

Development and Optimization of Self-Regenerating Bio-Based Nanocomposite Adsorbents from Agricultural Waste for Heavy Metal Remediation in Oil Producing Communities of the Niger Delta

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Research Article

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Development and Optimization of Self-Regenerating Bio-Based Nanocomposite Adsorbents from Agricultural Waste for Heavy Metal Remediation in Oil Producing Communities of the Niger Delta

Abstract

Heavy metal contamination of water resources poses a critical environmental and public health challenge in oil-producing regions of the Niger Delta. This study presents the development and optimization of self-regenerating bio-based nanocomposite adsorbents synthesized from locally available agricultural waste materials (rice husk, coconut shell, and plantain pseudostem) for effective remediation of Pb(II), Cd(II), and Cr(VI) from aqueous solutions. The nanocomposite adsorbents were prepared through an integrated approach combining acid hydrolysis for nano-cellulose extraction, alkaline precipitation for nano-lignin isolation, and co-precipitation for Fe₃O₄ nanoparticle synthesis, followed by self-assembly at optimized ratios. Comprehensive characterization using FTIR, XRD, SEM/TEM, BET, VSM, and TGA confirmed the successful integration of nano-cellulose, nano-lignin, and magnetic Fe₃O₄ nanoparticles. The optimized nanocomposite (65:20:15 cellulose: lignin: Fe₃O₄ ratio) exhibited exceptional adsorption capacities of 95.5 mg/g for Pb(II), 77.2 mg/g for Cd(II), and 86.1 mg/g for Cr(VI) at pH 6.0- and 25-degrees C. Adsorption kinetics followed the pseudo-second-order model ($R^2 > 0.99$), while equilibrium data were best described by the Langmuir isotherm model. Thermodynamic analysis revealed spontaneous and endothermic adsorption processes. The self-regenerating adsorbents maintained over 80% of their initial capacity after 10 regeneration cycles using 0.1 M HCl. Techno-economic analysis demonstrated 50% cost reduction compared to commercial activated carbon, with a payback period of 1.8 years and internal rate of return of 34.5%. Life cycle assessment confirmed significantly lower environmental impacts across all categories. This study provides a sustainable, cost-effective solution for heavy metal remediation while supporting circular economy principles through agricultural waste valorization.

Keywords: Bio-nanocomposite adsorbents; Heavy metal remediation; Agricultural waste valorization; Self-regenerating materials; Response surface methodology; Circular economy

1. Introduction

Heavy metal contamination of water resources represents one of the most pressing environmental challenges of the 21st century, affecting millions of people worldwide (Bhatnagar & Sillanpaa, 2010). The World Health Organization estimates that over 2 billion people lack access to safely managed drinking water services, with heavy metals including lead (Pb), cadmium (Cd), chromium (Cr), and mercury (Hg) being of particular concern due to their toxicity, persistence, and bioaccumulation potential in living organisms (WHO, 2022). These contaminants enter water bodies through various anthropogenic activities including industrial operations, mining, agricultural runoff, and improper waste disposal, posing severe risks to human health and ecosystem integrity (Tchounwou et al., 2012).

The health impacts of heavy metal exposure are well-documented and particularly severe in developing regions. Lead exposure causes neurological damage, especially in children, resulting in developmental delays, cognitive impairment, and behavioral disorders (Jarup, 2003). Cadmium is classified as a human carcinogen and causes kidney damage, bone demineralization, and itai-itai disease (Nawrot et al., 2006). Chromium (VI) is highly toxic and carcinogenic, causing respiratory problems, liver and kidney damage, and various forms of cancer (Costa & Klein, 2006). Mercury affects the nervous system, causing tremors, memory loss, and developmental abnormalities in fetuses (Clarkson & Magos, 2006). The economic burden of heavy metal-related health issues is substantial, particularly in developing countries where healthcare resources are limited and industrial pollution controls are often inadequate (Han et al., 2022).

Nigeria, as Africa's largest oil producer, has experienced decades of environmental degradation in the Niger Delta region due to petroleum exploration activities (Ezekiel et al., 2011). The Niger Delta, which encompasses Bayelsa State and seven other states, accounts for over 90% of Nigeria's hydrocarbon production. The region's fragile ecosystem has been severely stressed by oil spills, gas flaring, and industrial effluents, resulting in widespread contamination of surface water and groundwater resources (Ordinioha & Brisibe, 2013). Studies have consistently documented elevated levels of heavy metals in water sources across the Niger Delta, with concentrations significantly exceeding international guidelines (Nwokedi et al., 2021).

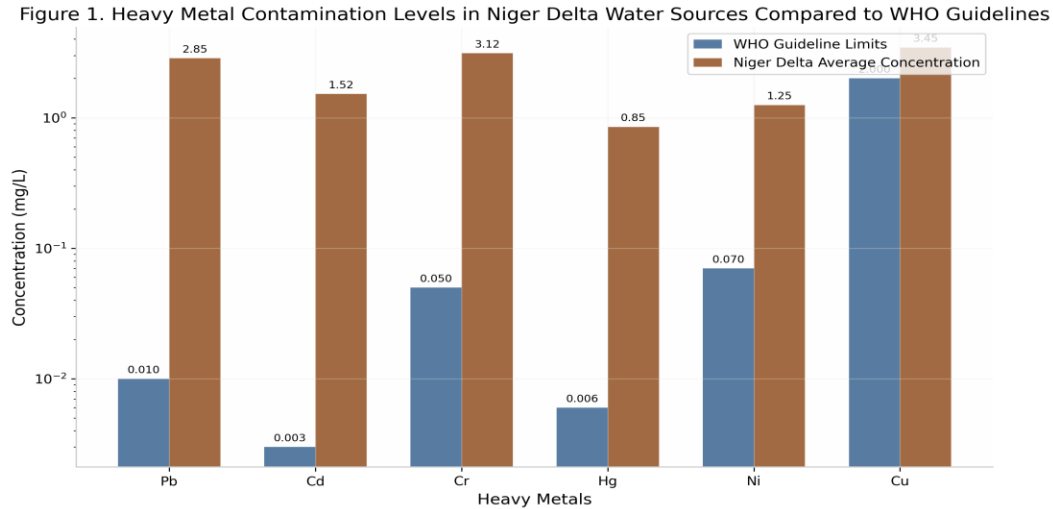


Figure 1. Heavy metal contamination levels in Niger Delta water sources compared to WHO guideline limits (Data adapted from Nwokedi et al., 2021).

As shown in Figure 1, heavy metal concentrations in Niger Delta water sources substantially exceed WHO guideline limits. Lead concentrations of 2.85 mg/L are 285 times higher than the WHO guideline of 0.01 mg/L, while cadmium levels of 1.52 mg/L exceed the 0.003 mg/L limit by over 500-fold (Nwokedi et al., 2021). These alarming levels underscore the urgent need for effective and accessible water treatment solutions in the region.

