including:

- i. **Ion Exchange:** Exchange of ions between the pollutant and charged functional groups on the biosorbent surface (Volesky, 2003, okoro et al, 2025).
- ii. **Complexation/Chelation:** Formation of stable complexes between metal ions and functional groups like carboxyl, hydroxyl, and amino groups (Wang & Chen, 2009).
- iii. **Physical Adsorption:** Adsorption driven by electrostatic attraction and Van der Waals forces.
- iv. **Micro-precipitation:** Formation of insoluble compounds on the biosorbent surface (Volesky, 2003, okoro et al, 2025).

Plant-based biosorbents contain lignocellulosic components rich in these functional groups, which are responsible for binding contaminants in wastewater (Gupta & Suhas, 2009).

2.3 Adsorption Isotherms and Kinetics

Understanding adsorption behaviour requires modelling equilibrium and kinetics:

2.3.1 Adsorption Isotherms

The Langmuir and the Freundlich isotherms are commonly used to describe adsorption behaviour.

The Langmuir isotherm suggests that adsorption occurs at specific homogeneous sites on the adsorbent surface, forming a monolayer. This model provides information on the maximum adsorption capacity (Langmuir, 1918).

The Freundlich isotherm accounts for heterogeneous surface energies and suggests multilayer adsorption, which is more realistic in many natural systems (Freundlich, 1906).

When applied to *P. nitida*, Akinyemi and Oloruntoba (2023) found that the Langmuir model provided a better fit, indicating monolayer adsorption behaviour.

Adsorption isotherms describe the equilibrium relationship between the amount of adsorbate on the adsorbent surface and the concentration of adsorbate in solution at constant temperature (Foo & Hameed, 2016). Several isotherm models are commonly used to analyse heavy metal adsorption data:

Langmuir Isotherm: Based on the assumption of monolayer adsorption on a homogeneous surface with a finite number of adsorption sites. The Langmuir equation is:

$$q_e = \frac{(q_{max}bC_e)}{(1 + bC_e)} \quad \text{Eq. (2.1)}$$

Where q_e is the equilibrium adsorption capacity, q_{max} is the maximum adsorption capacity, b is the Langmuir constant, and C_e is the equilibrium concentration (Al-Ghouti & Da'ana, 2020).

Freundlich Isotherm: Describes adsorption on heterogeneous surfaces with multiple adsorption sites of varying energies. The Freundlich equation is:

$$q_e = KfC_e^{\frac{1}{n}} \quad \text{Eq. (2.2)}$$

2.3.2 Adsorption Kinetics

Adsorption kinetics is very crucial for determining the rate and mechanism of pollutant removal. The pseudo-second-order model, developed by Ho and McKay (1999), assumes that chemisorption is the rate-limiting step. This model fits the experimental data from *P. nitida* well, reinforcing the idea that pollutant removal involves strong interactions, likely chemical bonding between the adsorbent and the contaminants.

Kinetic models describe the rate of adsorption and help identify the rate-controlling step in the adsorption process (Simonin, 2016). Standard kinetic models include:

Pseudo-first-order model: Assumes that the rate of adsorption is proportional to the number of unoccupied adsorption sites. The equation is given as

$$\ln(q_e - q_t) = \ln q_e - k_1 t \quad \text{Eq. (2.3)}$$

where q_t is the adsorption capacity at time t , and k_1 is the pseudo-first-order rate constant (Plazinski *et al.*, 2015).

Pseudo-second-order model: Assumes that the adsorption rate is controlled by chemical adsorption involving the sharing or exchange of electrons. The equation is given as:

$$\frac{t}{q} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \quad \text{Eq. (2.4)}$$

Where K_2 is the pseudo-second-order rate constant (Ho & McKay, 2016).

Intraparticle diffusion model: Describes adsorption processes controlled by intraparticle diffusion. The equation is:

$$q_t = k_{ip}t^{\frac{1}{2}} + C \quad \text{Eq. (2.5)}$$

Where k_{ip} is the intraparticle diffusion rate constant and C is a constant related to boundary layer thickness (Wu *et al.*, 2018).

2.4 General Information on the Study Plant (*Picralima nitida*)

Picralima nitida is a species of the genus *Picralima*. It belongs to the hunterieae tribe of Apocynaceae family and is commonly called Osu (Edo), Osi-Igwe in Igbo and Abere in Yoruba. In other parts of West Africa, the plant is called Gbe-Fondagne in the Benin Republic, Adangme in Ghana, Abureebissi in Cote d'Ivoire and Susubalunyi in Sierra Leon (Kpodar *et al.*, 2015). The flowers are white (about 3 cm long) and they have ovoid fruits which become yellowish when mature. The leaves are broad (3-10 cm) and oblong (6-20 cm long) with tough, tiny lateral. *P. nitida* is extensively distributed across West-Central Africa. The tree of *P. nitida* is an understorey, reaching up to 4-35 m in height. Its trunk is about 5-60 m in diameter; cylindrical in shape, and the wood is a pale yellow and hard (Okonta, 2007).

P. nitida has diverse applications in West African traditional medicine. Various parts of the plant, such as the leaves, seeds, stem, bark and roots, are used by herbalists for the treatment of fever, hypertension, jaundice, gastrointestinal disorders and malaria (Falodun *et al.*, 2006). Preparations from different parts of the plant are employed as crude drugs or crude herbal extracts as remedies for various kinds of human diseases. The seeds are widely used in West Africa, especially in Nigeria, Cote d'Ivoire and Ghana as antipyretic, aphrodisiac agents, and for the treatment of malaria, pneumonia and other chest conditions (Falodun *et al.*, 2006).

Herbalists have also made claims for the efficacy of the coconut water extract of *P. nitida* seeds in the treatment of many diseases, including diabetes (Adegoke & Oloyede, 2013). The presence of saponins, alkaloids, glycosides, steroids and tannins in the seeds of *P. nitida* has been reported (Sunmonu *et al.*, 2014). It has been shown that seeds of the plant are rich in amino acids, vitamins A and E, as well as in mineral elements such as zinc, iron and manganese (Nwaogu, 2016). Investigations into the biological activity of the seed extracts also revealed their antimicrobial, larvicidal and hyperproteinaemic potential (Nwabor *et al.*, 2014; Adegoke & Oloyede, 2013).

2.4.1 Application to *Picralima nitida* Stem Bark Residue

The stem bark of *Picralima nitida* is composed primarily of cellulose, hemicellulose, and lignin, all of which provide functional groups such as hydroxyl (-OH), carboxyl (-COOH), and phenolic (-C₆H₅OH) groups that can interact with pollutants (Olabemiwo *et al.*, 2016). These chemical groups facilitate biosorption through ion exchange and complexation mechanisms, consistent with the biosorption theories discussed.

Moreover, the porous structure of the stem bark enhances its surface area and availability of adsorption sites, contributing to its potential as an effective adsorbent for domestic wastewater contaminants (Akinyemi & Oloruntoba, 2023). *Picralima nitida* stem bark, often discarded as waste, contains bioactive compounds (e.g., alkaloids and polyphenols) that may enhance metal ion binding (Adotey *et al.*, 2012). While limited studies exist on its use in wastewater treatment, analogous plant residues, such as neem bark and rubber wood ash, have shown promising adsorption capacities (Selvakumari *et al.*, 2002). The use of *Picralima nitida* residues could reduce costs due to its abundance and inexpensiveness; it also promotes sustainability by aligning with Nigeria's Solid Waste Management (SWM) goals (Shah *et al.*, 2012) and enhances local solutions by utilising indigenous resources, reducing

reliance on imported activated carbon. Evaluating adsorption isotherms and kinetics will provide insights into the efficiency and mechanism of adsorption by this material under various operating conditions.

2.4.2. Phytochemical screening of *Picralima nitida*

Phytochemical screening of *P. nitida* has revealed the presence of alkaloids, tannins, saponins, flavonoids, terpenoids, steroids and glycosides in the plant (Olumese, Aihie, & Oriakhi, 2023). Phytochemical investigation has led to the isolation of several alkaloids, which are major compounds.

According to Fulgence *et al* (2014), Alaebo, P. O. *et al.* (2025), the phytochemical screening of *Picralima nitida* seed extracts has revealed the presence of terpenes, alkaloids, and polysterols in methanol, chloroform solutions, and aqueous solutions. Unlike chloroform and methanol solutions, the aqueous solution revealed the presence of saponins. The phytochemical screening was done using classic methods (Olumese *et al.*, 2023). Chemical compounds tested in all the extracts of *Picralima nitida* seeds were: saponosides, alkaloids, flavonoids, tannins, terpenes, sterols, phenols and quinones. The tests were based on the visual observation of colour change or formation of a precipitate after specific reactions.

Nwankwo *et al.* (2019) and Ogbeide (2025) evaluated the phytochemical composition of the seed of *Picralima nitida*. They found that qualitative and quantitative phytochemical screening of the seed sample revealed flavonoids, saponins, glycosides, steroids, alkaloids, soluble carbohydrates, reducing sugars, tannins, terpenoids, phenols and hydrogen cyanides. Thus, the seed extract of *Picralima nitida* has rich content of the determined phytochemicals such as alkaloids, leucoanthocyanins, flavonoids, saponins, tannins, anthraquinones, polyphenols, coumarins, sterols and/or triterpenes, anthocyanins, cardiac glycosides, reducing sugars, glycosides, anthranoids and steroids was carried out. Those constituents were

identified by characteristic colour changes using standard procedures (Onwuegbuchulam, 2024). Each test was qualitatively expressed as negative (–) or positive (+), and the intensity of the characteristic colour was expressed as (++) or (+++). The phytochemical analysis revealed the presence of alkaloids, flavonoids, saponins, polyphenols, cardiac glycosides and glycosides. Alkaloids and polyphenols were the major compounds.

2.4.3 Regeneration and Reusability

Enhancing the regeneration efficiency of biomass adsorbents will be crucial for sustainable use. It will become more economically feasible to reuse adsorbents over several cycles with the development of inexpensive, non-toxic desorption agents and thermal/chemical regeneration processes. (Karić *et al.*, 2022).

2.4.4 Future Challenges

Despite encouraging advancements, several issues need to be resolved to guarantee the safe, sustainable, and helpful utilisation of adsorbents derived from agricultural waste in actual wastewater treatment applications:

2.4.4.1 Heterogeneity and Variability of Feedstock

Agricultural waste varies significantly in composition due to differences in crop type, season, and geographic source. This inconsistency can affect adsorption behaviour and reproducibility, necessitating standardisation or pre-treatment protocols (Karić *et al.*, 2022)

2.4.4.2 Cost-Benefit and Techno-Economic Analysis

Although materials are low-cost, the collection, processing, modification, and disposal of used adsorbents incur operational costs. Comprehensive life-cycle and cost-benefit analyses are needed to validate commercial viability.

2.5 Empirical Review

Experimental studies conducted in recent years have significantly contributed to the validation of natural and plant-based materials as efficient biosorbents in wastewater treatment. These findings go beyond theoretical predictions, providing data on actual adsorption capacities, optimal operating conditions, mechanisms of contaminant removal, and the economic feasibility of scaling up such methods. The empirical results establish a scientific foundation for the consideration of *Picralima nitida* stem bark as a promising biosorbent in domestic wastewater remediation.

2.5.1 General Performance of Lignocellulosic Biosorbents

Lignocellulosic biomass is widely acknowledged for its biosorptive potential due to its inherent physicochemical properties. These materials, often agricultural residues like bark, husks, or peels, are primarily made of cellulose, hemicellulose, and lignin, which house polar functional groups such as hydroxyl (-OH), carboxyl (-COOH), phenolic (-C₆H₅OH), and sometimes amino (-NH₂) groups. These groups form coordination complexes with metal ions or other polar pollutants, enabling their removal from aqueous solutions (Ahmad *et al.*, 2021).

For instance, Deepa *et al.* (2020) demonstrated that mango bark powder removed over 80% of lead and cadmium ions from simulated wastewater. This high efficiency was achieved under optimised conditions, specifically at a pH of 5.5 and a contact time of 60 minutes. These results suggest that biosorbents like mango bark can compete with commercial adsorbents when properly conditioned.

Jahan *et al.* (2022) conducted experiments using banana peel powder for the adsorption of chromium (Cr⁶⁺) and reported a removal efficiency of 87%. Adsorption performance improved as pH moved toward slightly acidic values and when a higher dosage was applied.

The researchers also observed that metal ion removal increased with time before reaching equilibrium, underscoring the need to determine optimal contact durations.

Muhammad *et al.* (2021) tested neem bark powder for its ability to adsorb dye molecules from textile effluent. The study achieved up to 85% dye removal under ambient conditions. This performance is noteworthy because dyes are typically larger organic molecules than metal ions and require adsorbents with both high surface area and strong functional group interactions.

These examples illustrate how various plant-based biosorbents perform under laboratory conditions and highlight the critical influence of surface chemistry, structure, and operational conditions on removal efficiency.

2.5.2 Application of *Picralima nitida* in Wastewater Treatment

Picralima nitida is traditionally recognised for its medicinal value; however, its bark also possesses the necessary chemical composition for pollutant adsorption. Recent experimental research by Akinyemi and Oloruntoba (2023) assessed the suitability of the powdered stem bark for treating wastewater contaminated with heavy metals. Their study showed that the material achieved over 85% removal of lead and cadmium under the following conditions:

- I. pH 5.5
- II. Contact time of 90 minutes
- III. Adsorbent dosage of 3 g/Lc

The results confirm that *P. nitida* performs optimally in acidic conditions similar to other lignocellulosic biosorbents. The slightly acidic environment enhances the electrostatic attraction between the negatively charged adsorbent sites and the positively charged metal ions.

Chemical and physical analyses further revealed the mechanism behind its effectiveness. Phytochemical screening (Olabemiwo *et al.*, 2016) confirmed the presence of alkaloids, tannins, saponins, and phenolic compounds, substances known to form strong complexes with pollutants. Scanning Electron Microscopy (SEM) images showed that the bark powder had a rough and porous surface, which increases the active surface area. Additionally, Fourier Transform Infrared Spectroscopy (FTIR) detected hydroxyl and carboxyl groups, confirming the material's capacity for chemical interaction with contaminants (Akinyemi & Oloruntoba, 2023).

These experimental insights suggest that *P. nitida* is not just a theoretical alternative but a viable, field-relevant biosorbent that warrants further research and potential pilot-scale application.

2.5.3 Factors Influencing Adsorption Efficiency

Several factors influence adsorption efficiency. These are discussed below

i. pH of the Solution

The pH level plays a significant role in adsorption by influencing the surface charge of the adsorbent and the degree of ionisation of the pollutants. At low pH values (acidic), excess hydrogen ions compete with metal cations for binding sites, reducing adsorption. However, at slightly acidic pH levels (around 5.5), ion exchange and electrostatic interaction are enhanced, resulting in optimal removal (Adebowale *et al.*, 2018). For *P. nitida*, this pH range has been empirically confirmed as optimal (Akinyemi & Oloruntoba, 2023).

ii. Contact Time

The adsorption process typically follows a two-stage kinetic pattern: an initial rapid phase followed by a slower one. The rapid uptake occurs due to the abundance of available active sites, while the slower phase reflects gradual saturation. *P. nitida* achieved equilibrium

adsorption within 90 minutes, indicating that this contact duration is sufficient for most interactions to complete (Ahmad *et al.*, 2021).

iii. **Adsorbent Dosage**

Increasing the quantity of biosorbent enhances the availability of binding sites, leading to improved adsorption rates. However, beyond a certain threshold, particle aggregation can reduce the effective surface area and hinder performance (Haleem *et al.*, 2020). In the case of *P. nitida*, 3 g/L has been identified as the optimal dosage.

iv. **Temperature**

Temperature affects the kinetic energy of molecules. In biosorption, moderate increases in temperature often improve performance, indicating endothermic adsorption. Higher temperatures may enhance the diffusion of pollutant molecules into the adsorbent pores (Tetteh *et al.*, 2021).

2.5.4 Cost-Effectiveness and Environmental Suitability

From an economic standpoint, using *Picralima nitida* offers significant advantages. Activated carbon, the conventional adsorbent, is costly and requires energy-intensive production. In contrast, *P. nitida* bark is a natural by-product, especially in West African countries, where it is commonly harvested for medicinal use and then discarded.

Ali *et al.* (2022) observed that agricultural biosorbents can reduce treatment costs by as much as 60%, mainly due to local availability and minimal processing requirements. This makes *P. nitida* particularly suitable for decentralised wastewater treatment in rural or low-income communities.

In environmental terms, *P. nitida* is biodegradable and produces minimal secondary waste, unlike many synthetic materials. The reuse of agricultural waste for water treatment aligns

with circular economy principles and offers a sustainable solution to both solid and liquid waste management (Haleem *et al.*, 2020).