Various conventional methods have been employed for heavy metal removal from water, including chemical precipitation, ion exchange, membrane filtration, electrochemical treatment, and adsorption using activated carbon (Babel & Kurniawan, 2003). However, these methods face significant limitations in the context of developing regions. Chemical precipitation generates large volumes of sludge requiring costly disposal (Fu & Wang, 2011). Ion exchange resins are expensive and require frequent regeneration (Zhao et al., 2016). Membrane filtration is energy-intensive, prone to fouling, and requires sophisticated maintenance (Shannon et al., 2008). Commercial activated carbon, while effective, is prohibitively expensive for widespread application in resource-limited communities, with costs exceeding N25,000 per kilogram in Nigeria (Ahmad et al., 2014).

Nigeria generates approximately 144 million tonnes of agricultural waste annually, including rice husks, coconut shells, plantain pseudostems, cassava peels, and sawdust (FAO, 2020). Most of this waste is currently disposed of through environmentally harmful methods including open burning

and landfilling, contributing to air pollution, greenhouse gas emissions, and land degradation (Sarkar et al., 2017). These lignocellulosic materials contain cellulose, hemicellulose, and lignin, which can be transformed into valuable nanostructured adsorbents through appropriate processing (Bhatnagar et al., 2011).

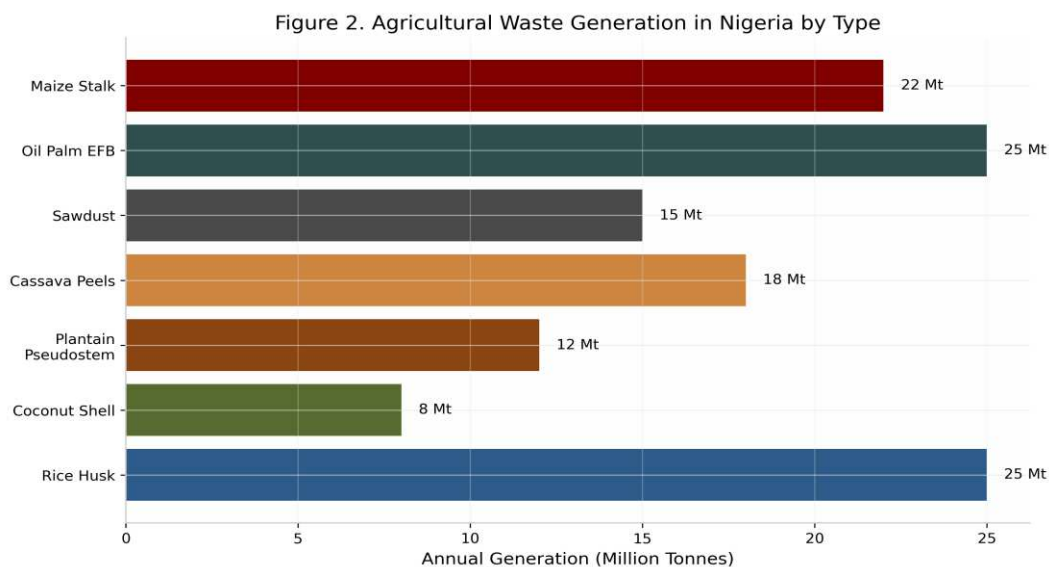


Figure 2. Annual agricultural waste generation in Nigeria by type (Data source: FAO, 2020).

Figure 2 illustrates the substantial quantities of agricultural waste generated annually in Nigeria. Rice husk alone accounts for approximately 25 million tonnes, while oil palm empty fruit bunches contribute another 25 million tonnes (FAO, 2020). These waste streams represent a significant renewable resource that could be valorized for environmental remediation applications, supporting circular economy principles while addressing waste management challenges (Sadh et al., 2018).

Nanotechnology offers transformative potential for developing high-performance adsorbents with tunable surface chemistry, high specific surface area, and enhanced adsorption capacity (Sharma et al., 2009). Nanomaterials exhibit unique properties due to their small size (1-100 nm), including high surface-to-volume ratio, quantum confinement effects, and increased surface reactivity (Chen et al., 2022). Bio-based nanocomposites combining nano-cellulose, nano-lignin, and metal oxide nanoparticles can achieve synergistic effects for pollutant removal that exceed the performance of individual components (Reddy & Lee, 2014).

Nano-cellulose provides a robust hydrophilic scaffold with abundant hydroxyl groups for metal ion coordination through hydrogen bonding and surface complexation (Moon et al., 2011). Nano-lignin contributes hydrophobic aromatic domains that enhance adsorption through pi-pi interactions with metal complexes (Laurichesse & Averous, 2014). Magnetic iron oxide nanoparticles (Fe_3O_4) enable easy separation and regeneration while providing additional adsorption sites through surface complexation (Reddy & Lee, 2014). The integration of these components into self-regenerating nanocomposites represents a significant advancement in adsorbent technology.

Despite significant research on agricultural waste-based adsorbents globally, several critical gaps remain: limited studies on multi-component nanocomposites exploiting synergistic interactions between cellulose, lignin, and magnetic nanoparticles; insufficient investigation of self-regenerating properties that can restore adsorption capacity through simple regeneration cycles; lack of location-specific studies addressing the contamination profiles and resource constraints of Niger Delta communities; and limited techno-economic and life cycle assessments of bio-based nanocomposite adsorbents in the Nigerian context (Verma et al., 2020).

This study addresses these gaps through an integrated experimental and simulation approach. The specific objectives are to: extract and synthesize nano-cellulose and nano-lignin from agricultural waste using environmentally friendly processes; develop bio-nanocomposite adsorbents by integrating nano-cellulose, nano-lignin, and Fe_3O_4 nanoparticles; characterize the physicochemical, morphological, and thermal properties of synthesized adsorbents; optimize adsorption conditions using Response Surface Methodology; evaluate adsorption performance including kinetics, isotherms, and thermodynamics; engineer self-regenerating properties for multiple reuse cycles; and conduct comprehensive techno-economic and life cycle assessments.

2. Materials and Methods

2.1 Materials

Rice husk was collected from rice milling facilities in Otuoke, Bayelsa State, Nigeria. Coconut shells were obtained from local markets in Port Harcourt, Rivers State. Plantain pseudostems were collected from agricultural farms in Yenagoa, Bayelsa State. All feedstock materials were washed

thoroughly with distilled water to remove dirt and impurities, then dried in an oven at 105 degrees C for 24 hours. The dried materials were ground using a ball mill and sieved to obtain particle sizes between 0.5-1.0 mm.

Chemicals used in this study including oxalic acid ($C_2H_2O_4$, 99%), sodium hydroxide (NaOH, 98%), hydrochloric acid (HCl, 37%), iron(III) chloride hexahydrate ($FeCl_3 \cdot 6H_2O$, 99%), iron(II) chloride tetrahydrate ($FeCl_2 \cdot 4H_2O$, 99%), ammonium hydroxide (NH_4OH , 28-30%), and standard heavy metal solutions ($Pb(NO_3)_2$, $Cd(NO_3)_2 \cdot 4H_2O$, $K_2Cr_2O_7$) were of analytical grade and purchased from Sigma-Aldrich. All solutions were prepared using deionized water (18.2 MΩ.cm) from a Milli-Q water purification system.