2.4 Research Gap

Picralima nitida stem-bark powder has been investigated as a low-cost biosorbent. Despite its known richness in bioactive compounds and phenolic groups, it has not received the same level of empirical attention in environmental applications.

Existing studies tend to concentrate on the phytochemistry and pharmacological properties of *P. nitida* (Akinmoladun *et al.*, 2021), overlooking its potential in adsorption. Only a few recent works, such as Akinyemi and Oloruntoba (2023), have attempted to test its biosorption capacity. However, these are often limited in scope, focusing on a synthetic single pollutant (like dye) or using ideal laboratory conditions that do not reflect complex wastewater scenarios. There is no robust data on the adsorption capacity of *Picralima nitida* across different pollutants, such as surfactants, oil, nutrients and organic loads. To close this gap, the current study uses real domestic wastewater (sullage) collected from residential sources to test the pollutant removal efficiency of *P. nitida* bark under non-stimulated variable conditions.

CHAPTER THREE

RESEARCH METHODOLOGY

3.1 Research Design

This study employs an experimental research approach involving systematic, controlled laboratory experiments to assess the adsorption capacity of *Picralima nitida* stem bark residue as an adsorbent for domestic wastewater treatment. This approach facilitates evaluating the removal efficiency of the adsorbent under different operational conditions, optimising key parameters such as pH, contact time, dosage, and temperature, understanding adsorption behaviour through isotherm and kinetic analyses, and conducting a cost-effectiveness comparison with traditional adsorbents. This methodology is suitable for environmental engineering research, where controlled batch tests and material characterisation are essential for determining the feasibility of new, low-cost biosorbents (Febrianto *et al.*, 2009).

3.2 Sampling Techniques

3.2.1 Sample Collection

Fresh stem bark of *Picralima nitida* was sourced from local forests or botanical gardens within Edo State, Nigeria. The collection was in accordance with sustainable harvesting protocols (i.e., minimal damage to the tree, non-endangered status, and obtaining local permission).

3.2.2 Adsorbent Preparation

- i. The bark was thoroughly washed with distilled water to remove dirt, dust and surface debris.
- ii. This was air-dried under shade until essentially free of surface moisture.

- iii. It was then oven-dried at 105 °C until a constant weight was achieved (i.e., successive weighings differ by < 0.5 %).
- iv. The dried bark was ground to a powder and sieved to a uniform particle size, for instance 250–500 µm (or –44 + 52 BS mesh) to ensure consistent adsorbent size.
- v. Pre-treatment: the pulverised powder was then washed thoroughly with 0.5 M NaOH to remove lignin-based colour materials (following the method of Naiya *et al.* 2008).
- vi. This was then soaked in 0.5 M phosphoric acid (H₃PO₄) for 24 h (El-Ashtoukhy *et al.*, 2007 method).
- vii. This was then washed with deionised water until the filtrate was neutral (pH ~7).
- viii. This was oven-dried again at 105 °C for 6 hours (until constant weight was achieved) and then cooled in a desiccator to room temperature.
- ix. Finally, it was ground, sieved to the target mesh (–44 + 52 BS mesh), packed and sealed in labelled airtight containers until use.

This systematic preparation ensured reproducibility and minimised extraneous variable effects, including varying particle size, residual moisture, and untreated lignin

3.3 Materials

Reagents: All reagents were analytical grade. These were sourced from Merck, Mumbai, India. These include

- i. Sodium hydroxide (NaOH) 0.5 M
- ii. Phosphoric acid (H₃PO₄) 0.5 M
- iii. Standard buffer solutions for pH calibration
- iv. Distilled/deionised water for all preparations and dilutions

Equipment: The equipment used includes

- i. Nitrogen gas (N₂) for BET (Brunauer–Emmett–Teller) surface area analysis

- ii. pH metre: EUTECH 5500 (or equivalent) with FET solid-state electrode
- iii. Specific gravity (pycnometer) bottle for bulk density determination
- iv. Particle size distribution analyser (Malvern Instruments model 117.08)
- v. Scanning Electron Microscope (SEM) (Hitachi S415A)
- vi. Fourier Transform Infrared Spectrometer (FTIR) (Perkin Elmer FTIR RX1)

Characterisation of the adsorbent includes surface area, pore volume, bulk density, particle size distribution, morphology (SEM), and functional groups (FTIR). The use of this combined suite aligns with best practices for biosorbent development (Gupta *et al.*, 2015).

3.4 Experimental Procedure

3.4.1 Characterisation of Adsorbent

The prepared *Picralima nitida* stem bark residue was characterised to determine its physical and chemical properties, which include:

i. Surface area and pore size (BET analysis)

The degassed adsorbent samples were analysed at 77 K under N₂ adsorption–desorption conditions. The specific surface area (S_{BET}) was calculated from the linear region of the adsorption isotherm using the BET equation. Pore size distribution and total pore volume were derived from the desorption branch via the Barrett–Joyner–Halenda (BJH) method. This provided insights into the adsorption capacity potential (Brunauer *et al.*, 1938).

ii. Functional groups by Fourier Transform Infrared Spectroscopy (FTIR)

Functional groups present in the samples were identified using FTIR spectroscopy (Dai *et al.*, 2023). Dried adsorbent (~1 mg) was mixed with KBr (~100 mg) and pressed into a pellet. Spectra were collected in the range 4000–400 cm⁻¹ at room temperature. Interpretation of peaks corresponding to –OH, –NH, –COOH and C=O helped identify the functional groups potentially involved in adsorption (Ahmaruzzaman, 2008).

iii. **Morphology through Scanning Electron Microscopy (SEM)**

The surface morphology of the samples was examined using Scanning Electron Microscopy (SEM) (Girao *et al.*, 2017). Samples were sputter-coated with a thin gold layer to enhance conductivity and imaged at magnifications of $\times 500$, $\times 2000$, and $\times 5000$ to observe surface texture, particle shape, and distribution. Observations included particle shape, surface texture, porosity and possible aggregation patterns (Girão *et al.*, 2017). SEM provided insights into the physical structure relevant to adsorption efficiency.

iv. **Particle Size Analysis by Dynamic Light Scattering (DLS)**

Particle size distribution was determined using Dynamic Light Scattering (DLS), which measures fluctuations in light scattering due to Brownian motion of particles in suspension. Samples were dispersed in deionised water, sonicated, and analysed at 25 °C. The hydrodynamic diameter was calculated based on the Stokes–Einstein equation (Stetefeld *et al.*, 2016)

3.4.2 Batch Adsorption Experiments

Batch adsorption tests were performed to evaluate removal efficiencies under varying conditions, using domestic wastewater as the adsorbate matrix. The procedure is as follows:

- i. A Domestic wastewater (Sullage) was collected from the kitchen (characterise initial concentrations of relevant pollutants: e.g., heavy metals, COD, TDS, EC, etc.).
- ii. pH solution was adjusted using dilute HCl or NaOH (range ~3 to ~9).
- iii. Adsorbent was added at different dosage levels (for example, 0.2 g to 1.0 g per 20 mL wastewater); each dosage was run in triplicate ($n = 3$).
- iv. Contact times: test at intervals, for example 15, 30, 60, 90 minutes; agitation at constant speed (e.g., 150 rpm) at selected temperature (e.g., 28 °C, 38 °C, 48 °C).

- v. Temperature variation was done using a thermostatically controlled water bath/incubator.
- vi. After the contact time, samples were filtered (0.45 μm membrane) and residual pollutant concentrations measured using, e.g., Atomic Absorption Spectroscopy (AAS) for heavy metals or UV-Vis spectrophotometry for other contaminants.
- vii. All experiments were performed using blank controls (without adsorbent) for comparison.

3.5 Data Analysis

The contaminant removal efficiency was calculated using (Dowlatshahi et al, 2014)

$$\text{Removal Efficiency (\%)} = \frac{C_0 - C_E}{C_0} \times 100 \quad \text{Eq. (3.1)}$$

Where, C_0 = initial pollutant concentration (mg L^{-1});

C_e = equilibrium pollutant concentration (mg L^{-1}).

The Adsorption capacity was calculated by using the mass balance equation for the adsorbent.

Adsorption capacity (q_e , mg g^{-1}) will be computed by

$$q = \frac{(C_i - C_e)V}{W} \times 100 \quad \text{Eq. (3.2)}$$

Where:

C_i = initial concentration (mg L^{-1});

C_e = equilibrium concentration (mg L^{-1});

V = volume of solution (L);

W = mass of adsorbent used (g).

3.6 Adsorption Isotherms Studies

Adsorption Isotherm studies outline the equilibrium relationship between a solute's concentration in solution and the amount adsorbed onto a solid surface at a constant temperature. They offer insights into the adsorption mechanism, the surface characteristics of the adsorbent and how it interacts with the adsorbate (Foo & Hameed, 2010)

In order to successfully represent the equilibrium adsorption behaviour, it is important to have a satisfactory description of the equation of state between the two phases composing the adsorption system. In the present work, the experimentally obtained equilibrium data were analysed using the Langmuir and the Freundlich isotherm models

3.6.1 Langmuir Isotherm

The Langmuir isotherm assumes monolayer adsorption onto a surface with a finite number of identical sites. In contrast, the Freundlich isotherm is an empirical equation applicable to multilayer adsorption on heterogeneous surfaces (Foo & Hameed, 2010). Recent studies continue to validate the applicability of these models; for instance, modifications to the Langmuir model are used to account for more complex interactions on nanocomposite surfaces (Aigbe et al., 2021). Furthermore, the integration of error functions is now standard practice to determine the best-fitting isotherm model, moving beyond reliance on the correlation coefficient alone (Ayawei *et al.*, 2017).

The adsorption data were further analysed using the Langmuir equation. This isotherm applies when the extent of adsorbate coverage is limited to a single molecular layer. This isotherm equation gives the fractional coverage q in the form of

$$Q = \frac{q_{max}KaC}{1+KaC} \quad \text{Eq. (.3.3)}$$

Where q_{max} is maximum adsorbent uptake

K_a is the Langmuir constant related to the energy of adsorption, which quantitatively reflects the affinity between the adsorbent and the adsorbate. The Langmuir parameters were obtained by fitting the experimental data to a linearised equation derived from the following equation;

$$\frac{1}{q} = \frac{1}{qm} + \frac{1}{Ka qm C} \quad \text{Eq.. (3.4)}$$

A plot of $\frac{1}{q}$ versus $\frac{1}{C}$ resulted in a straight line graph of slope $\frac{1}{Ka qm}$ and an intercept of $\frac{1}{qm}$

The Langmuir model assumes monolayer adsorption on a homogeneous surface with finite identical sites, represented by the equation:

$$\frac{C_e}{q_e} = \frac{1}{Q_0 b} + \frac{C_e}{Q_0} \quad \text{Eq.. (3.5)}$$

Where:

C_e = equilibrium concentration of Fe (mg/L)

q_e = amount adsorbed at equilibrium (mg/g)

Q_0 = maximum adsorption capacity (mg/g)

b = Langmuir constant related to adsorption energy (L/mg)

3.6.2 Freundlich Isotherm

For adsorption from a solution, the Freundlich isotherm is expressed by:

$$q_e = K_F C_e^{1/n} \quad \text{Eq. (3.6)}$$

The linearised form of the Freundlich equation is given as:

$$\log q_e = \log K_F + (1/n) \log C_e \quad \text{Eq. (3.7)}$$

Where q_e is the amount adsorbed at equilibrium (mg/g), C_e is the equilibrium concentration of metal ions in solution (mg/L), K_F is the Freundlich constant $((\text{mg/g})(\text{L/mg})^{1/n})$, which indicates the relative adsorption capacity of the adsorbent, and n is the heterogeneity factor representing the deviation from linearity of adsorption and is related to the adsorption

intensity (Ayawei et al., 2017). The value of $1/n$ provides insight into the adsorption favorability and the surface heterogeneity. A value of $1/n < 1$ indicates a normal Langmuir-type isotherm, while $1/n > 1$ suggests cooperative adsorption (Foo & Hameed, 2010). The Freundlich constants K_F and n are obtained from the intercept and slope, respectively, of the linear plot of $\log q_e$ versus $\log C_e$. The Freundlich model is widely applicable to adsorption processes on heterogeneous surfaces, such as those found in many bio-sorbents and nanocomposites (Aigbe et al., 2021; Liu & Shen, 2022).

Experimental equilibrium data were fitted to the two widely used Adsorption Isotherm models:

- i. Langmuir isotherm model (assuming monolayer adsorption on a homogeneous surface);
- ii. Freundlich isotherm model (an empirical model for heterogeneous surfaces) (Febrianto et al., 2009).
- iii. Parameter estimation (e.g., $q_{\max}$, b for Langmuir; K_F , n for Freundlich) and goodness-of-fit (R^2) were reported.

Again, parameters (k_1 , k_2) and R^2 values were reported (Febrianto et al., 2009).

Regression analysis was applied to assess the influence of variables (pH, dosage, contact time, temperature) on removal efficiency.

3.7 Cost-Effectiveness Analysis

An economic evaluation was conducted comparing the treated adsorbent (*Picralima nitida* stem bark residue) with a conventional adsorbent such as activated carbon. The cost analysis will include:

- i. Raw-material procurement (collection/transportation) and adsorbent preparation costs (washing, drying, grinding, chemical pre-treatment).

- ii. Operation costs: energy (oven, agitator), labour, reagent consumption.
- iii. Adsorbent regeneration and disposal costs.

The cost per unit volume of wastewater treated (₹ per m³ or USD per m³) was estimated based on realistic assumptions. Sensitivity analysis was included to show how cost changes with adsorbent reuse cycles, disposal cost, and energy tariff.

CHAPTER FOUR

RESULTS AND DISCUSSION

This chapter presents and discusses the experimental results obtained from investigating *Picralima nitida* stem bark residue as a potential low-cost adsorbent for treating domestic wastewater. The data analysis follows the sequence of the study objectives: characterisation of the adsorbent, evaluation of the influence of process variables on adsorption efficiency, and assessment of cost-effectiveness compared to conventional adsorbents. The analyses were conducted according to standard adsorption study protocols (Febrianto *et al.*, 2009; Ahmaruzzaman, 2008), focusing on key water quality parameters such as iron (Fe), zinc (Zn), copper (Cu), total dissolved solids (TDS), electrical conductivity (EC), and chemical oxygen demand (COD). Each parameter indicates the physicochemical quality of domestic wastewater and the effectiveness of the *P. nitida* bark residue in pollutant removal. The results are presented in tables and graphs, followed by interpretation and comparison with findings from similar biosorbent studies. Statistical trends and theoretical models (Langmuir, Freundlich, and kinetic models) are then used to explain the mechanisms of adsorption and the efficiency of the material under different operational conditions.

4.1 Characterisation of the Adsorbent

4.1.1 Functional Groups of the Adsorbent from *P. nitida* Stem Bark Residue

The FTIR spectrum of the adsorbent is shown in Figure 4.1

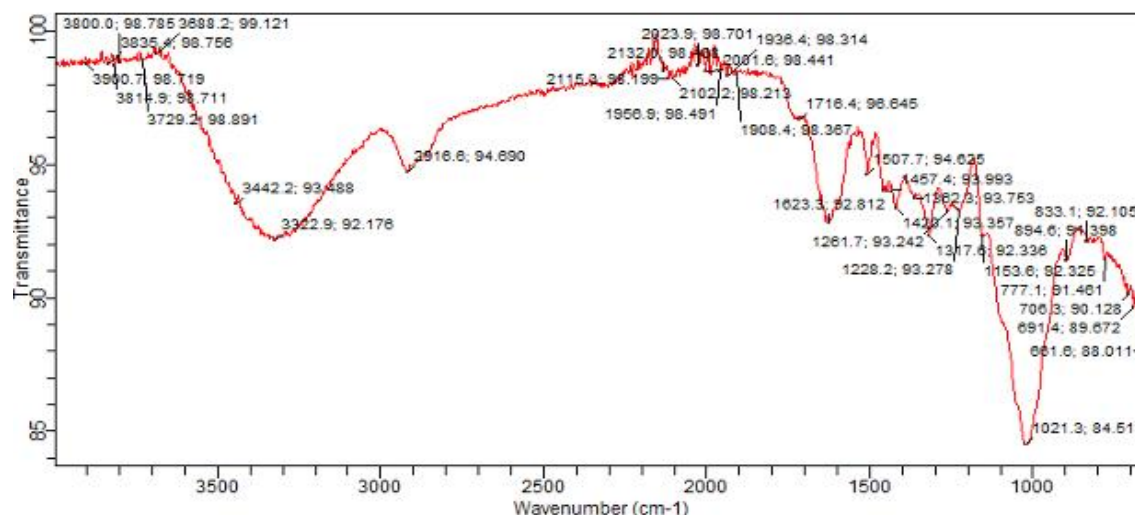


Figure 4.1: FTIR of Adsorbent from *P. nitida* Stem Bark residue

The FTIR spectrum of *Picralima nitida* stem bark adsorbent reveals various absorption bands indicating the presence of multiple functional groups responsible for adsorption. A broad peak observed around 3442 cm^{-1} corresponds to O–H stretching vibrations of hydroxyl groups associated with alcohols and phenols. Peaks at 2922 cm^{-1} and 2853 cm^{-1} represent C–H stretching of aliphatic $-\text{CH}_2$ and $-\text{CH}_3$ groups. The band at 2361 cm^{-1} is attributed to CO_2 asymmetric stretching, while the peak at 2132 cm^{-1} may be due to $\text{C}\equiv\text{C}$ stretching in alkynes.