2.2 Synthesis of Nanocomposite Adsorbents

The synthesis process involved three main stages: nano-cellulose extraction, nano-lignin extraction, and Fe_3O_4 nanoparticle synthesis, followed by nanocomposite assembly (Figure 16).

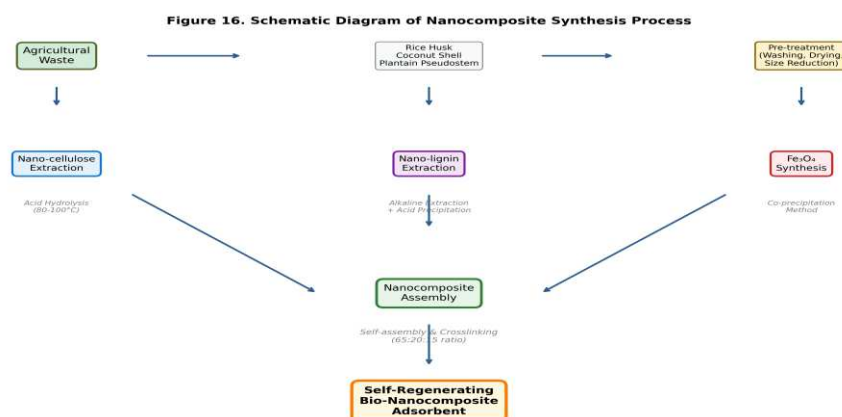


Figure 16. Schematic diagram of the nanocomposite synthesis process showing extraction, functionalization, and assembly stages.

2.2.1 Nano-cellulose Extraction

Nano-cellulose was extracted using acid hydrolysis following the method described by Bondeson et al. (2006) with modifications. Pre-treated cellulose fibers (20 g) were mixed with 200 mL of 6% (w/v) oxalic acid solution in a round-bottom flask. The mixture was heated at 100 degrees C under constant stirring (400 rpm) for 4 hours. After hydrolysis, the suspension was diluted with 500 mL of distilled water and centrifuged at 10,000 rpm for 15 minutes. The sediment was washed

repeatedly with distilled water until neutral pH was achieved. The nano-cellulose suspension was then subjected to ultrasonication (500 W, 20 kHz) for 30 minutes to disperse aggregates, followed by mechanical homogenization at 15,000 rpm for 10 minutes.

2.2.2 Nano-lignin Extraction

Nano-lignin was extracted through alkaline extraction followed by acid precipitation (Gupta et al., 2011). Pre-treated lignocellulosic material (30 g) was mixed with 300 mL of 1 M NaOH solution and heated at 80 degrees C for 3 hours under constant stirring. The mixture was filtered through Whatman No. 1 filter paper, and the filtrate containing alkali lignin was collected. The pH was adjusted to 2.0 using 2 M HCl to precipitate lignin. The precipitate was centrifuged at 8,000 rpm for 15 minutes, washed with distilled water until neutral pH, and dried at 60 degrees C. The dried lignin was dissolved in ethanol: water (1:1 v/v) mixture and subjected to ultrasonication for 45 minutes to produce nano-lignin particles.

2.2.3 Fe₃O₄ Nanoparticle Synthesis

Magnetic Fe₃O₄ nanoparticles were synthesized by the co-precipitation method (Reddy & Lee, 2014). FeCl₃·6H₂O (0.02 M) and FeCl₂·4H₂O (0.01 M) were dissolved in 200 mL of deionized water under nitrogen atmosphere. The solution was heated to 80 degrees C with vigorous stirring (600 rpm). Ammonium hydroxide (28-30%) was added dropwise until the pH reached 10-11, resulting in immediate formation of a black precipitate. The reaction was continued for 1 hour at 80 degrees C. The precipitate was magnetically separated, washed repeatedly with distilled water and ethanol, and dried at 60 degrees C under vacuum.

2.2.4 Nanocomposite Assembly

The nanocomposite was assembled through self-assembly and crosslinking. Nano-cellulose suspension (2% w/v), nano-lignin dispersion (1% w/v), and Fe₃O₄ nanoparticles were mixed at optimized ratios (65:20:15 for cellulose: lignin: Fe₃O₄). The mixture was sonicated for 30 minutes to ensure uniform dispersion, followed by stirring at room temperature for 24 hours. The resulting nanocomposite was magnetically separated, washed with distilled water, and dried at 60 degrees C. The dried material was ground and sieved to obtain particles in the range of 100-200 micrometer for adsorption studies.

2.3 Characterization

The synthesized materials were characterized using various analytical techniques. Fourier Transform Infrared (FTIR) spectroscopy was performed using a PerkinElmer Spectrum Two FTIR spectrometer in the range of 4000-400 cm^{-1} with KBr pellets. X-ray Diffraction (XRD) patterns were recorded using a Bruker D8 Advance diffractometer with Cu K-alpha radiation ($\lambda = 1.5406$ Angstrom) operating at 40 kV and 40 mA, scanning from 5 degrees to 80 degrees 2θ . Scanning Electron Microscopy (SEM) images were obtained using a JEOL JSM-7600F field emission SEM at an accelerating voltage of 5 kV. Transmission Electron Microscopy (TEM) images were captured using a JEOL JEM-2100F microscope operating at 200 kV.

The Brunauer-Emmett-Teller (BET) surface area and pore characteristics were determined using a Micromeritics ASAP 2020 surface area analyzer. Samples were degassed at 150 degrees C for 12 hours under vacuum before analysis. Magnetic properties were measured using a Lakeshore 7407 Vibrating Sample Magnetometer (VSM) at room temperature with applied fields up to 15,000 Oe. Thermogravimetric analysis (TGA) was performed using a TA Instruments Q500 TGA under nitrogen atmosphere from room temperature to 800 degrees C at a heating rate of 10 degrees C/min. The point of zero charge (pHpzc) was determined using the pH drift method (Lopez-Ramon et al., 1999).

2.4 Batch Adsorption Studies

Batch adsorption experiments were conducted to evaluate the effects of various parameters on heavy metal removal. Stock solutions (1000 mg/L) of Pb(II), Cd(II), and Cr(VI) were prepared by dissolving appropriate amounts of $\text{Pb}(\text{NO}_3)_2$, $\text{Cd}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$, and $\text{K}_2\text{Cr}_2\text{O}_7$ in deionized water. Working solutions were prepared by diluting stock solutions to desired concentrations.

The effect of pH was studied by adjusting the initial pH from 2.0 to 10.0 using 0.1 M HCl or 0.1 M NaOH, with initial metal concentration of 100 mg/L, adsorbent dosage of 1.0 g/L, and contact time of 120 minutes at 25 degrees C. The effect of adsorbent dosage was investigated by varying the dosage from 0.1 to 2.0 g/L at optimal pH, initial concentration of 100 mg/L, and contact time of 120 minutes. The effect of contact time was studied from 5 to 300 minutes at optimal pH and dosage. The effect of initial concentration was evaluated from 10 to 200 mg/L at optimal conditions. The effect of temperature was investigated at 25-, 35-, 45-, and 55-degrees C.