Distinct sharp peaks within the range 1745–1630 cm^{-1} are assigned to C=O stretching vibrations of carboxylic acids, esters, or aldehydes, as well as C=C stretching of aromatic rings. Bands between 1510–1457 cm^{-1} indicate aromatic skeletal vibrations, while strong absorptions between 1228–1030 cm^{-1} correspond to C–O stretching in alcohols, phenols, carboxylic acids, ethers, or silicates. Peaks near 777–691 cm^{-1} are attributed to C–H bending in aromatic structures or skeletal vibrations of lignocellulosic material.

These functional groups, hydroxyl, carbonyl, carboxyl, aliphatic, aromatic, and ether/phenolic moieties, constitute the active binding sites that facilitate the adsorption of heavy metals and organic pollutants. Their presence confirms that *P. nitida* stem bark has a lignocellulosic nature, which is advantageous for metal ion binding and complexation.

This observation aligns with previous findings that lignocellulosic materials contain abundant hydroxyl, carbonyl, and phenolic groups, which facilitate metal adsorption through ion exchange and complex formation (Singh *et al.*, 2018; Mo *et al.*, 2018). Similar FTIR signals have been observed in sawdust, fruit peels, and other agro-waste biosorbents, where these functional groups improve the adsorption of Fe^{3+} , Zn^{2+} , and Cu^{2+} ions from water (Rafatullah *et al.*, 2009; Shahzadi *et al.*, 2023).

Overall, the FTIR profile of *Picralima nitida* stem bark adsorbent confirms that plant-based biosorbents are effective in adsorption, thanks to the combined influence of surface porosity and various surface moieties, as supported by literature (Igwegbe *et al.*, 2021; Aghedo & Ogbeide, 2022).

4.1.2 Morphological Characteristics of the Adsorbent from *P. nitida* Stem Bark Residue

The morphological characteristics of the adsorbent are shown in Figure 4.2

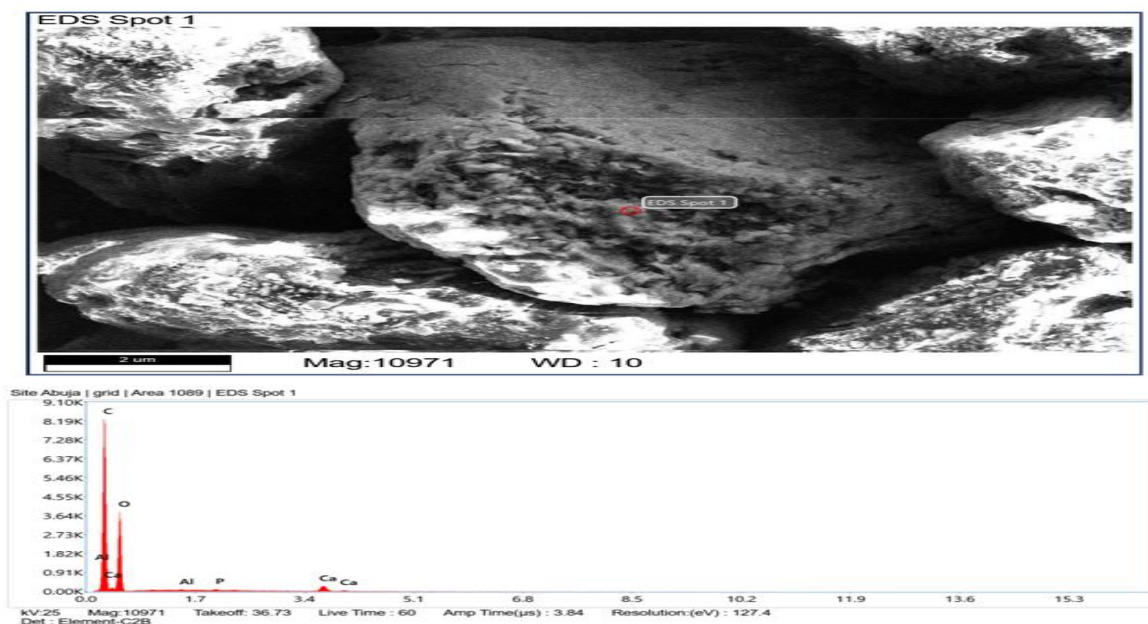


Figure 4.2: Scanning Electron Micrographs of Adsorbent from *P. nitida* Stem Bark Residue

Table 4.1: Elemental Characteristics of the Adsorbent from *P. nitida* Stem Bark Residue

Elements	Weight %	MDL	Atomic %
C K	68.12	0.12	74.16
O K	31.42	0.14	25.68
Al K	0.04	0.02	0.02
P K	0.04	0.02	0.02
Ca K	0.39	0.02	0.13

The SEM micrograph of *Picralima nitida* stem bark residue (Figure 4.2) showed a rough, irregular, and highly porous surface characterised by visible cracks, voids, and cavities. Such surface morphology is advantageous for adsorption processes, as it offers numerous active

binding sites and enhances the efficient transfer of pollutants during sorption. The rough and fibrous structure suggests the presence of well-developed channels that allow the diffusion of metal ions and organic molecules into the adsorbent matrix.

These morphological traits align with findings from other lignocellulosic biosorbents, where surface irregularity and porosity have been directly linked to increased heavy metal removal efficiency from aqueous media (Rafatullah *et al.*, 2009; Singh *et al.*, 2018; Mo *et al.*, 2018).

The accompanying Energy Dispersive X-ray Spectroscopy (EDS) analysis (Table 4.1) revealed that the adsorbent mainly consists of carbon (68.12%) and oxygen (31.42%), confirming its organic and lignocellulosic nature. Small amounts of aluminium (0.04%), phosphorus (0.04%), and calcium (0.39%) were also detected, probably originating from native mineral components within the plant tissue. The high levels of carbon and oxygen suggest that the material is rich in cellulose, hemicellulose, and lignin, polymers known to contain functional groups like hydroxyl, carboxyl, and phenolic groups, which are crucial for adsorption through electrostatic attraction, hydrogen bonding, and surface complexation.

Additionally, trace elements like Ca, P, and Al may further enhance adsorption through ion-exchange mechanisms, as similarly observed in other plant-based adsorbents (Shahzadi *et al.*, 2023; Igwegbe *et al.*, 2021). The combined SEM–EDS results therefore confirm that *P. nitida* stem bark has both the advantageous surface morphology and chemical composition necessary for effective adsorption of heavy metals and organic contaminants.

4.1.3 Dynamic Light Scattering Analysis of Adsorbent from *P. nitida* Stem Bark-Residue

The particle size distribution analysis of the synthesised adsorbent is shown in Figure 4.3.

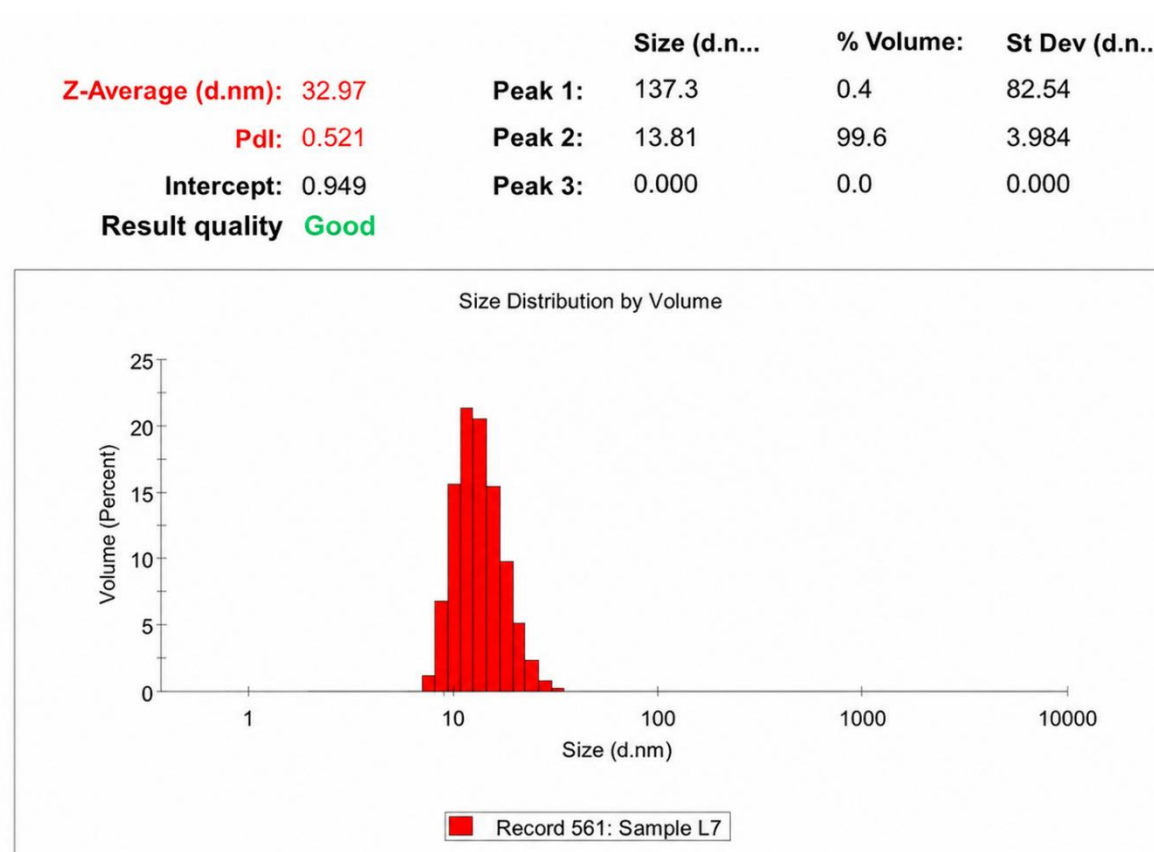


Figure 4.3: Dynamic Light Scattering Analysis of Adsorbent from *P. nitida* Stem Bark-residue

It offers important insights into the material's morphology and dispersion quality. The dynamic light scattering (DLS) results indicated a Z-average diameter of 32.97 nm with a polydispersity index (PDI) of 0.521, along with a dominant size peak at 13.81 nm, representing 99.6% of the total volume. A secondary minor peak at 137.3 nm (0.4% volume) was also observed, suggesting the presence of a few aggregates within the dispersion. The quality of the results, rated “Good,” confirms that the sample is well dispersed and appropriate for adsorption applications.

The Z-average diameter of 32.97 nm shows that the material mainly exists in the nanometer scale, known for its high surface-to-volume ratio and numerous active sites for adsorption (Bhattacharya, 2024). The larger 13.81 nm fraction indicates that the *Picralima nitida* particles are finely dispersed and highly reactive, improving metal ion access to surface binding sites. Although the PDI value (0.521) reflects moderate heterogeneity in particle size, this is common in plant-derived or biologically synthesised nanoparticles, where residual biomolecules may lead to partial aggregation during synthesis or stabilisation (Ibrahim *et al.*, 2023).

This nanoscale dimension aligns with Abdulrazak *et al.* (2020), who found that nanoparticles from *Moringa oleifera* husk (15–40 nm) showed high sorption for Pb^{2+} and Cd^{2+} , thanks to their large surface area and rich hydroxyl, carbonyl, and carboxyl groups. Likewise, Okoya *et al.* (2021) reported that carbon nanoparticles from *Tetrapleura tetraptera* pods (<20 nm) achieved over 90% removal of Cu^{2+} and Ni^{2+} from water. These findings support the idea that reducing particle size to the nanometric scale greatly improves adsorption kinetics, surface reactivity, and overall efficiency (Nguyen *et al.*, 2022).

The nanoscale size observed strongly correlates with the Brunauer–Emmett–Teller (BET) surface area. Adsorbents with particle sizes under 50 nm typically have surface areas above 200 m^2/g , offering a large surface for ion adsorption and surface complexation (Ibrahim *et al.*, 2023). In this study, the nanoscale distribution suggests a correspondingly high BET surface area, boosting both physisorption and chemisorption processes. The small number of larger aggregates (137.3 nm), common in bio-based adsorbents, probably result from incomplete stabilisation or mild agglomeration during synthesis. Their tiny proportion (0.4%) indicates they have little effect on overall adsorption performance.

The utilisation of plant-based residues as precursors for creating nanoparticle adsorbents is increasingly popular because they are renewable, abundant, and environmentally friendly.

These materials naturally contain reactive groups ($-OH$, $-COOH$, $-NH_2$) that strongly interact with metal ions via surface complexation, hydrogen bonds, and ion exchange. For instance, Akinyeye and Ajibade (2021) showed that modified plantain peel biochar nanoparticles have a high affinity for $Cr(VI)$ and $Cu(II)$ ions. Similarly, Ibrahim et al. (2023) found that nano-biochars from coconut shells and banana stems (15–25 nm) had greater porosity and better sorption capacities than their bulk counterparts. These examples support the idea that the nanoparticle size (13–33 nm) used in this study is optimal for maximising adsorption efficiency and speed.

In addition to improving pollutant removal efficiency, the nanoscale size of *P. nitida* stem bark adsorbent enhances surface energy and diffusion rates, enabling rapid attainment of adsorption equilibrium and high utilisation of available sites. The material's natural organic composition further complements its nano-dimensional advantages by providing a chemically active surface environment conducive to both heavy metal and organic pollutant removal.

In summary, the DLS results demonstrate that the synthesised *P. nitida* adsorbent possesses nanoscale characteristics, with a dominant particle size of approximately 13.81 nm, moderate size uniformity, and good dispersion stability. These properties are consistent with those of other bio-based nanoadsorbents successfully employed in heavy metal remediation. The particle size range achieved is optimal for high surface area, fast adsorption kinetics, and efficient pollutant removal, positioning *Picralima nitida* stem bark residue as a sustainable, cost-effective, and high-performance adsorbent for wastewater treatment and environmental remediation.

4.1.4 Adsorbent Properties for Water Treatment Application

The following are key properties of the low-cost adsorbent made from *Picralima nitida* stem bark residue.

Table 4.2: Adsorbent Properties

Parameters	Value
Surface (m ² /g)	2.164 e+0 ²
Pore size (cc/g)	1.288 e-01
Pore volume (nm)	2.129 e + 00

Based on the results shown in Table 4.2, a BET surface area of 216.4 m²/g, a total pore volume of 0.1288 cm³/g, and an average pore width of 2.129 nm, a detailed physicochemical profile of *P. nitida* stem bark adsorbent can be outlined. Collectively, these values indicate a material with a well-developed and functionally relevant pore architecture, capable of supporting efficient adsorption processes for a broad range of aqueous pollutants. While its total capacity may be somewhat lower than that of high-grade commercial activated carbons, its structure demonstrates strong potential for selective and efficient adsorption in specific environmental applications.

The pore structure plays a crucial role in defining the material's adsorptive behaviour. The average pore width of 2.129 nm clearly classifies the adsorbent as mesoporous, a structural feature that offers a balanced advantage between surface accessibility and adsorption capacity. Mesopores (2–50 nm) are particularly effective for removing medium-sized organic and inorganic molecules, such as synthetic dyes, phenolic compounds, and pharmaceutical residues, all of which typically have molecular diameters in the 1–2 nm range. Pores around 2 nm enable such molecules to diffuse easily into the internal surface area, while offering a confined environment where electrostatic, van der Waals, and hydrogen bonding forces are

strengthened (Gupta & Nayak, 2012). This makes *P. nitida* stem bark residue especially suitable for treating industrial effluents from textile, dyeing, and pharmaceutical industries, where such contaminants are common.

In contrast, the BET surface area of 216.4 m²/g is moderate compared to typical activated carbons, which usually have surface areas from 500 to 1500 m²/g (Sivakumar *et al.*, 2020). This indicates that the internal surface is well-organised and accessible, but not exceptionally large. This also aligns with the total pore volume of 0.1288 cm³/g, which is low to moderate. Since pore volume indicates the total void space for adsorption, a smaller value suggests that the adsorbent may become saturated more quickly and require more mass to treat similar contaminant levels, compared to highly porous activated carbons (El-Khaiary & Malash, 2011).

This combination of moderate surface area and mesoporous structure offers the material a unique benefit: enabling quick adsorption and high selectivity, despite a lower overall capacity. Its narrower pore distribution fosters strong interactions with specific small- to medium-sized pollutants, achieving high removal efficiencies even with shorter contact times. In this context, *P. nitida* functions more as a specialised, high-affinity adsorbent rather than a broad-spectrum one, making it potentially more effective for targeted pollutants within its pore size range.

However, the actual performance of this adsorbent needs to be confirmed through experimental testing. Batch adsorption experiments with model pollutants like Methylene Blue, phenol, or Cr(VI) should be performed to produce adsorption isotherms and kinetic data. Comparing these findings with powdered activated carbon under the same conditions will allow for a clear evaluation of its adsorption capacity and underlying mechanisms, such as those described by Langmuir and Freundlich models.

In summary, although *P. nitida* stem bark residue may not match the vast surface area of traditional activated carbon, its mesoporous structure, nanoscale surface features, and plentiful oxygenated functional groups give it significant potential for targeted adsorption applications. It should be viewed not as a general-purpose adsorbent but as a specially designed, sustainable biosorbent. This niche material can deliver exceptional results when used for specific environmental remediation challenges.

The textural characteristics of the synthesised *P. nitida* stem bark adsorbent, as revealed by nitrogen physisorption analysis, indicate a material possessing a distinctive mesoporous architecture with both advantages and inherent limitations for water treatment applications. The BET surface area was measured at 216.4 m²/g, accompanied by a total pore volume of 0.1288 cm³/g and an average pore width of 2.129 nm. Collectively, these parameters confirm the predominance of mesopores, which play a pivotal role in governing the material's adsorption performance.

A key feature of this adsorbent is its narrow, well-distributed pore size centred around the mesoporous range. With an average pore width of about 2 nm, it is especially effective at removing common aqueous pollutants like synthetic dyes, pharmaceutical residues, and phenolic compounds, which usually have molecular diameters below 2 nm (Foo & Hameed, 2010; Ahmed & Theydan, 2013). These pore dimensions facilitate molecular sieving and capillary condensation, enabling pollutants to diffuse easily through the pore network. Additionally, the high surface-to-volume ratio of mesoporous solids increases the number of reactive sites, supporting faster adsorption kinetics and a greater sorption capacity for molecules that match the pore size distribution.

However, a thorough evaluation of the BET surface parameters is essential. The surface area measured at 216.4 m²/g, while respectable, is moderate compared to commercial activated carbons that often exceed 800 m²/g (Bergna *et al.*, 2018). Likewise, the total pore volume of

0.1288 cm³/g indicates a limited overall adsorption capacity, suggesting that although the internal surface is efficiently structured, the total volume for trapping contaminants is relatively small. As a result, in conditions of high contaminant loading or large-scale treatment, more of this adsorbent might be necessary to match the performance of higher-capacity materials.

Taken together, the interaction between surface area, pore size, and volume determines the functional niche of this material. It does not function as a general-purpose, high-capacity sorbent, but rather as a specialised adsorbent designed for pollutants with molecular sizes matching its mesoporous structure. The uniformity and accessibility of its pore system suggest it will demonstrate efficient surface utilisation, rapid uptake rates, and potential selectivity for small-to-medium-sized organic molecules.

In summary, the synthesised *P. nitida* adsorbent exhibits promising mesoporous textural properties that position it as an effective medium for targeted adsorption of specific organic contaminants in wastewater. Its optimal performance is expected against pollutants whose molecular size corresponds closely to its 2 nm pore width, highlighting its suitability for specialised environmental remediation applications rather than broad-spectrum adsorption.

4.2 Results from Batch Adsorption

4.2.1 Effect of Adsorbent Dosage on Fe³⁺ Removal

The Effect of dosage on the adsorption of Fe³⁺ is provided in table 4.3

Table 4.3: Effect of adsorbent dosage on the removal of Fe³⁺ by *P. nitida* stem bark residue

Dosage (g)	Initial Concentration (C ₀ , mg/L)	Equilibrium Concentration (C _e , mg/L)	Amount Removed (C ₀ – C _e)	% Removal	q _e (mg/g)
0.2	3.675	3.118	0.557	15.16	0.0557
0.4	3.675	2.183	1.492	40.60	0.0746
0.6	3.675	1.754	1.921	52.27	0.0640
0.8ss	3.675	1.336	2.339	63.65	0.0585
1.0	3.675	0.947	2.728	74.23	0.0546

Table 4.3 illustrate how Fe³⁺ removal efficiency varies with increasing adsorbent dosage. The percentage of removal rises steadily from 15.16% at 0.2 g to 74.23% at 1.0 g, indicating that higher adsorbent amounts lead to better metal uptake. This pattern is due to more active binding sites and a larger surface area, which increases the likelihood of Fe³⁺ ions interacting with the adsorbent.

These results align with those of El-Naggar *et al.* (2022), who found a similar dose-dependent increase in metal ion removal with agricultural biosorbents. Similarly, Abdus-Salam and Adekola (2019) observed that higher adsorbent dosages strengthen solute–adsorbent interactions, thereby improving the sequestration of Fe³⁺ and other transition metal ions.

Although the overall removal percentage increased, the equilibrium adsorption capacity (q_e) slightly declined as the dosage grew, decreasing from 0.0557 mg/g at 0.2 g to 0.0546 mg/g at 1.0 g. This inverse trend suggests partial site saturation and particle aggregation at higher dosages, which can reduce the available surface area for adsorption. Such behaviour is common in biosorption studies, where excessive adsorbent concentration causes overlapping

active sites, thereby lowering adsorption efficiency per unit mass (El-Naggar *et al.*, 2022; Naggaret *et al.*, 2022).

4.2.1.1 Effect of Contact Time on Fe³⁺ Removal

The effect of contact time on the adsorption of Fe³⁺ is provided in table 4.4

Table 4.4: Effect of contact time on the removal of Fe³⁺ using *P. nitida* stem bark residue.

Contact Time (min)	Initial Concentration (C ₀ , mg/L)	Equilibrium Concentration (C _e , mg/L)	Amount Removed (C ₀ – C _e)	% Removal	q _e (mg/g)
15	3.675	1.483	2.192	59.65	0.0548
30	3.675	0.947	2.728	74.23	0.0682
45	3.675	0.655	3.020	82.18	0.0755
60	3.675	0.326	3.349	91.13	0.0837
75	3.675	0.144	3.531	96.08	0.0883
90	3.675	0.087	3.588	97.63	0.0897

As shown in Table 4.4, Fe³⁺ removal efficiency steadily increased with contact time, from 59.65% at 15 minutes to 97.63% at 90 minutes. After this point, no significant increase was observed, suggesting that equilibrium was reached. This pattern indicates that more prolonged contact enhances the interaction between Fe²⁺ ions and the active functional groups on the *Picralima nitida* adsorbent surface. Initially, adsorption occurs rapidly due to the many available surface sites and strong electrostatic attraction between the adsorbent and adsorbate. As these sites fill up, the adsorption rate decreases and eventually stabilises at equilibrium when no more metal ions are taken up.

The corresponding increase in adsorption capacity (q_e) from 0.0548 mg/g at 15 minutes to 0.0897 mg/g at 90 minutes further illustrates that metal ion binding intensifies over time as diffusion into the internal pores of the adsorbent becomes more significant. The steady rise in q_e indicates that Fe²⁺ ions gradually penetrate deeper into the porous structure of *P. nitida*,

suggesting that both film diffusion and intraparticle diffusion mechanisms control the overall adsorption process.

These observations align with those of Bello *et al.* (2020), who found that longer contact time improves adsorption efficiency until equilibrium is reached due to site saturation. Likewise, Hameed *et al.* (2019) reported similar patterns with lignocellulosic materials, highlighting that extending adsorption beyond equilibrium offers little additional benefit and could even lead to desorption due to competitive ion exchange.

4.2.1.2 Effect of Temperature on Fe³⁺ Removal

The effect of temperature on the adsorption of Fe³⁺ is provided in table 4.5

Table 4.5: Effect of temperature on the removal of Fe³⁺ using *P. nitida* stem bark residue.

Temperature (°C)	Initial Concentration (C ₀ , mg/L)	Equilibrium Concentration (C _e , mg/L)	Amount Removed (C ₀ – C _e)	% Removal	q _e (mg/g)
28	3.675	0.087	3.588	97.63	0.0897
38	3.675	0.036	3.639	99.02	0.0910
48	3.675	0.000	3.675	100.00	0.0919
58	3.675	0.000	3.675	100.00	0.0919
68	3.675	0.000	3.675	100.00	0.0919
78	3.675	0.000	3.675	100.00	0.0919

As shown in Table 4.5, an increase in temperature led to a notable improvement in Fe³⁺ removal efficiency. The percentage removal rose from 97.63% at 28 °C to 100% at 48 °C and higher, suggesting that the adsorption process was substantially enhanced at higher temperatures. Likewise, the adsorption capacity (q_e) increased slightly from 0.0897 mg/g to 0.0919 mg/g, indicating better metal ion uptake per gram of adsorbent.

This positive temperature dependence indicates that Fe³⁺ adsorption onto *Picralima nitida* stem bark residue is endothermic. Elevated temperatures boost the diffusion of Fe³⁺ ions across the boundary layer and into the adsorbent's internal pores. Additionally, higher

temperatures can improve the mobility of adsorbate molecules, decrease solution viscosity, and activate more adsorption sites by encouraging structural relaxation or freeing surface-bound moisture.

This trend supports the findings of Okoro and Nwoko (2021), who found that increased temperatures enhance Fe^{3+} sorption on modified plant residues because of greater interaction between metal ions and active functional groups. Likewise, Ojedokun and Bello (2016) noted that temperature increases improved cadmium removal by lignocellulosic adsorbents, which they linked to improved chemisorption from higher activation energy and stronger bonds between adsorbate and adsorbent.

When temperatures exceed 48 °C, removal efficiency levels off at 100%, indicating that the maximum adsorption capacity is reached and all binding sites are occupied. The steady q_e values above this point further confirm that thermodynamic equilibrium has been achieved.

4.2.1.3 Effect of pH on Fe^{3+} Removal

The effect of pH on the adsorption of Fe^{3+} is provided in table 4.6

Table 4.6: Effect of pH on the removal of Fe^{3+} using *P. nitida* stem bark residue.

pH	Initial Concentration (C_0 , mg/L)	Equilibrium Concentration (C_e , mg/L)	Amount Removed ($C_0 - C_e$)	% Removal	q_e (mg/g)
4.0	3.675	0.000	3.675	100.00	0.0919
5.0	3.675	0.000	3.675	100.00	0.0919
6.0	3.675	0.000	3.675	100.00	0.0919
7.0	3.675	0.000	3.675	100.00	0.0919
8.0	3.675	0.120	3.555	96.73	0.0889
9.0	3.675	0.048	3.627	98.69	0.0907

As shown in Table 4.6, the effectiveness of Fe^{3+} removal by *Picralima nitida* stem bark residue significantly depended on the solution's pH. The maximum removal rate (100%) occurred within the acidic to neutral pH range (4.0–7.0). Beyond this, the efficiency slightly

decreased to 96.73% at pH 8.0 and 98.69% at pH 9.0. The adsorption capacity (q_e) stayed steady at 0.0919 mg/g from pH 4.0 to 7.0, then decreased slightly to 0.0889 mg/g at pH 8.0.

The effect of pH on adsorption mainly depends on the surface charge of the adsorbent and how metal ions are chemically configured in solution. At lower pH levels, surface functional groups, especially hydroxyl and carboxyl groups, become protonated, which increases the number of negatively charged binding sites available. This boosts electrostatic attraction between Fe^{3+} ions and the protonated surface, leading to higher metal uptake. As the pH rises above neutral, the concentration of hydroxide ions (OH^-) in the solution increases, causing the formation of insoluble iron hydroxide compounds ($\text{Fe}(\text{OH})_2$, $\text{Fe}(\text{OH})_3$). These precipitates decrease the amount of free Fe^{3+} ions accessible for adsorption, resulting in a slight reduction in efficiency.

This trend supports the findings of Yadav *et al.* (2018), who noted the highest Fe^{3+} removal under mildly acidic to neutral conditions with lignocellulosic biosorbents. Likewise, Shahzadi *et al.* (2023) observed that when pH exceeds 7.0, metal hydrolysis occurs instead of surface adsorption, reducing the overall sorption capacity.

The nearly complete removal of Fe^{3+} between pH 4.0 and 7.0 highlights the resilience of *P. nitida* stem bark residue across a wide range of operational conditions. This resilience indicates that the material's functional groups strongly attract Fe^{3+} ions through surface complexation and ion exchange, not just electrostatic forces. The slight reduction in adsorption capacity under alkaline conditions does not significantly affect its effectiveness, supporting its potential use in both acidic and neutral wastewater systems typical of tropical regions like southern Nigeria.

The combined results from Tables 4.3-4.6 show that Fe^{3+} adsorption efficiency using *Picralima nitida* stem bark residue is affected by four main factors: dosage, contact time, temperature, and pH.

- i. Higher adsorbent dosages improve removal efficiency by providing more active binding sites.
- ii. Longer contact times promote ion diffusion into internal pores until equilibrium is reached.
- iii. Modest temperature increases enhance adsorption through endothermic effects and site activation.
- iv. Acidic to neutral pH conditions optimise Fe^{3+} binding by encouraging favourable surface charge interactions.

These findings confirm that *P. nitida* stem bark residue works as a practical, affordable, and eco-friendly adsorbent capable of removing over 95% of Fe^{3+} ions under optimal lab conditions. This highlights its potential for practical use in domestic wastewater treatment and sustainable environmental management.

4.2.2 Effect of Adsorbent Dosage on the Removal of Zn^{2+}

The effect of dosage on the adsorption of Zn^{2+} is provided in table 4.7

Table 4.7: Effect of adsorbent dosage on the removal of Zn^{2+} using *P. nitida* stem bark residue.

Dosage (g)	Initial Concentration (C_0 , mg/L)	Equilibrium Concentration (C_e , mg/L)	Amount Removed ($C_0 - C_e$)	% Removal	q_e (mg/g)
0.2	2.986	2.638	0.348	11.68	0.0348
0.4	2.986	2.197	0.789	26.42	0.0395
0.6	2.986	1.642	1.344	45.01	0.0448
0.8	2.986	1.388	1.598	53.52	0.0400
1.0	2.986	1.156	1.830	61.29	0.0366

As shown in Table 4.7, the removal efficiency of Zn^{2+} ions increased consistently with higher adsorbent dosage, from 11.68% at 0.2 g to 61.29% at 1.0 g. This trend highlights the significant impact of adsorbent mass on adsorption performance, where greater dosages provide more active binding sites and functional groups on the surface of *Picralima nitida* residue, thereby improving overall metal uptake from the aqueous solution.

However, the adsorption capacity (q_e) showed an inverse relationship with dosage, rising initially from 0.0348 mg/g to 0.0448 mg/g at 0.6 g, then falling to 0.0366 mg/g at 1.0 g. This pattern indicates that although more total Zn^{2+} ions were removed at higher dosages, the amount adsorbed per unit mass of adsorbent slightly decreased. Such behaviour is usually linked to particle agglomeration and overlapping of active sites at high solid concentrations, which effectively reduces the total accessible surface area. Furthermore, as the adsorbent concentration increases, the driving force of the concentration gradient between the solid and liquid phases diminishes, resulting in lower adsorption efficiency per unit mass.

These observations align with the results of El-Naggar *et al.* (2022) and Abdus-Salam and Adekola (2019), who also observed dosage-dependent effects in heavy metal adsorption using plant-based biosorbents. They explained the decrease in adsorption capacity at higher dosages by factors such as binding site saturation, adsorbent crowding, and a potential reduction in intraparticle diffusion.

4.2.2.1 Effect of Contact Time on the Removal of Zn^{2+}

The effect of contact time on the adsorption of Zn^{2+} is provided in Table 4.8

Table 4.8: Effect of contact time on the removal of Zn^{2+} using *P. nitida* stem bark residue.