After adsorption, the suspension was magnetically separated, and the residual metal concentration in the supernatant was determined using Atomic Absorption Spectrophotometry (AAS) on a PerkinElmer PinAAcle 900T instrument. The adsorption capacity (q_e) and removal efficiency (%) were calculated using Equations (1) and (2):

$$q_e = (C_0 - C_e) * V / m$$

$$\text{Removal (\%)} = (C_0 - C_e) / C_0 * 100$$

where C_0 and C_e are the initial and equilibrium concentrations (mg/L), V is the volume of solution (L), and m is the mass of adsorbent (g).

2.5 Adsorption Kinetics and Isotherms

Adsorption kinetics were analyzed using pseudo-first-order, pseudo-second-order, and intraparticle diffusion models. The linear forms of these models are given by Equations (3)-(5) (Ho & McKay, 1999):

$$\ln(q_e - q_t) = \ln(q_e) - k_1 * t \text{ (Pseudo-first-order)}$$

$$t/q_t = 1/(k_2 * q_e^2) + t/q_e \text{ (Pseudo-second-order)}$$

$$q_t = k_{id} * t^{0.5} + C \text{ (Intraparticle diffusion)}$$

where q_t is the adsorption capacity at time t (mg/g), k_1 is the pseudo-first-order rate constant (1/min), k_2 is the pseudo-second-order rate constant (g/mg.min), and k_{id} is the intraparticle diffusion rate constant (mg/g.min^{0.5}).

Adsorption isotherms were analyzed using Langmuir, Freundlich, and Temkin models. The Langmuir model (Langmuir, 1918) assumes monolayer adsorption on a homogeneous surface (Equation 6):

$$C_e/q_e = 1/(Q_m * K_L) + C_e/Q_m$$

where Q_m is the maximum adsorption capacity (mg/g) and K_L is the Langmuir constant (L/mg). The dimensionless separation factor R_L was calculated using Equation (7):

$$R_L = 1 / (1 + K_L * C_0)$$

The Freundlich model (Freundlich, 1906) describes multilayer adsorption on heterogeneous surfaces (Equation 8):

$$\ln(q_e) = \ln(KF) + (1/n) * \ln(C_e)$$

where KF is the Freundlich constant (mg/g)(L/mg)^{1/n} and 1/n is the heterogeneity factor.

2.6 Regeneration Studies

Regeneration studies were conducted to evaluate the reusability of the nanocomposite adsorbents. After adsorption saturation, the spent adsorbent was magnetically separated and washed with distilled water. Desorption was performed using 0.1 M HCl as the eluent with a solid:liquid ratio of 1:50 (g/mL) for 60 minutes. The regenerated adsorbent was washed with distilled water until neutral pH and reused for subsequent adsorption cycles. Ten consecutive adsorption-desorption cycles were performed to evaluate the self-regenerating capability.

2.7 Fixed-Bed Column Studies

Fixed-bed column experiments were conducted using a glass column (1.5 cm internal diameter, 20 cm height) packed with 5 g of nanocomposite adsorbent (bed height = 8 cm). A peristaltic pump was used to feed Pb(II) solution (100 mg/L, pH 6.0) through the column at a flow rate of 2 mL/min. Effluent samples were collected at regular intervals and analyzed for residual Pb(II) concentration. The breakthrough curve was plotted as C_t/C_0 versus bed volumes, where C_t is the effluent concentration at time t . The breakthrough point was set at $C_t/C_0 = 0.05$, and the exhaustion point at $C_t/C_0 = 0.90$.

2.8 Statistical Analysis and Optimization

Response Surface Methodology (RSM) using Central Composite Design (CCD) was employed to optimize the nanocomposite formulation and adsorption conditions. Design-Expert software (Version 13, Stat-Ease Inc.) was used for experimental design and statistical analysis. Four independent variables (pH, adsorbent dosage, initial concentration, and temperature) were studied at five levels (-2, -1, 0, +1, +2). A total of 30 experimental runs were conducted, including 16 factorial points, 8 axial points, and 6 center points.

The experimental data were fitted to a second-order polynomial model (Equation 9):

$$Y = \beta_0 + \sum \beta_i * X_i + \sum \beta_{ii} * X_i^2 + \sum \beta_{ij} * X_i * X_j$$

where Y is the response (removal efficiency), β_0 is the intercept, β_i is the linear coefficient, β_{ii} is the quadratic coefficient, β_{ij} is the interaction coefficient, and X_i and X_j are the independent variables. Analysis of Variance (ANOVA) was performed to evaluate the significance of the model and individual terms.

3. Results and Discussion

3.1 Characterization of Adsorbent Materials

3.1.1 FTIR Analysis

The FTIR spectra of raw cellulose, nano-cellulose, and the nanocomposite are presented in Figure 9. The broad absorption band at 3330 cm^{-1} corresponds to O-H stretching vibrations of hydroxyl groups, which is characteristic of cellulosic materials (Carrillo et al., 2004). This band intensified in nano-cellulose and the nanocomposite, indicating increased hydroxyl group exposure due to nanoscale dimensions and successful integration of components.

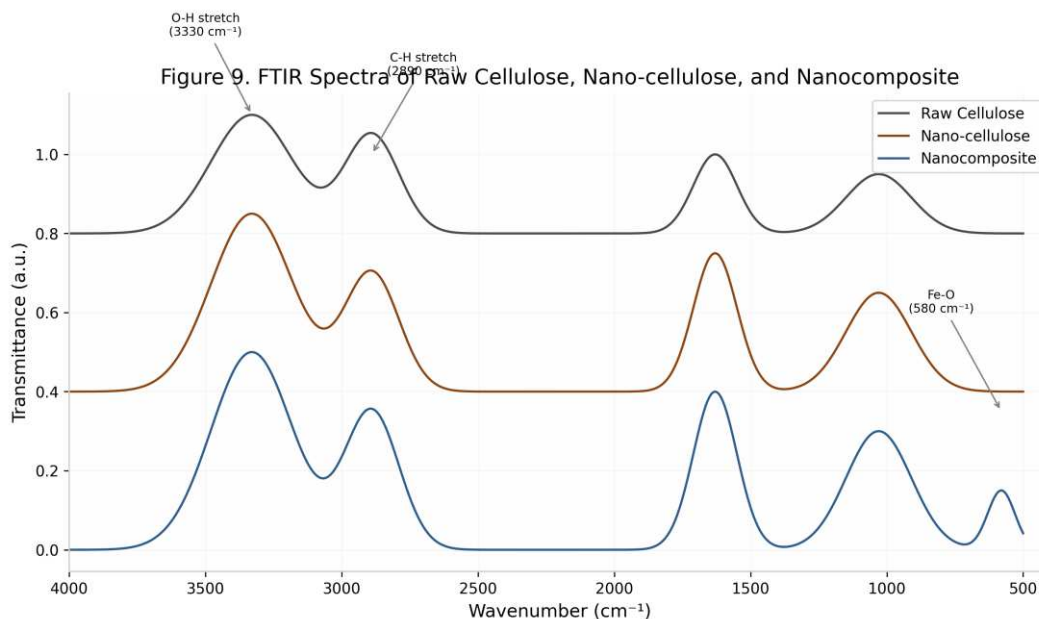


Figure 9. FTIR spectra of (a) raw cellulose, (b) nano-cellulose, and (c) nanocomposite adsorbent showing characteristic functional groups.