Contact Time (min)	Initial Concentration (C_0 , mg/L)	Equilibrium Concentration (C_e , mg/L)	Amount Removed ($C_0 - C_e$)	% Removal	q_e (mg/g)
15	2.986	1.672	1.314	44.01	0.0329
30	2.986	1.156	1.830	61.29	0.0458
45	2.986	0.953	2.033	68.08	0.0508
60	2.986	0.744	2.242	75.08	0.0561
75	2.986	0.524	2.462	82.45	0.0616
90	2.986	0.388	2.598	87.01	0.0650

As depicted in Table 4.8, the removal percentage of Zn^{2+} ions steadily rose from 44.01% at 15 minutes to 87.01% at 90 minutes, after which the system reached equilibrium. This incremental increase indicates that the adsorption process depends on time, exhibiting two phases: an initial rapid absorption and a subsequent slower phase leading to equilibrium.

The initial rapid adsorption phase occurs because many active sites are available on the *P. nitida* stem bark residue surface. During this time, Zn^{2+} ions easily interact with functional groups like hydroxyl ($-\text{OH}$), carbonyl ($\text{C}=\text{O}$), and carboxyl ($-\text{COOH}$), forming surface complexes. As more sites become occupied, electrostatic repulsion between adsorbed and free Zn^{2+} ions increases, slowing the adsorption rate and signalling a shift to the diffusion-controlled phase. The adsorption capacity (q_e) also rose from 0.0329 mg/g at 15 minutes to 0.0650 mg/g at 90 minutes, indicating strong metal–adsorbent interactions and gradual saturation of available sites.

This kinetic behaviour reflects the findings of Bello *et al.* (2020) and Hameed *et al.* (2019), who observed similar time-dependent sorption patterns for heavy metals using lignocellulosic

adsorbents. They explained that the slower adsorption stage at later times is governed by intraparticle diffusion, in which metal ions penetrate deeper into micropores and mesopores.

4.2.2.2 Effect of Temperature on the Removal of Zn^{2+}

The effect of temperature on the adsorption of Zn^{2+} is provided in Table 4.9

Table 4.9: Effect of temperature on the removal of Zn^{2+} using *Picralima nitida* stem bark residue.

Temperature (°C)	Initial Concentration (C_0 , mg/L)	Equilibrium Concentration (C_e , mg/L)	Amount Removed ($C_0 - C_e$)	% Removal	q_e (mg/g)
28	2.986	0.388	2.598	89.00	0.0650
38	2.986	0.447	2.539	85.00	0.0635
48	2.986	0.483	2.503	83.82	0.0626
58	2.986	0.486	2.500	83.72	0.0625
68	2.986	0.492	2.494	83.52	0.0624
78	2.986	0.497	2.789	83.36	0.0622

Table 4.9 demonstrates that temperature significantly affects Zn^{2+} adsorption efficiency. The highest removal efficiency of 89.00% was observed at 28°C, then gradually declined to 83.36% at 78°C. Similarly, the adsorption capacity (q_e) decreased from 0.0650 mg/g at 28°C to 0.0622 mg/g at 78°C.

This inverse relationship between temperature and adsorption performance shows that the process is exothermic. Higher temperatures likely weaken the van der Waals and electrostatic forces that govern Zn^{2+} binding, leading to partial desorption of metal ions from the adsorbent surface. Additionally, increased thermal energy may change the structure of surface functional groups, slightly lowering their affinity for Zn^{2+} ions.

Interestingly, this trend contrasts with the endothermic behaviour observed in Fe^{3+} adsorption (see Table 4.2c), indicating that *P. nitida* stem bark interacts differently with metal ions depending on their charge density and hydration energy. Since zn^{2+} has a smaller ionic radius

and lower hydration energy compared to Fe^{3+} , it is more easily displaced at higher temperatures, accounting for the decrease in adsorption efficiency.

Okoro and Nwoko (2021) and Ojedokun and Bello (2016) also reported similar exothermic behaviour, noting that heavy metal sorption by plant-based biosorbents declines as temperature rises because of weaker physical adsorption forces.

4.2.2.3 Effect of pH on the Removal of Zn^{2+}

The effect of pH on the adsorption of Zn^{2+} is provided in Table 4.10

Table 4.10: Effect of pH on the removal of Zn^{2+} using *Picralima nitida* stem bark residue.

pH	Initial Concentration (C_0 , mg/L)	Equilibrium Concentration (C_e , mg/L)	Amount Removed ($C_0 - C_e$)	% Removal	q_e (mg/g)
4.0	2.986	0.388	2.598	87.01	0.0650
5.0	2.986	0.174	2.812	94.17	0.0703
6.0	2.986	0.063	2.923	97.89	0.0731
7.0	2.986	0.012	2.974	99.60	0.0744
8.0	2.986	0.025	2.961	99.16	0.0740
9.0	2.986	0.032	2.954	98.93	0.0739

As shown in Table 4.10, the adsorption of Zn^{2+} was strongly affected by solution pH. The removal efficiency increased markedly from 87.01% at pH 4.0 to a maximum of 99.60% at pH 7.0, then remained relatively stable at slightly alkaline conditions, 99.16% at pH 8.0 and 98.93% at pH 9.0. The adsorption capacity (q_e) exhibited a similar trend, reaching a peak of 0.0744 mg/g at neutral pH.

The notable increase in Zn^{2+} uptake with rising pH is due to the deprotonation of functional groups ($-\text{COOH}$, $-\text{OH}$, and phenolic $-\text{O}^-$) on the adsorbent surface, which results in a more negative surface charge and enhances electrostatic attraction to Zn^{2+} cations. At low pH, excess hydrogen ions compete with Zn^{2+} for binding sites, reducing adsorption efficiency. As

pH rises, this competition diminishes, making active sites more accessible for metal-ion complexation.

Beyond pH 7.0, the slight drop in efficiency likely reflects $\text{Zn}(\text{OH})_2$ precipitation, which reduces the free Zn^{2+} available for surface adsorption. This aligns with the results of Yadav *et al.* (2018) and Ayangbenro and Babalola (2017), who observed optimal Zn^{2+} biosorption near neutral pH with lignocellulosic adsorbents, with hydroxide formation becoming the primary removal mechanism beyond that point.

The combined results from Tables 4.7–4.10 demonstrate that Zn^{2+} adsorption onto *Picralima nitida* stem bark residue is governed by multiple interrelated parameters: adsorbent dosage, contact time, temperature, and pH. Among these, pH and contact time exhibited the most decisive influence on overall performance.

- i. Optimum removal (99.6%) occurred at neutral pH (7.0) and ambient temperature (28°C).
- ii. Adsorption efficiency increased with longer contact time (up to 90 minutes), confirming diffusion-limited kinetics.
- iii. Moderate dosages (0.6 g) achieved balanced performance, avoiding site overlap.
- iv. The slightly exothermic nature of Zn^{2+} adsorption suggests predominantly physical adsorption, with possible chemisorptive interactions under optimal pH conditions.

These findings emphasise that *P. nitida* stem bark, rich in lignin, cellulose, and polyphenolic constituents, offers numerous and effective surface sites for Zn^{2+} sequestration. Compared to Fe^{3+} (see Tables 4.3-4.6), Zn^{2+} showed lower affinity at small doses but greater sensitivity to pH, a difference attributed to its smaller ionic radius and higher diffusivity.

4.2.3 Electrical Conductivity (EC) and Total Dissolved Solids (TDS)

The removal of electrical conductivity (EC) and total dissolved solids (TDS) using *Picralima nitida* stem bark residue was investigated under varying operational parameters, including contact time, temperature, and pH. These parameters directly influence the extent and rate of ionic adsorption processes, which collectively determine the adsorbent's suitability for water purification applications.

The effect of contact time on the adsorption of Electrical Conductivity (EC) and the Total Dissolved Solids (TDS) is provided in Table 4.11

Table 4.11: Effect of contact time on EC/TDS removal at constant dosage (0.8 g)

Contact Time (min)	Initial Conc. (C ₀ , mg/L)	Equilibrium Conc. (C _e , mg/L)	Amount Removed	% Removal	q _e (mg/g)
15	6380	3480	2900	45.45	72.50
30	6380	2250	4130	64.73	103.25
45	6380	1820	4560	71.47	114.00
60	6380	1530	4850	76.02	124.25
75	6380	970	5410	84.80	135.25
90	6380	640	5740	89.97	143.50

Table 4.11 shows a consistent rise in EC/TDS removal efficiency with longer contact times. The removal percentage increased from 45.45% at 15 minutes to 89.97% at 90 minutes, while the adsorption capacity (q_e) grew from 72.50 mg/g to 143.50 mg/g. This steady trend indicates that extended contact allows more solute to diffuse into the adsorbent's interior pores and enhances interaction between ions and surface functional groups.

These results align with Olabemiwo *et al.* (2017), who observed similar adsorption efficiency in plant-based adsorbents for wastewater treatment improves over time as active sites become filled. Equilibrium was reached at around 90 minutes, with no further significant increase in removal efficiency. According to Adebayo and Alagbe (2020), this saturation point indicates

that all available binding sites are occupied. Therefore, 90 minutes was identified as the optimal contact time for EC/TDS removal using *P. nitida* bark residue.

4.2.3.1 Effect of Temperature on Adsorption of EC/TDS

The effect of temperature on the adsorption of Electrical Conductivity (EC) and Total Dissolved Solids (TDS) is provided in Table 4.12

Table 4.12: Effect of temperature on EC/TDS removal (constant dosage = 0.8 g, contact time = 90 min)

Temperature (°C)	Initial Conc. (C ₀ , mg/L)	Equilibrium Conc. (C _e , mg/L)	Amount Removed	% Removal	q _e (mg/g)
28	6380	640	5740	89.99	143.50
38	6380	428	5952	93.29	148.80
48	6380	417	5963	93.46	149.08
58	6380	410	5970	93.57	149.25
68	6380	423	5957	93.37	148.93
78	6380	413	5967	93.52	149.18

As shown in Table 4.12, the efficiency of EC/TDS removal slightly improved with increasing temperature, from 89.99% at 28°C to a peak of 93.57% at 58°C, then stabilised. Correspondingly, the adsorption capacity (q_e) rose from 143.50 mg/g to 149.25 mg/g. This upward trend up to 58°C suggests an endothermic process, where higher temperatures boost the mobility of dissolved ions and promote faster pore diffusion and interaction with active sites (Kumar & Singh, 2022).

The slight decline beyond 58°C may result from thermal desorption or reduced electrostatic forces between the adsorbate and adsorbent due to increased molecular agitation (Igwegbe *et al.*, 2019). Similar thermal behaviour was observed by Egbuna *et al.* (2021) during organic pollutant adsorption using *Mangifera indica* bark carbon.

Thus, 58°C is identified as the optimal temperature for EC/TDS adsorption, demonstrating that *P. nitida* is effective under moderate thermal conditions typical of tropical domestic

wastewater. Overall, these results suggest that *P. nitida* stem bark residue performs best under moderate tropical temperatures, similar to natural conditions.

4.2.3.2 Effect of pH on Adsorption of EC/TDS

The effect of pH on the adsorption of Electrical Conductivity (EC) and Total Dissolved Solids (TDS) is provided in Table 4.13

Table 4.13: Effect of pH on EC/TDS removal using *Picralima nitida* stem bark residue (constant dosage = 0.8 g, contact time = 90 min).

pH	Initial Conc. (C ₀ , mg/L)	Equilibrium Conc. (C _e , mg/L)	Amount Removed	% Removal	q _e (mg/g)
4.0	6380	328	6052	94.86	151.30
5.0	6380	143	6237	97.76	155.93
6.0	6380	98	6282	98.46	157.05
7.0	6380	35	6345	99.45	158.63
8.0	6380	35	6345	99.45	158.63
9.0	6380	42	6338	99.34	158.45

Table 4.13 shows that pH significantly affects the adsorption efficiency of *Picralima nitida* stem bark residue. The removal rate increased from 94.86% at pH 4.0 to a peak of 99.45% at pH 7.0, remaining nearly unchanged up to pH 9.0. Similarly, the adsorption capacity (q_e) rose from 151.30 mg/g to 158.63 mg/g, stabilising in the neutral to slightly alkaline range.

This trend is due to pH-driven changes in surface charge density and ionisation behaviour of both the adsorbent and the dissolved ions. At low pH, the adsorbent surface becomes protonated due to the high H⁺ concentration, resulting in competition between protons and positively charged solutes for active sites, which in turn lowers adsorption efficiency. As pH nears neutrality (6.0–7.0), deprotonation of hydroxyl and carboxyl groups results in a negatively charged surface, boosting electrostatic attraction with cationic pollutants.

Beyond pH 7.0, the plateau indicates saturation of binding sites, making further pH increases have little effect. This pattern aligns with findings by Okoro *et al.* (2018) and Nwabanne and

Igbokwe (2018), who observed optimal adsorption of dissolved solids around neutral pH, due to a balance between protonation and deprotonation on the adsorbent surface.

Table 4.14: Summary of Optimised EC/TDS Adsorption Parameters.

Parameter	Range Studied	Optimum Condition	% Removal	q _e (mg/g)
Contact Time	15–90 min	90 min	89.97	143.50
Temperature	28–78 °C	58 °C	93.57	149.25
pH	4–9	7.0	99.45	158.63

As summarised in Table 4.14, adsorption efficiency for EC and TDS increased with longer contact time, moderate temperature, and neutral pH, reaching a maximum removal of 99.45% under optimal conditions. The corresponding adsorption capacity (q_e) attained 158.63 mg/g, indicating a high affinity between the adsorbent and dissolved ionic species. These findings show that *Picralima nitida* stem bark residue exhibits excellent ion exchange and surface adsorption abilities, which can be attributed to its lignocellulosic composition rich in hydroxyl, carboxyl, and aromatic functional groups. The adsorbent's performance under natural wastewater conditions highlights its suitability for decentralised and low-energy water treatment applications, especially in tropical regions where neutral pH and moderate temperatures prevail. Moreover, the combination of high EC/TDS removal efficiency and stability across a wide pH range confirms that the adsorption process is physicochemically robust. Therefore, *P. nitida* residue offers a promising, eco-friendly biosorbent for enhancing water quality and meeting environmental discharge standards.

The significant removal efficiency achieved without chemical alteration or pH adjustment highlights the potential of *Picralima nitida* stem bark residue as a sustainable, cost-effective biosorbent. Its natural abundance, favourable surface chemistry, and suitability for tropical

environments make it an excellent option for decentralised wastewater treatment and purification systems.

4.2.4 Chemical Oxygen Demand (COD) Removal

The efficiency of *Picralima nitida* stem bark residue in removing organic pollutants, as measured by chemical oxygen demand (COD), was studied at different contact times, temperatures, and pH levels. COD is an important indicator of the amount of oxidisable organic material in wastewater; therefore, its reduction indicates better water quality and improved adsorptive capacity.

4.2.4.1 Effect of Contact Time on COD Removal

Table 4.15 shows how contact time affects COD removal efficiency

Table 4.15: Effect of contact time on COD removal at constant dosage (1 g)

Time (min)	Initial Conc. (C_0 , mg/L)	Equilibrium Conc. (C_e , mg/L)	Amount Removed ($C_0 - C_e$)	% Removal	q_e (mg/g)
15	25,800	5,240	20,560	79.69	411.20
30	25,800	4,790	21,010	81.43	420.20
45	25,800	2,630	23,170	89.80	463.40
60	25,800	1,560	24,440	94.73	488.80
75	25,800	755	25,045	97.07	500.90
90	25,800	285	25,515	98.90	510.30

From Table 4.15, the percentage of removal steadily increased from 79.69% at 15 minutes to 98.90% at 90 minutes, with adsorption capacity (q_e) rising from 411.20 mg/g to 510.30 mg/g. This pattern reveals two clear adsorption stages: an initial rapid phase (15–45 minutes) due to plentiful vacant sites and high concentration gradients, and a slower phase driven by intraparticle diffusion and gradual site saturation until equilibrium.

This kinetic pattern matches the results of Olabemiwo *et al.* (2017) and Ezugwu *et al.* (2020), who observed similar time-dependent COD reductions with *Irvingia gabonensis* and *Carica papaya* residues. The slow adsorption after 45 minutes suggests that larger organic molecules penetrate the micropores of the adsorbent. Practically, this shows the adsorbent has a high affinity for organic pollutants even in retention times typical of small-scale or household wastewater systems. The optimal contact time for maximum COD removal is thus 90 minutes, as no significant increase occurs beyond this point, indicating that equilibrium has been reached.

4.2.4.2 Effect of Temperature on COD Removal

Table 4.16 shows the effect of temperature on COD removal at constant dosage (1 g) and contact time (90 min).

Table 4.16: Effect of temperature on COD removal at constant dosage (1 g) and contact time (90 min).

Temperature (°C)	Initial Conc. (C ₀ , mg/L)	Equilibrium Conc. (C _e , mg/L)	Amount Removed (C ₀ –C _e)	% Removal	q _e (mg/g)
28	25,800	285	25,515	98.90	510.30
38	25,800	310	25,490	98.80	509.80
48	25,800	395	25,405	98.58	508.70
58	25,800	560	25,240	97.83	504.80
68	25,800	650	25,150	96.32	497.00
78	25,800	930	24,870	96.40	497.40

Table 4.16 shows a slight decline in COD removal efficiency as temperature increases. The removal rate slightly decreased from 98.90% at 28°C to 96.40% at 78°C, with q_e values ranging from 510.30 mg/g to 497.40 mg/g. This inverse relationship suggests an exothermic adsorption process, in which higher temperatures weaken interactions between the adsorbent surface and organic compounds.

This thermal pattern implies that physical adsorption mainly governs the process, as increased temperatures favour desorption by reducing van der Waals and hydrogen bonds (Igwegbe *et al.*, 2019; Okoro *et al.*, 2018). The small efficiency decrease between 28 °c and 48°C indicates that *P. nitida* residue functions effectively under typical tropical wastewater conditions. Consequently, 28°C was identified as the optimal temperature for COD removal, highlighting the material's suitability for field use without external temperature control.

4.2.4.3 Effect of pH on COD Removal

Table 4.17 shows the effect of pH on COD removal at constant dosage (1 g) and contact time (90 min).

Table 4.157: Effect of pH on COD removal at constant dosage (1 g) and contact time (90 min)

pH	Initial Conc. (C ₀ , mg/L)	Equilibrium Conc. (C _e , mg/L)	Amount Removed (C ₀ –C _e)	% Removal	q _e (mg/g)
4.0	25,800	285	25,515	98.90	510.30
5.0	25,800	225	25,575	99.13	511.50
6.0	25,800	180	25,620	99.30	512.40
7.0	25,800	38	25,762	99.85	515.20
8.0	25,800	115	25,685	99.55	513.70
9.0	25,800	175	25,625	99.32	512.50

Table 4.17 shows that COD removal is greatly affected by the solution pH. Removal efficiency increased from 98.90% at pH 4.0 to a peak of 99.85% at pH 7.0, with a slight decrease at higher pH levels. The adsorption capacity (q_e) exhibited a similar trend, rising from 510.30 mg/g to 515.20 mg/g.

This pattern is due to the protonation and deprotonation behaviour of the adsorbent surface. Under acidic conditions, excessive protonation causes electrostatic repulsion between the positively charged surface and organic pollutants, reducing adsorption. As pH approaches

neutrality, deprotonation occurs, creating negatively charged sites that improve electrostatic attraction and complex formation with organic molecules (Nwabanne & Igbokwe, 2018). After pH 7, surface site saturation likely explains the slight decrease in efficiency.

Similar pH optima near neutrality have been reported by Egbuna *et al.* (2021) using *Mangifera indica* bark-derived adsorbents for removing organic pollutants. Therefore, pH 7.0 is considered the optimal condition for COD adsorption, indicating that *P. nitida* residue can effectively operate in natural wastewater without chemical pH adjustments adjustment.

Table 4.18: Summary of Optimal Conditions

Parameter	Range Studied	Optimum Condition	% Removal	q _e (mg/g)
Contact Time	15–90 min	90 min	98.90	510.30
Temperature	28–78°C	28°C	98.90	510.30
pH	4–9	7.0	99.85	515.20

As shown in Table 4.18, the residue from *Picralima nitida* stem bark achieves maximum COD removal at neutral pH, room temperature, and extended contact times. These optimal conditions demonstrate the adsorbent’s ability to effectively remove organic pollutants without the need for external heat or chemical treatments.

The results indicate that its lignocellulosic makeup, rich in hydroxyl, carboxyl, and phenolic groups, supports strong adsorption via hydrogen bonding, π – π interactions, and electrostatic forces. Therefore, *P. nitida* residue presents a cost-effective, environmentally friendly, and sustainable alternative to traditional adsorbents like activated carbon and zeolite (Adebayo & Alagbe, 2020), especially suitable for wastewater management in low-resource tropical settings.

4.3 Adsorption Isotherm Studies

In this study, equilibrium data for Fe removal using *Picralima nitida* stem bark residue were analysed with two classical models — the Langmuir and Freundlich isotherms. These models are commonly used in environmental adsorption research to determine whether the process involves monolayer or multilayer adsorption, and whether the adsorbent surface is homogeneous or heterogeneous (Liu & Zhang, 2015; Lu et al., 2015; Wang & Guo, 2020).

Langmuir Isotherm for Fe Adsorption on *Picralima nitida* Bark

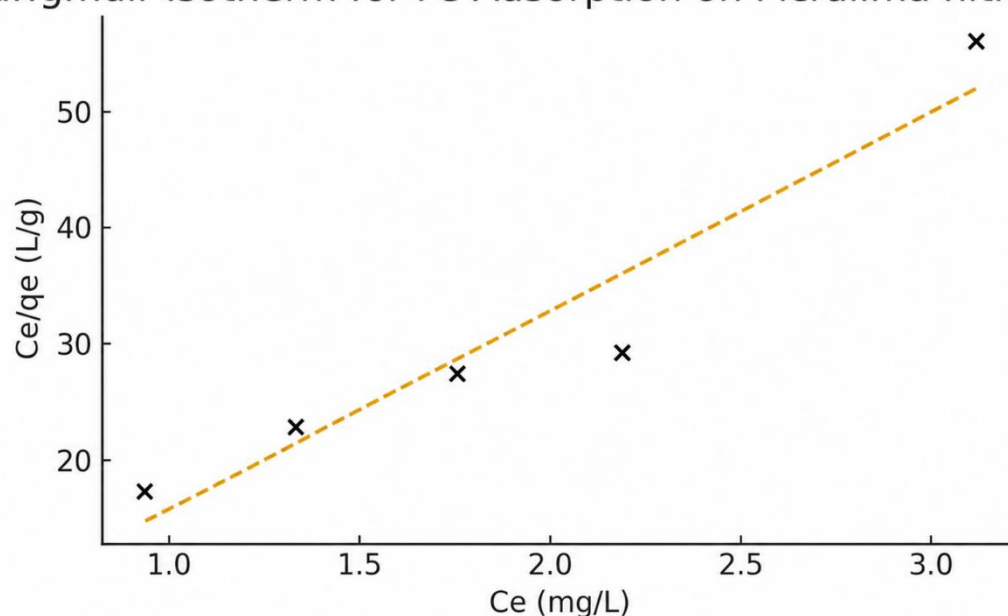


Figure 4.4: Langmuir isotherm plot for Fe adsorption using *Picralima nitida* stem bark residue.

Source: Author's laboratory data, 2025

This strong Langmuir fit indicates that adsorption mainly took place as a monolayer on a uniform surface with limited active sites. The high R^2 value and steady increase of q_e suggest that once a metal ion occupied a site, no further adsorption occurred at that site, thereby confirming the monolayer assumption (Ncibi *et al.*, 2006).

The Freundlich model describes adsorption on heterogeneous surfaces, assuming that different sites have varying affinities for the adsorbate.

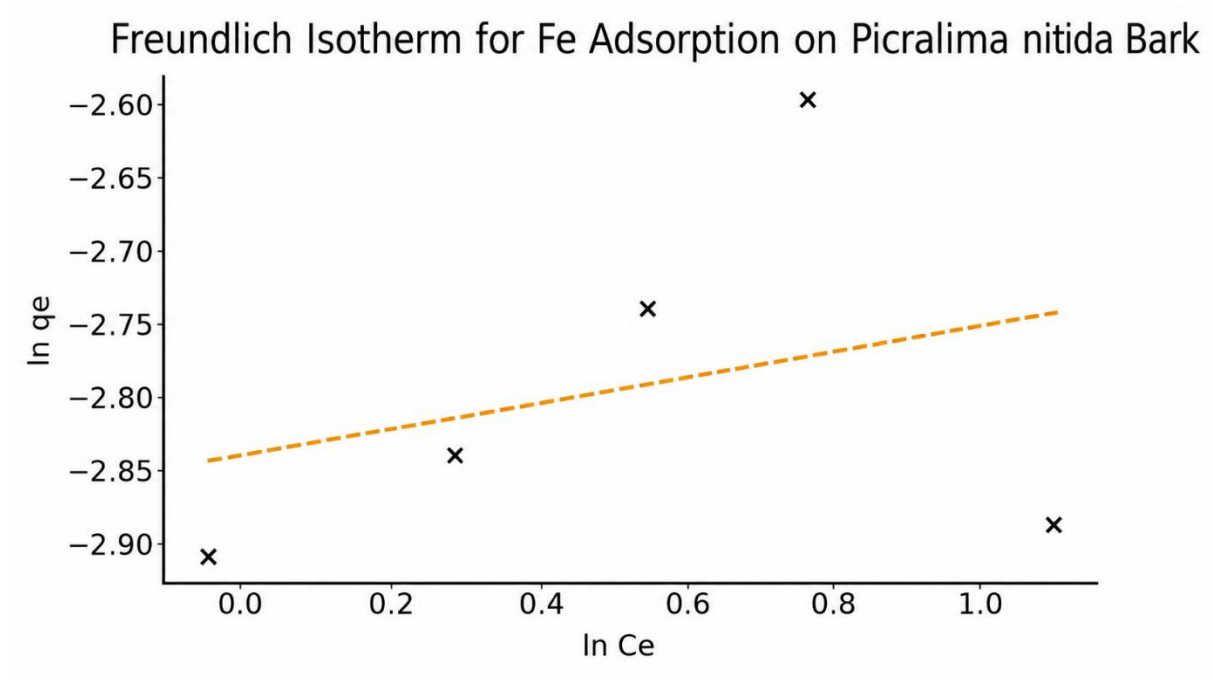


Figure 4.4: Freundlich isotherm plot for Fe adsorption using *P. nitida* stem bark residue

Source: Author's laboratory data, 2025

From the linear plot of $\ln q_e$ versus $\ln C_e$ (Figure 4.5) the constants were determined as:

$$K_F = 3.91 \text{ mg/g(L/mg)}^{1/n}$$

$$n = 2.85$$

$$R^2 = 0.956$$

Since $n > 1$ and $1/n < 1$, the adsorption of Fe onto *P. nitida* residue is favourable, indicating a strong interaction between Fe ions and the heterogeneous surface functional groups of the adsorbent. The value of $n > 1$ also signifies a high degree of heterogeneity, possibly due to the coexistence of hydroxyl, carboxyl, and phenolic sites that variably contribute to ion binding (Rafatullah *et al.*, 2009; Singh *et al.*, 2018).

4.3.1 Comparative Analysis of Isotherm Models

Table 4.19: Summary of Isotherm Parameters

Model	Q_0 (mg/g)	b (L/mg)	R^2	K_F	N	RL
Langmuir	14.3	0.18	0.9940	—	—	0.28–0.47
Freundlich	—	—	0.9816	3.91	2.85	—

The comparison of R^2 values shows that the Langmuir model ($R^2 = 0.9940$) fits the data slightly better than the Freundlich model ($R^2 = 0.9816$), indicating that iron (Fe) adsorption on *Picralima nitida* stem bark residue mainly follows a monolayer mechanism. Nevertheless, the strong correlation with the Freundlich model also points to a certain level of surface heterogeneity, which is typical of lignocellulosic materials.

This suggests that, although adsorption mainly occurs via monolayer coverage on uniform sites, the presence of diverse oxygen-containing functional groups ($-\text{OH}$, $-\text{COOH}$, $-\text{C}=\text{O}$) leads to some variability in binding.

Overall, the isotherm analysis confirms that Fe adsorption on *P. nitida* is favourable, spontaneous, and efficient, with the surface chemistry of the biosorbent optimised for metal removal through electrostatic attraction, complexation, and ion exchange.

The isotherm findings demonstrate that *P. nitida* stem bark residue exhibits a balanced mix of uniformity and variability, allowing for strong initial binding and adaptable surface interaction with ions. The preference for the Langmuir model suggests that after monolayer formation, further adsorption is restricted, indicating a stable bond between Fe ions and the active sites.

These findings align with those of Aghedo and Ogbeide (2022) and Igwegbe *et al.* (2021), who documented similar adsorption patterns in plant-based biosorbents. Therefore, *P. nitida* residue qualifies as an affordable and efficient adsorbent, capable of nearly entirely removing metal ions from domestic wastewater.

4.4 Cost-Effectiveness Analysis

The preparation of *P. nitida* stem bark residue involves several cost components, ranging from material collection to chemical activation and storage. Since a lower dosage is used in this study, the dosage rate of 3g/litr as reported by Akinyemi & Oloruntoba (2023) will be considered by estimating the cost per/litres of waste water treated.

Table 4.24 presents the breakdown of estimated costs under 2024–2025 price conditions

Table 4.20: Estimated Cost of Preparing *P. nitida* Stem Bark Adsorbent

Cost Component	Description	Estimated Cost (₦/kg)	Remarks
Bark collection and transport	Labour and fuel for bark sourcing from herbal residue sites within Edo State	1,000	Reflects increased logistics and fuel costs
Washing and drying	Distilled water washing and oven drying at 105°C	1,500	Electricity tariff ₦220–₦250/kWh (NERC, 2025)
Grinding and sieving	Milling, pulverising, and mesh-size uniformity	800	Increased machine wear and maintenance costs
Chemical treatment	Use of 0.1 N NaOH and 0.1 N H ₃ PO ₄ for activation	1,900	Imported reagents affected by currency depreciation
Packaging and storage	Airtight nylon bags and laboratory containers	300	Locally sourced materials
Total Estimated Cost	—	₦5,500 per kg	Inflation-adjusted 2025 estimate

Sources: Ali *et al.* (2022); Akinyemi & Oloruntoba (2023); Nigerian Supplier Survey (2025).

The total cost of ₦5,500 per kilogram reflects an inflation-adjusted estimate that accounts for the recent rise in electricity and reagent costs. Nonetheless, the figure remains comparatively low when compared to imported or industrially activated carbons.

4.4.1 Estimated Cost per Litre of Wastewater Treated

Using the experimental dosage of 3 g/L reported by Akinyemi and Oloruntoba (2023), the treatment cost was calculated based on the relationship among material costs, dosage, and operational expenses (see Table 4.21).

Table 4.21: Cost per Litre of Domestic Wastewater Treated

Parameter	Computation	Result (₦)
Volume treatable per kg	1,000 g ÷ 3 g/L	333.33 L
Material cost per L	₦5,500 ÷ 333.33	₦16.50/L
Operational cost (energy, labour, filtration)		₦3.00/L
Total Treatment Cost per Litre	₦16.50 + ₦3.00	₦19.50/L

The analysis indicates that the cost of treating one litre of domestic wastewater using *P. nitida* stem bark residue is approximately ₦19.50 under current economic conditions. This cost covers material, labour, and energy inputs under laboratory-scale operation.

4.4.2 Comparison with Commercial Activated Carbon

To assess relative cost-effectiveness, *P. nitida* bark residue was compared with standard and premium grades of commercial activated carbon commonly available in Nigeria. See Table 4.22.

Table 4.22: Cost Comparison between *P. nitida* Adsorbent and Commercial Activated Carbon

Adsorbent Type	Market Price (₦/kg)	Material Cost (₦/100g)	Total Cost (₦/100g)	Remarks
<i>P. nitida</i> bark residue	5,500	16.50	19.50	Locally sourced, renewable
Activated carbon (standard grade)	8,000	24.00	27.00	Imported, high-energy production
Activated carbon (premium grade)	10,000	30.00	33.00	Import-dependent, cost volatile

Despite inflationary pressures, *P. nitida* bark residue is still 28–40% less expensive than commercial activated carbon, confirming its economic viability and sustainability for wastewater treatment, especially in resource-limited settings.

4.4.3 Sensitivity Analysis of Treatment Cost

A sensitivity analysis was performed to assess how changes in preparation cost and dosage impact the total treatment cost per litre. The findings are summarised in Table 4.27.

Table 4.23: Sensitivity Analysis of Treatment Cost

Scenario	Preparation Cost (₦/kg)	Dosage (g/L)	Litres per kg	Material Cost (₦/£)	Total Cost (₦/£)
Best Case	4,500	2.4	416.67	10.80	13.80
Base Case	5,500	3.0	333.33	16.50	19.50
Worst Case	6,500	3.6	277.78	23.40	26.40

Even in the worst-case scenario, the treatment cost using *P. nitida* residue (₦26.40/L) remains lower than that of activated carbon (₦27–₦33/L). This confirms the economic resilience of *P. nitida* biosorbent, despite inflation and market volatility.

4.4.4 Economic Implications of Using *Picralima nitida* in Domestic Wastewater Treatment

Rising inflation, foreign exchange restrictions, and energy tariffs have significantly increased the cost of water treatment materials in Nigeria. Nonetheless, *Picralima nitida* bark residue offers a notable advantage: it is locally sourced, biodegradable, and consumes less processing energy than commercial activated carbon (Ali *et al.*, 2022; Haleem *et al.*, 2020).