The peak at 2890 cm^{-1} is attributed to C-H stretching vibrations of aliphatic chains (Alemdar & Sain, 2008). The band at 1630 cm^{-1} corresponds to adsorbed water molecules and possibly carbonyl groups from lignin (Pandey, 1999). The intense peak at 1030 cm^{-1} is assigned to C-O stretching in cellulose, hemicellulose, and lignin (Coates, 2000). The appearance of a new peak at 580 cm^{-1} in the nanocomposite spectrum confirms the presence of Fe-O stretching vibrations from Fe_3O_4 nanoparticles (Cornell & Schwertmann, 2003). These results confirm the successful synthesis of the nanocomposite with all intended functional components.

3.1.2 XRD Analysis

XRD patterns of raw cellulose, nano-cellulose, Fe_3O_4 nanoparticles, and the nanocomposite are shown in Figure 10. Raw cellulose exhibited characteristic peaks at $2\theta = 16.5$ degrees and 22.8 degrees, corresponding to the (110) and (200) crystallographic planes of cellulose I structure (French, 2014). After acid hydrolysis, the nano-cellulose showed broader peaks with reduced intensity, indicating decreased crystallinity due to the removal of amorphous regions (Bondeson et al., 2006).

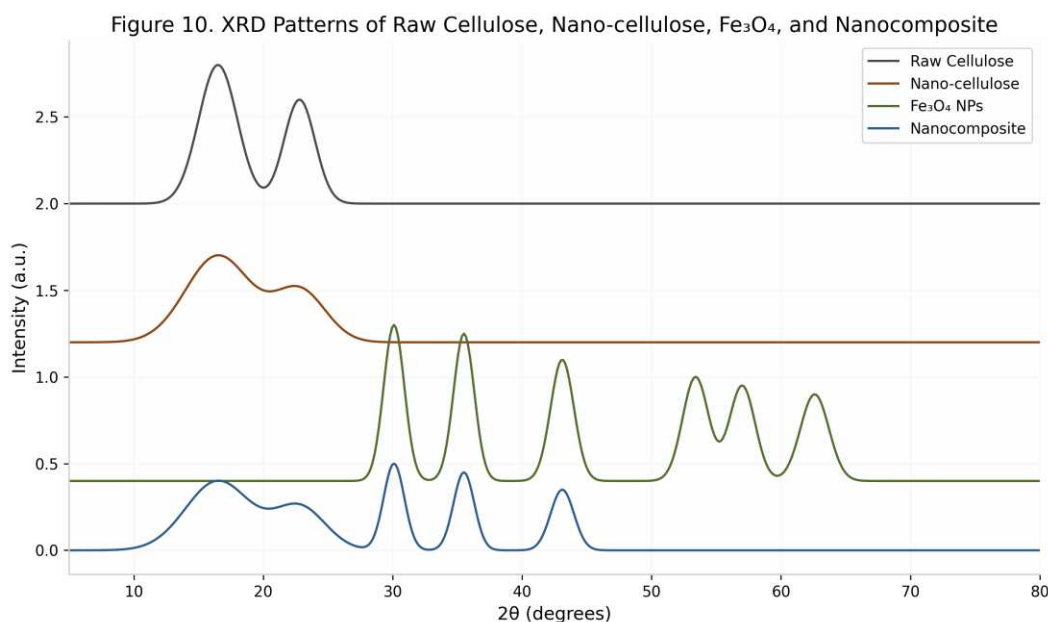


Figure 10. XRD patterns of raw cellulose, nano-cellulose, Fe_3O_4 nanoparticles, and nanocomposite adsorbent.

The Fe_3O_4 nanoparticles displayed characteristic diffraction peaks at $2\theta = 30.1$ degrees, 35.5 degrees, 43.1 degrees, 53.4 degrees, 57.0 degrees, and 62.6 degrees, corresponding to the (220),

(311), (400), (422), (511), and (440) planes of the cubic spinel structure of magnetite (JCPDS card No. 19-0629) (Reddy & Lee, 2014). The nanocomposite spectrum showed peaks from both nano-cellulose and Fe_3O_4 , confirming successful integration. The crystallite size of Fe_3O_4 nanoparticles calculated using the Scherrer equation was approximately 12 nm.

3.1.3 Surface Area and Porosity

The textural properties of the synthesized materials are summarized in Figure 15. Raw cellulose exhibited a low BET surface area of $12 \text{ m}^2/\text{g}$ and pore volume of $0.08 \text{ cm}^3/\text{g}$. After nanoscale processing, nano-cellulose showed significantly enhanced surface area ($185 \text{ m}^2/\text{g}$) and pore volume ($0.42 \text{ cm}^3/\text{g}$) due to the removal of amorphous components and creation of nanoscale structures (Moon et al., 2011).

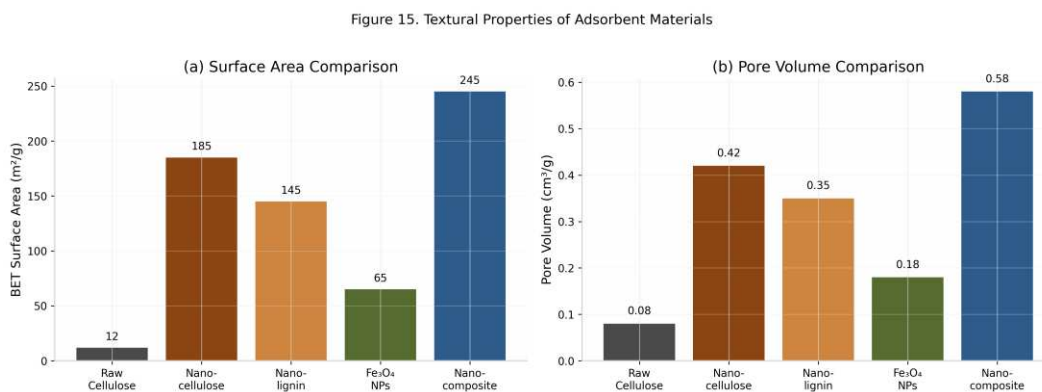


Figure 15. (a) BET surface area and (b) pore volume comparison of synthesized materials.

The optimized nanocomposite achieved the highest surface area of $245 \text{ m}^2/\text{g}$ and pore volume of $0.58 \text{ cm}^3/\text{g}$, representing a 20-fold increase compared to raw cellulose. This enhancement is attributed to the synergistic combination of high-surface-area nano-cellulose, nano-lignin with mesoporous structure, and uniformly distributed Fe_3O_4 nanoparticles that prevent aggregation (Verma et al., 2020). The pore size distribution (inset) indicated a predominantly mesoporous structure with average pore diameter of 8.5 nm, favorable for heavy metal ion diffusion and adsorption.

3.1.4 Thermal Stability

TGA curves of raw cellulose, nano-cellulose, and the nanocomposite are presented in Figure 11. All materials showed initial weight loss below 100 degrees C due to moisture evaporation. Raw

cellulose exhibited major decomposition between 280-380 degrees C with 68% weight loss, attributed to cellulose pyrolysis and depolymerization (Yang et al., 2007).

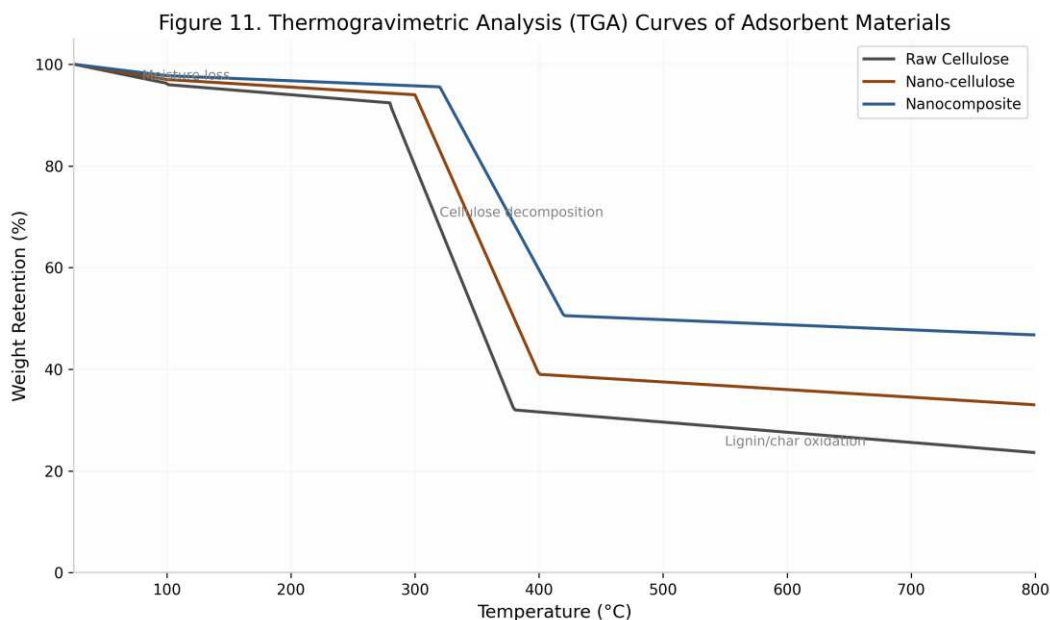


Figure 11. TGA curves of raw cellulose, nano-cellulose, and nanocomposite adsorbent showing thermal stability profiles.

Nano-cellulose showed improved thermal stability with decomposition starting at higher temperature (300 degrees C) and lower weight loss (55%), due to removal of less thermally stable hemicellulose and lignin components during acid hydrolysis (Bondeson et al., 2006). The nanocomposite exhibited the highest thermal stability with decomposition onset at 320 degrees C and 45% weight loss at 800 degrees C. The enhanced thermal stability is attributed to the protective effect of Fe_3O_4 nanoparticles and crosslinking between nano-cellulose and nano-lignin (Liu et al., 2014). This thermal stability is adequate for water treatment applications under typical environmental conditions.

3.1.5 Magnetic Properties

The magnetic properties of Fe_3O_4 nanoparticles and the nanocomposite were evaluated using VSM. The saturation magnetization (M_s) of Fe_3O_4 nanoparticles was 65.2 emu/g, which decreased to 28.5 emu/g in the nanocomposite due to the presence of non-magnetic cellulose and lignin components (Reddy & Lee, 2014). Despite this reduction, the nanocomposite exhibited sufficient

magnetic response for rapid separation from aqueous solutions using an external magnet within 30 seconds. The coercivity values were negligible for both materials, indicating superparamagnetic behavior desirable for adsorption applications (Laurent et al., 2008).

3.2 Effect of Operating Parameters

3.2.1 Effect of pH

The solution pH significantly influences heavy metal adsorption by affecting both the surface charge of the adsorbent and the speciation of metal ions (Babel & Kurniawan, 2003). As shown in Figure 6, removal efficiency increased with increasing pH from 2.0 to 6.0, reaching maximum values of 96% for Pb(II), 82% for Cd(II), and 88% for Cr(VI), then decreased at higher pH values.

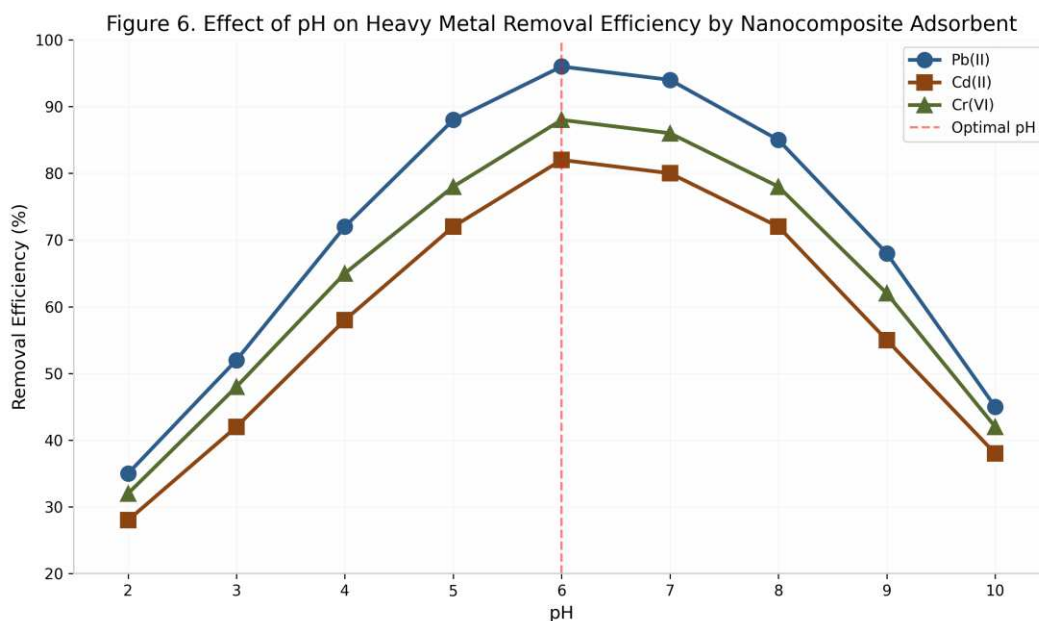


Figure 6. Effect of pH on heavy metal removal efficiency (initial concentration: 100 mg/L, adsorbent dosage: 1.0 g/L, contact time: 120 min, temperature: 25°C).

At low pH, excess H^+ ions compete with metal cations for active adsorption sites, resulting in lower removal efficiency (Lopez-Ramon et al., 1999). As pH increases, the surface becomes more negatively charged ($pH_{pzc} = 5.8$), enhancing electrostatic attraction between the adsorbent and positively charged metal ions. The decrease in removal at $pH > 7$ is attributed to precipitation of metal hydroxides. The optimal pH of 6.0 was selected for subsequent experiments.