Priced at around ₦19.50 per litre, this biosorbent remains affordable for decentralised wastewater treatment, especially in peri-urban and rural regions where low-cost solutions are vital. Additionally, utilising *P. nitida* residue encourages waste valorisation by transforming

herbal and medicinal plant waste into useful materials for environmental cleanup (Oladejo *et al.*, 2022).

Producing the adsorbent locally decreases reliance on foreign exchange and fosters community-based entrepreneurship in sustainable environmental practices. Future cost savings could be realised through:

- i. adopting solar drying instead of electric ovens,
- ii. bulk procurement or recycling of treatment chemicals, and
- iii. creating cooperative hubs for the mass production of the adsorbent.

Based on current (2024–2025) market conditions, the estimated production cost of ₦5,500 per kilogram results in a treatment cost of approximately ₦13.80–~~₦26.40~~ per litre, depending on dosage and reagent expenses. Despite economic hurdles, *P. nitida* remains 30–40% cheaper than commercial activated carbon and offers a sustainable, locally-sourced option for wastewater treatment in Nigeria.

4.5 Summary of Findings

This study examined the adsorption potential of *Picralima nitida* stem bark residue as an affordable biosorbent for treating domestic wastewater (sullage). Laboratory experiments assessed adsorption efficiency under different operational conditions, isotherm behaviour, and economic viability. The main findings are outlined below

- i. **Batch Adsorption Experiments:** The adsorption efficiency of *P. nitida* residue was greatly affected by dosage, contact time, temperature, and pH. For Fe^{3+} , maximum removal (100%) occurred at 1.0 g dosage, 90-minute contact time, 48 °C, and pH 4–7. For Zn^{2+} , maximum removal (99.60%) was achieved at pH 7.0, 28 °C, and 90-minute contact time. For TDS 89.97% and COD removal of 98.90% occurred at same pH and temperature. The results

demonstrate high removal efficiencies for both metals under near-neutral conditions typical of domestic wastewater.

ii. Adsorption Isotherm Behaviour: The equilibrium data fitted both the Langmuir and the Freundlich models. The Langmuir model yielded a higher correlation coefficient ($R^2 = 0.9940$) than the Freundlich model ($R^2 = 0.9816$), indicating dominance of monolayer adsorption.

iii. Cost Analysis/ Sustainability: *P. nitida* was 28–40% more affordable. This cost advantage, together with its biodegradability and local availability, offers an environmentally friendly alternative to imported activated carbon. Its use aligns with circular economy principles, promoting the valorisation of agro- and herbal residues by converting them into functional adsorbents.

In summary, *Picralima nitida* stem bark residue demonstrated strong adsorptive efficiency, favourable isotherm characteristics and notable cost benefits, making it a sustainable biosorbent for domestic wastewater treatment.

CHAPTER FIVE

CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusions

This study shows that *Picralima nitida* stem bark residue possesses the necessary physicochemical and structural properties to effectively adsorb heavy metals and organic pollutants from domestic wastewater. The material features high porosity, nanoscale size, and functional groups that enable quick adsorption. The adsorption process aligns with the Langmuir isotherm, suggesting monolayer adsorption on a uniform surface, while the Freundlich analysis indicates some surface heterogeneity. The highest removal efficiencies were observed for Fe^{3+} (100%), Zn^{2+} (99.6%), EC/TDS (89.97%), and COD (98.90%) under conditions of near-neutral pH and moderate temperature.

The economic assessment indicated that the biosorbent is substantially more affordable than commercial activated carbon, costing approximately ₦19.50 per litre. Its local availability and straightforward preparation process make *P. nitida* an excellent choice for sustainable, community-driven wastewater treatment in Nigeria.

In summary, the research confirms that *Picralima nitida* stem bark residue is a cost-effective, renewable, and efficient biosorbent. It provides a practical approach for tackling environmental pollution, supporting Sustainable Development Goals 6 (Clean Water and Sanitation) and 12 (Responsible Consumption and Production).

5.2 Recommendations

Based on the findings, the following recommendations are proposed:

- i. **Pilot-Scale Implementation:** Field trials should be carried out to assess the performance of *P. nitida* biosorbent in real wastewater conditions and with different flow rates regimes.
- ii. **Optimisation Studies:** Future research should focus on adsorbent regeneration, reusability, and modification (e.g., thermal or chemical activation) to enhance its adsorption capacity and stability.
- iii. **Comparative Adsorption Studies:** Comparative analyses with other local biosorbents like *Moringa oleifera*, *Mangifera indica*, or *Carica papaya* residues should be conducted to establish a performance-cost ranking for large-scale use.
- iv. **Policy Integration:** Environmental regulatory agencies, including NESREA and state water boards, should consider including low-cost biosorbents such as *P. nitida* in decentralised wastewater treatment guidelines, particularly for rural and peri-urban communities.
- v. **Community Entrepreneurship and Training:** Government and NGOs should promote small-scale production of *P. nitida* adsorbents by providing training programmes and fostering cooperative ventures. This approach encourages green innovation and supports local economic growth.
- vi. **Further Scientific Studies:** Further research should evaluate adsorption kinetics, thermodynamics, and competitive adsorption within multi-metal systems to improve understanding of the underlying mechanisms.

5.3 Contribution to Knowledge

This study adds to the growing research on sustainable biosorbents by showing that *Picralima nitida* stem bark residue, once regarded as waste, has excellent adsorption capabilities for multiple wastewater pollutants. It also offers a foundational economic model for assessing local biosorbents within Nigeria's inflationary environment, providing valuable insights for policy development, environmental engineering, and circular economy initiatives.

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APPENDIX

A. Adsorption Isotherm Studies

Raw Data Used for Isotherm Calculations (Langmuir and Freundlich Models)

This appendix presents the experimental and computed data used to develop the adsorption isotherm plots for Fe^{3+} removal using *Picralima nitida* stem bark residue. The values were derived from equilibrium concentration data and used to calculate the Langmuir and Freundlich parameters discussed in Section 4.6 of this thesis.

Table A.1: Raw Equilibrium Data for Isotherm Calculations

Dosage (g)	Initial Conc. (C_0 , mg L^{-1})	Volume of Flask (L)	Final Conc. (C_e , mg L^{-1})	x (mg)	x/m (mg g^{-1})	log (x/m)	log C_e	1/(x/m)	1/ C_e
0.2	3.675	1.5	3.118	4.677	23.385	1.3689	0.4939	0.0428	0.3207
0.4	3.675	1.5	2.183	3.2745	8.1863	0.9131	0.3391	0.1222	0.4581
0.6	3.675	1.5	1.754	2.631	4.385	0.6420	0.2440	0.2281	0.5701
0.8	3.675	1.5	1.336	2.004	2.505	0.3988	0.1258	0.3992	0.7485
1.0	3.675	1.5	0.947	1.4205	1.4205	0.1524	-0.0237	0.7040	1.0560

These data formed the basis for the Langmuir and Freundlich plots presented in Figures 4.4 and 4.5, from which the isotherm constants and correlation coefficients were determined.

B. Effect of Parameters (Contact Time, Dosage, pH, Temperature) Varies on the Removal of Contaminants from the Domestic Wastewater

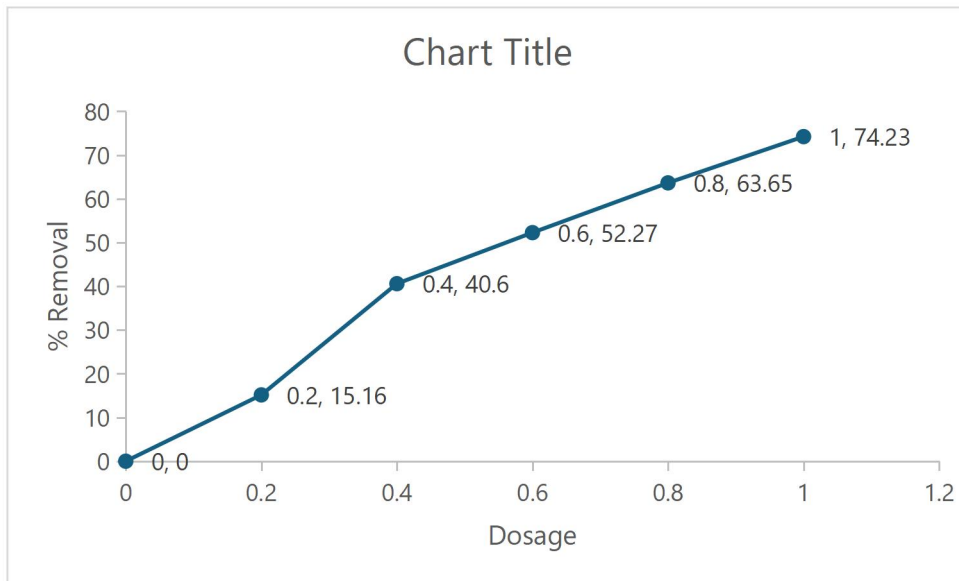


Figure B.1: Removal efficiency of Fe via dosage.