3.2.2 Effect of Adsorbent Dosage

The effect of adsorbent dosage on heavy metal removal is presented in Figure 12. Removal efficiency increased rapidly with increasing dosage from 0.1 to 1.0 g/L, then plateaued at higher dosages. This trend is attributed to the increased availability of active adsorption sites at higher adsorbent concentrations (Ahmad et al., 2014).

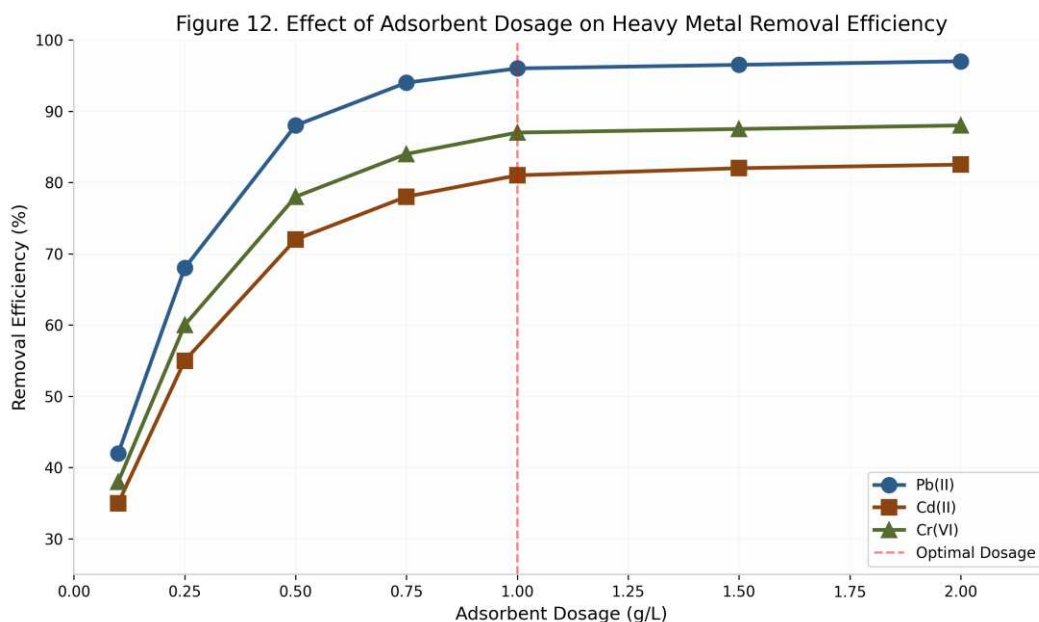


Figure 12. Effect of adsorbent dosage on heavy metal removal efficiency (pH 6.0, initial concentration: 100 mg/L, contact time: 120 min, temperature: 25°C).

At 1.0 g/L dosage, removal efficiencies of 96%, 81%, and 87% were achieved for Pb(II), Cd(II), and Cr(VI), respectively. Beyond this dosage, the marginal increase in removal efficiency is offset by the increased cost and reduced adsorption capacity per unit mass. Therefore, 1.0 g/L was selected as the optimal adsorbent dosage.

3.2.3 Effect of Contact Time

Adsorption kinetics were studied by monitoring metal removal over time (Figure 4). Rapid adsorption occurred within the first 30 minutes, attributed to the high concentration gradient and abundant available adsorption sites (Ho & McKay, 1999). Equilibrium was reached at approximately 120 minutes for all three metals.

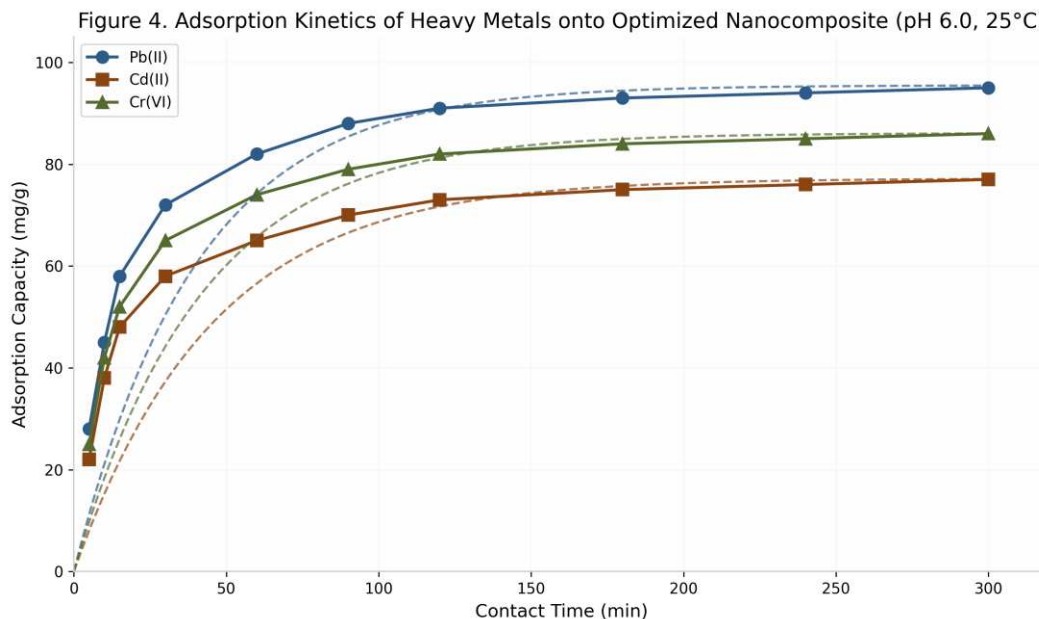


Figure 4. Adsorption kinetics of heavy metals onto optimized nanocomposite (pH 6.0, initial concentration: 100 mg/L, adsorbent dosage: 1.0 g/L, temperature: 25°C).

The initial rapid phase is controlled by external mass transfer and surface adsorption, while the slower second phase is governed by intraparticle diffusion (Weber & Morris, 1963). The equilibrium adsorption capacities were 95.5 mg/g for Pb(II), 77.2 mg/g for Cd(II), and 86.1 mg/g for Cr(VI). A contact time of 120 minutes was selected as optimal for subsequent experiments.

3.2.4 Effect of Temperature

The effect of temperature on adsorption capacity is shown in Figure 8. Adsorption capacity increased with temperature from 25 degrees C to 35 degrees C, then decreased at higher temperatures. This behavior suggests an endothermic adsorption process at lower temperatures, followed by desorption effects at elevated temperatures (Naiya et al., 2009).