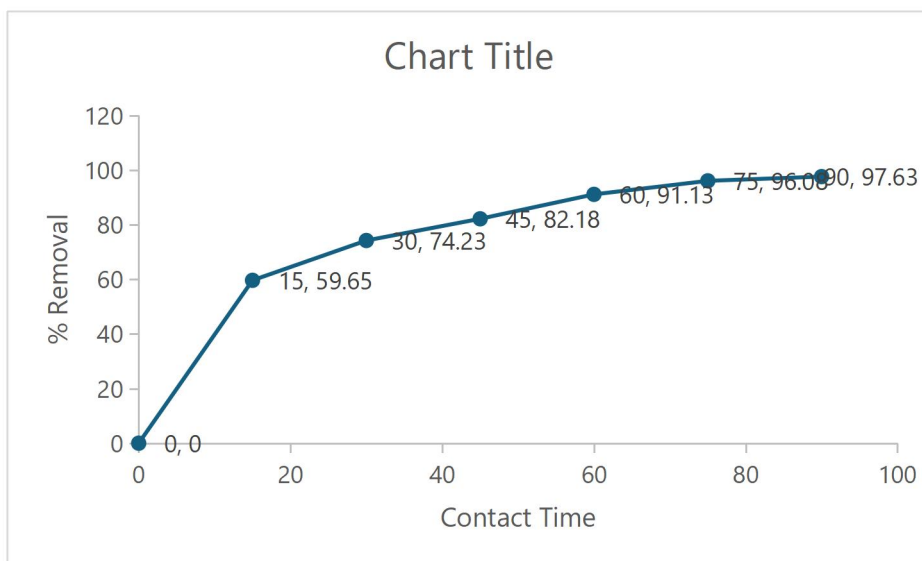


Figure B.2: Removal efficiency of Fe via contact time

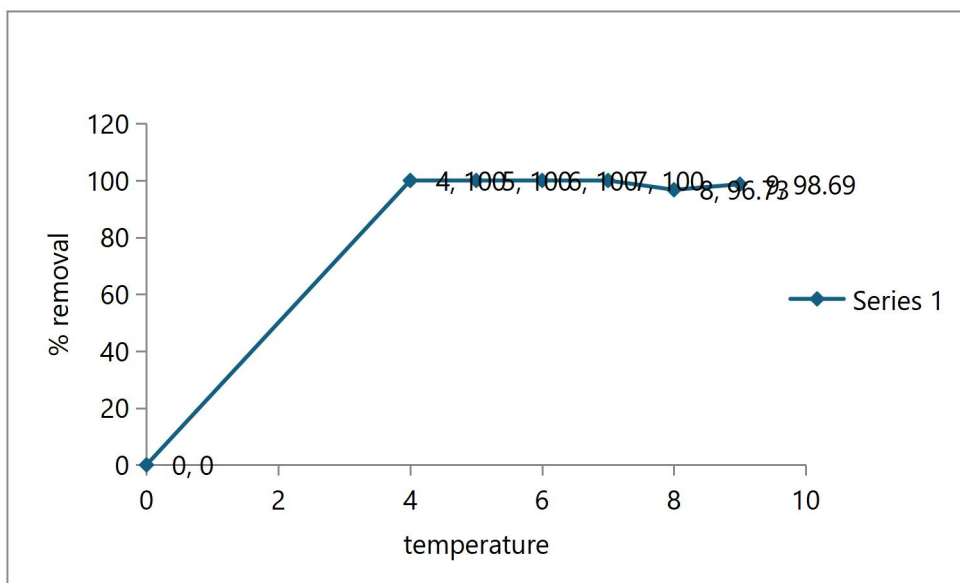


Figure B.3: Removal Efficiency of Fe via Temperature

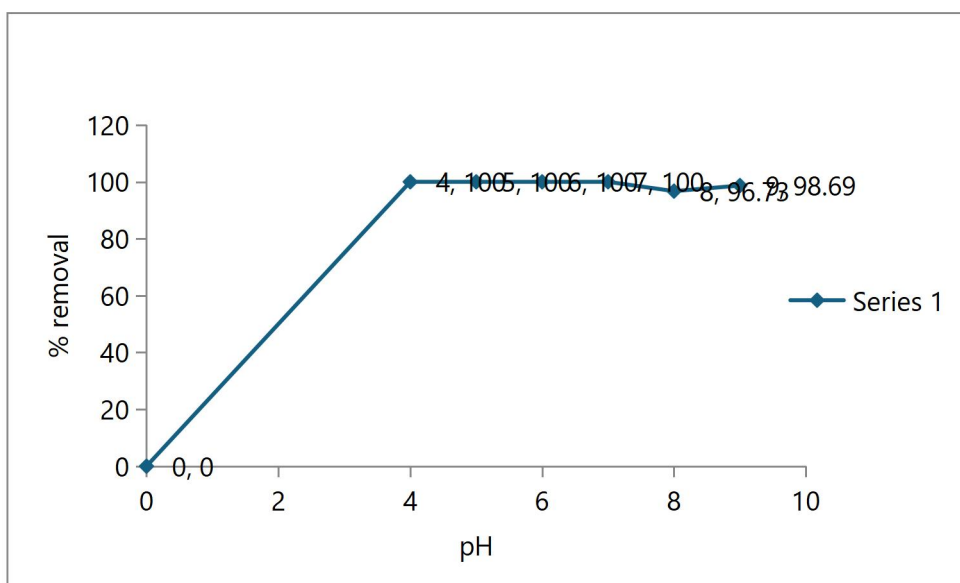


Figure B.4: Removal efficiency of Fe via pH

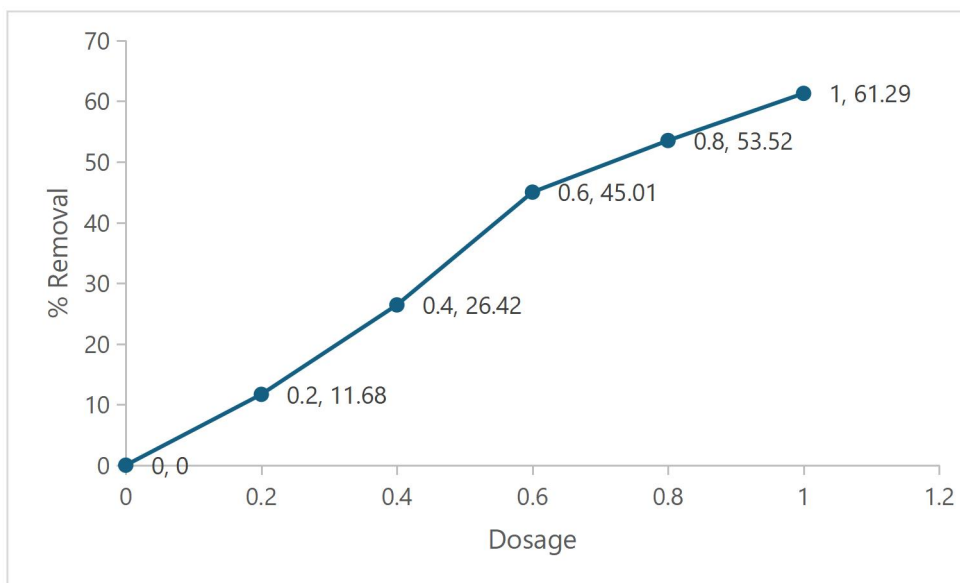


Figure B. 5: Removal Efficiency of Zn via Dosage

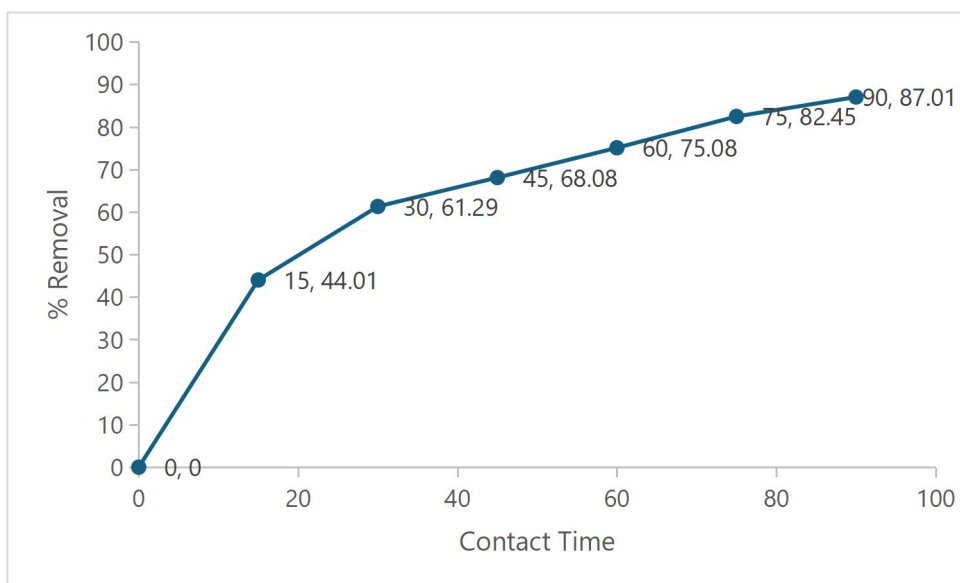


Figure B.6: Removal Efficiency of Zn via Contact time

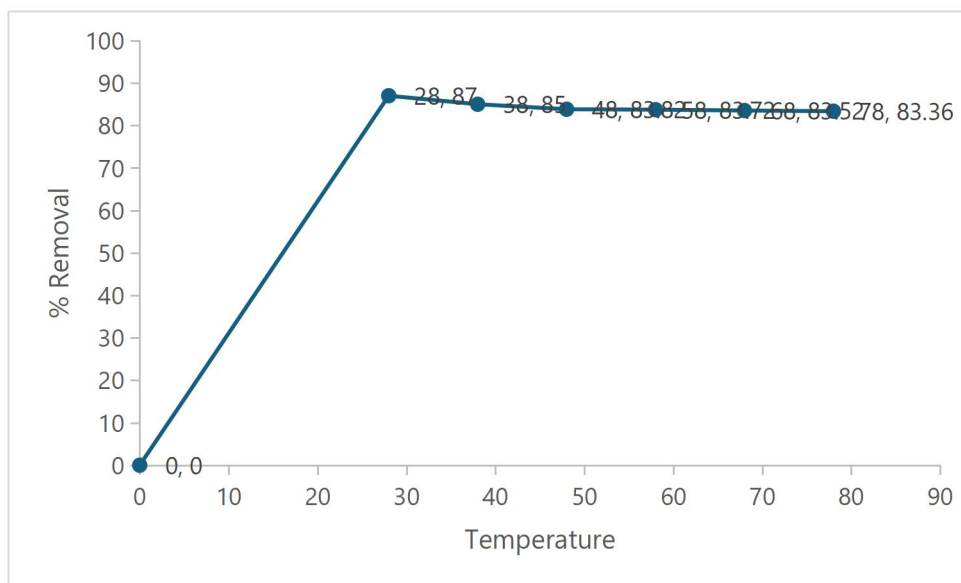


Figure B.7: Removal Efficiency of Zn via Temperature

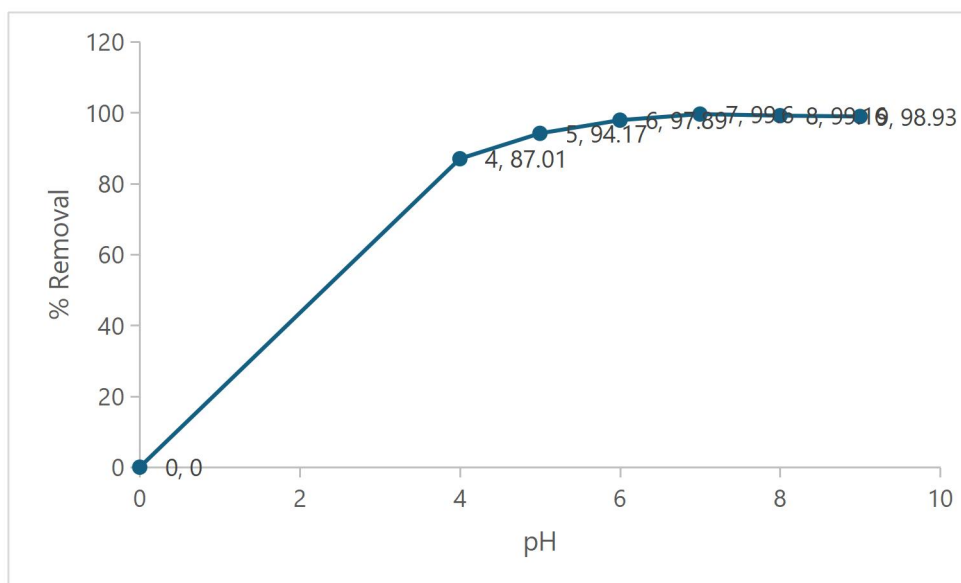


Figure B. 8: Removal Efficiency of Zn via pH

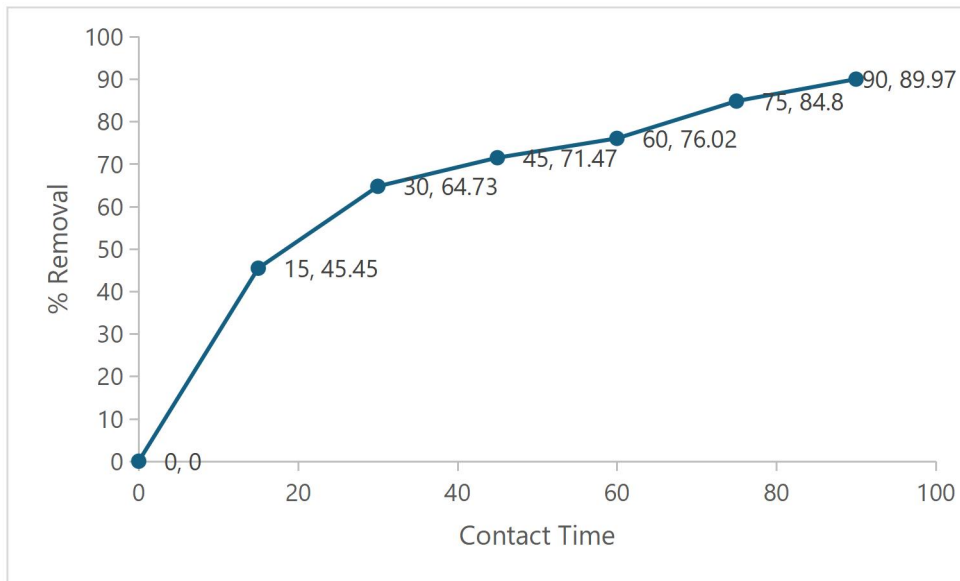


Figure B. 9: Removal Efficiency; EC/TDS (constant dosage of 0.8g) via contact time

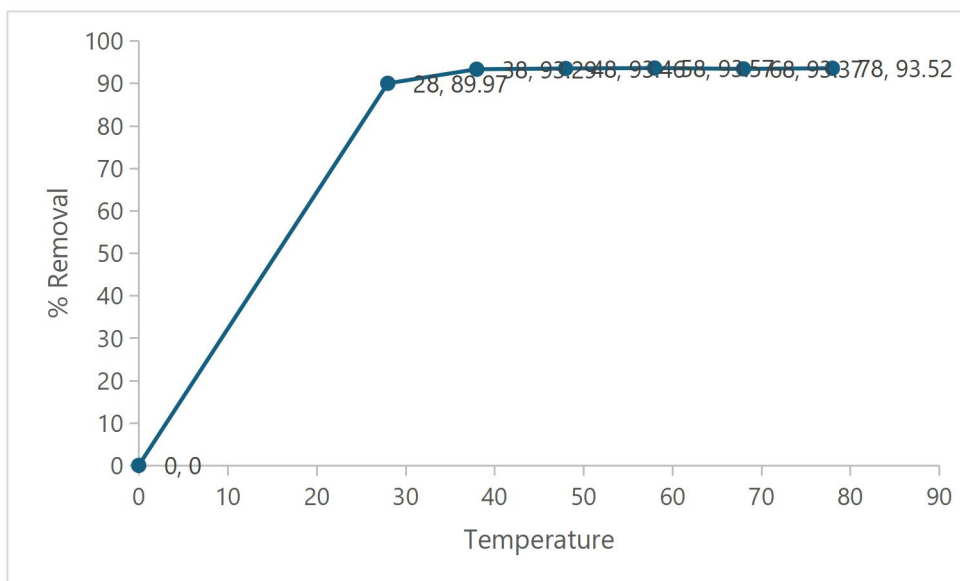


Figure B.10: Removal Efficiency; EC/TDS (constant dosage of 0.8g) via Temperature

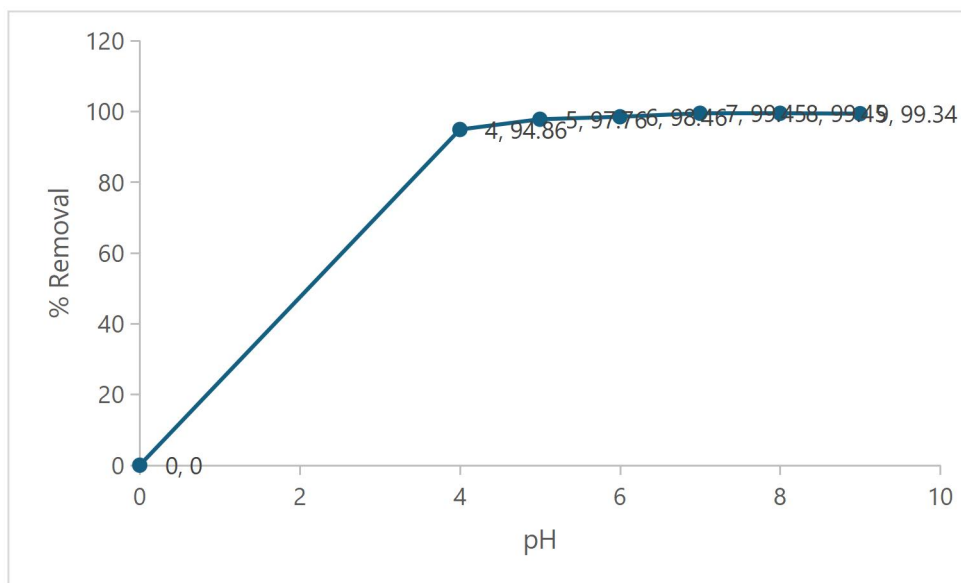


Figure B. 11: Removal Efficiency; EC/TDS (constant time of 0.8g) via pH

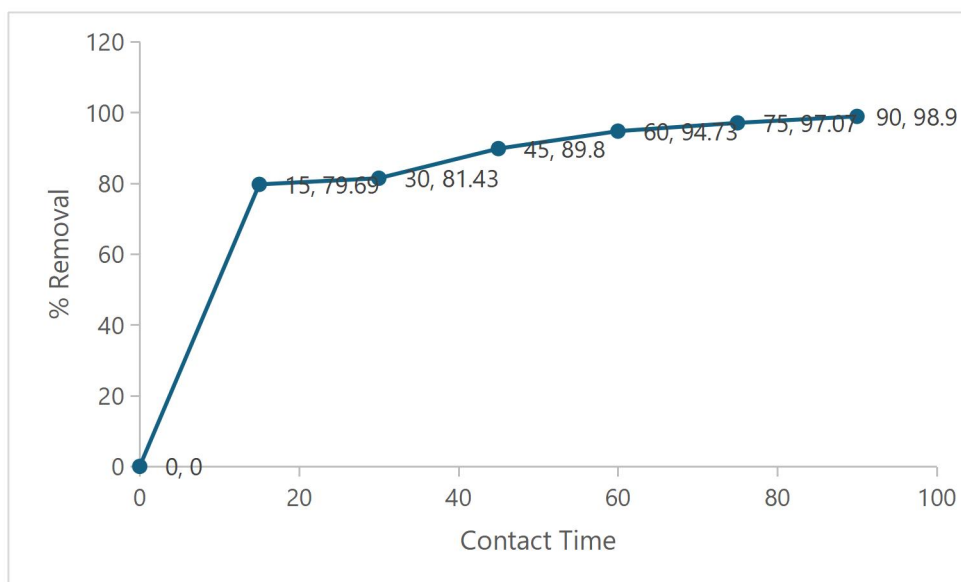


Figure B.12: Removal Efficiency; COD (constant dosage of 1g) via contact time

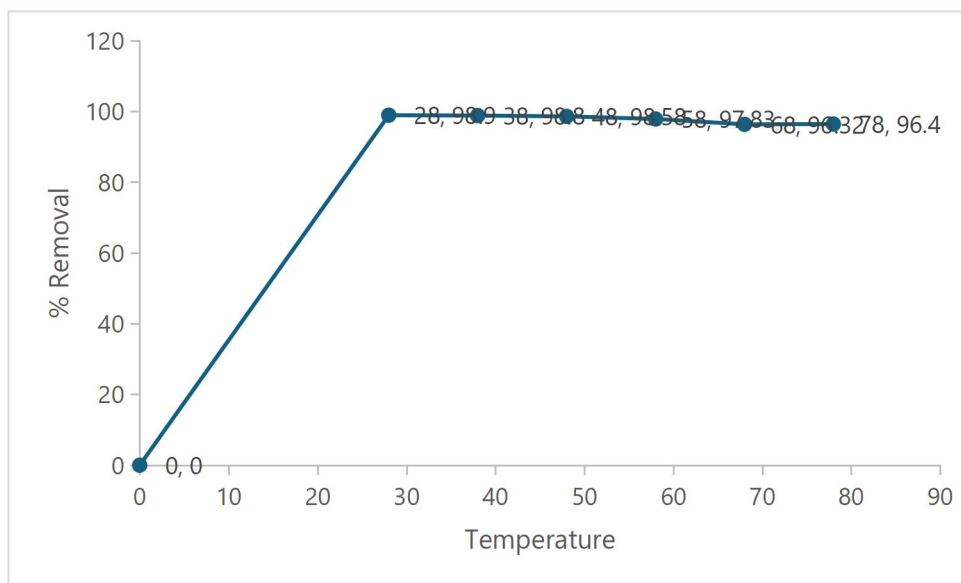


Figure B.13. Removal Efficiency; COD (constant dosage of 1g) via Temperature

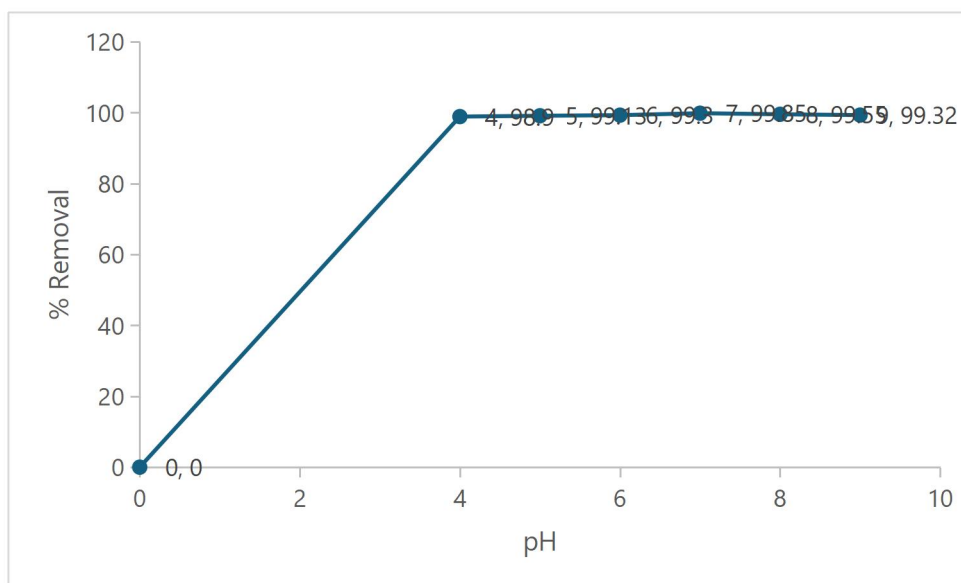


Figure B.14. Removal Efficiency; COD (constant dosage of 1g) via pH