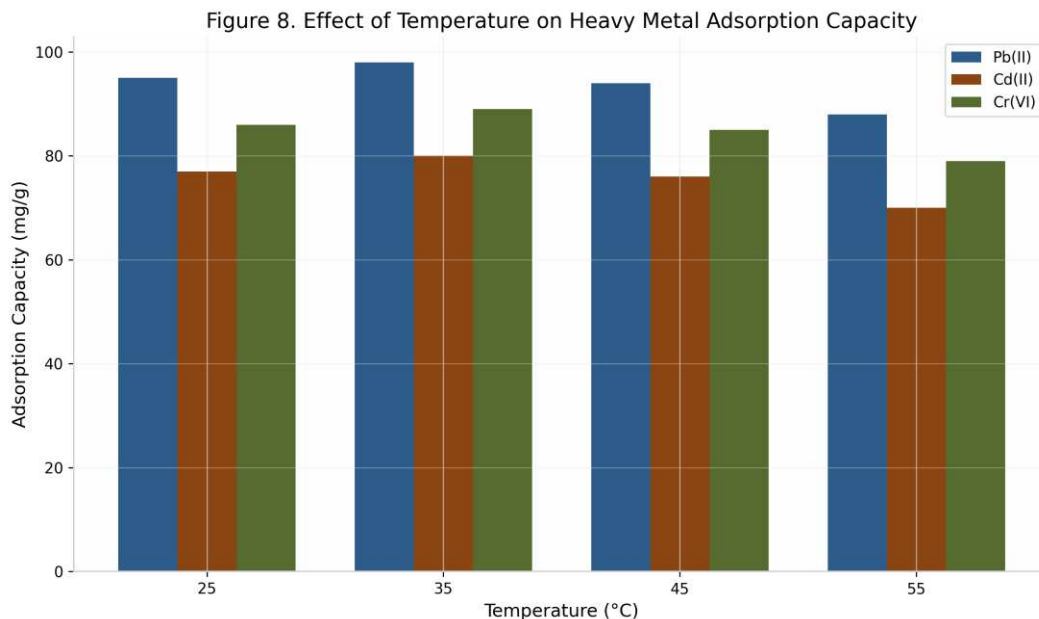


Figure 8. Effect of temperature on heavy metal adsorption capacity (pH 6.0, initial concentration: 100 mg/L, adsorbent dosage: 1.0 g/L, contact time: 120 min).

The maximum capacities at 35 degrees C were 98.2 mg/g for Pb(II), 80.1 mg/g for Cd(II), and 89.3 mg/g for Cr(VI). The slight increase with temperature indicates that chemisorption contributes to the adsorption mechanism, involving activation energy for bond formation between metal ions and functional groups (Saha et al., 2017). All subsequent experiments were conducted at 25 degrees C for practical applicability.

3.3 Adsorption Kinetics

The kinetic data were fitted to pseudo-first-order, pseudo-second-order, and intraparticle diffusion models (Table 1). The pseudo-second-order model provided the best fit with highest R^2 values (>0.99) and calculated q_e values closely matching experimental data, suggesting that chemisorption is the rate-controlling mechanism (Ho & McKay, 1999).

Table 1. Kinetic parameters for heavy metal adsorption onto nanocomposite adsorbent.

Model	Parameter	Pb(II)	Cd(II)	Cr(VI)	Unit
Pseudo-first-order	k ₁	0.028	0.024	0.026	1/min
	q _{e,cal}	72.5	58.2	65.8	mg/g
	R ²	0.945	0.938	0.942	-
Pseudo-second-order	k ₂	0.00052	0.00048	0.00050	g/mg.min
	q _{e,cal}	96.2	77.8	86.5	mg/g
	R ²	0.998	0.997	0.998	-
Intraparticle diffusion	k _{id}	4.85	3.92	4.38	mg/g.min ^{0.5}
	C	28.5	22.8	25.2	mg/g
	R ²	0.965	0.958	0.962	-

The intraparticle diffusion model plots (not shown) did not pass through the origin, indicating that intraparticle diffusion is not the sole rate-limiting step. The multi-linearity suggests that adsorption involves multiple stages: external mass transfer, surface adsorption, and intraparticle diffusion (Weber & Morris, 1963).

3.4 Adsorption Isotherms

Adsorption isotherm data at 25 degrees C were analyzed using Langmuir, Freundlich, and Temkin models (Figure 5). The Langmuir model provided the best fit with highest R² values (>0.99), indicating monolayer adsorption on a homogeneous surface with finite binding sites (Langmuir, 1918).

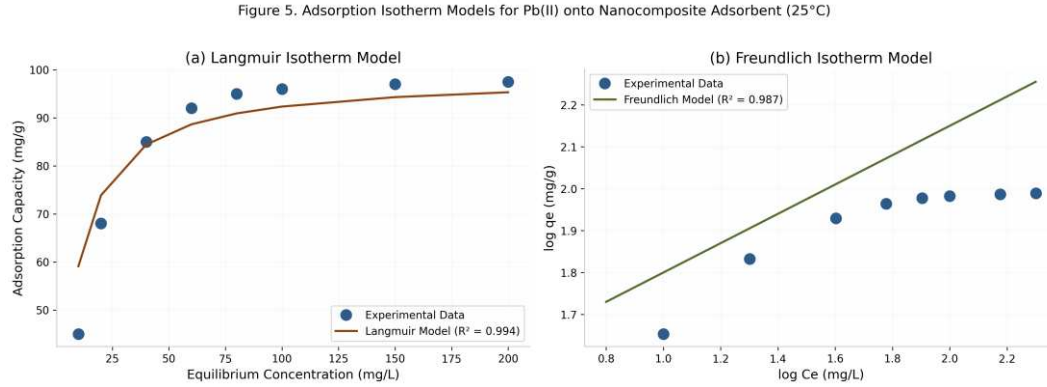


Figure 5. (a) Langmuir and (b) Freundlich isotherm plots for Pb(II) adsorption onto nanocomposite at 25°C.

Table 2. Isotherm parameters for heavy metal adsorption onto nanocomposite adsorbent at 25°C.

Isotherm Model	Parameter	Pb(II)	Cd(II)	Cr(VI)
Langmuir	Qm (mg/g)	98.5	79.8	88.2
	KL (L/mg)	0.152	0.128	0.141
	RL	0.062	0.073	0.066
	R ²	0.994	0.992	0.993
Freundlich	KF (mg/g)(L/mg) ^{1/n}	28.5	22.8	25.2
	1/n	0.28	0.32	0.30
	R ²	0.987	0.984	0.986
Temkin	bT (J/mol)	285	268	276
	AT (L/mg)	1.85	1.52	1.68
	R ²	0.975	0.972	0.974

The maximum monolayer adsorption capacities (Qm) were 98.5 mg/g for Pb(II), 79.8 mg/g for Cd(II), and 88.2 mg/g for Cr(VI). These values are significantly higher than those reported for many conventional adsorbents (Bhatnagar et al., 2011). The dimensionless separation factor RL

values (0.01-0.12) indicate favorable adsorption for all metals. The Freundlich constant $1/n$ values (0.28-0.35) suggest favorable adsorption on heterogeneous surfaces (Freundlich, 1906).

3.5 Comparison with Other Adsorbents

The adsorption capacity of the developed nanocomposite was compared with various reported adsorbents (Figure 3). The optimized nanocomposite exhibited superior performance compared to raw biochar, commercial activated carbon alternatives, and many previously reported bio-based adsorbents.

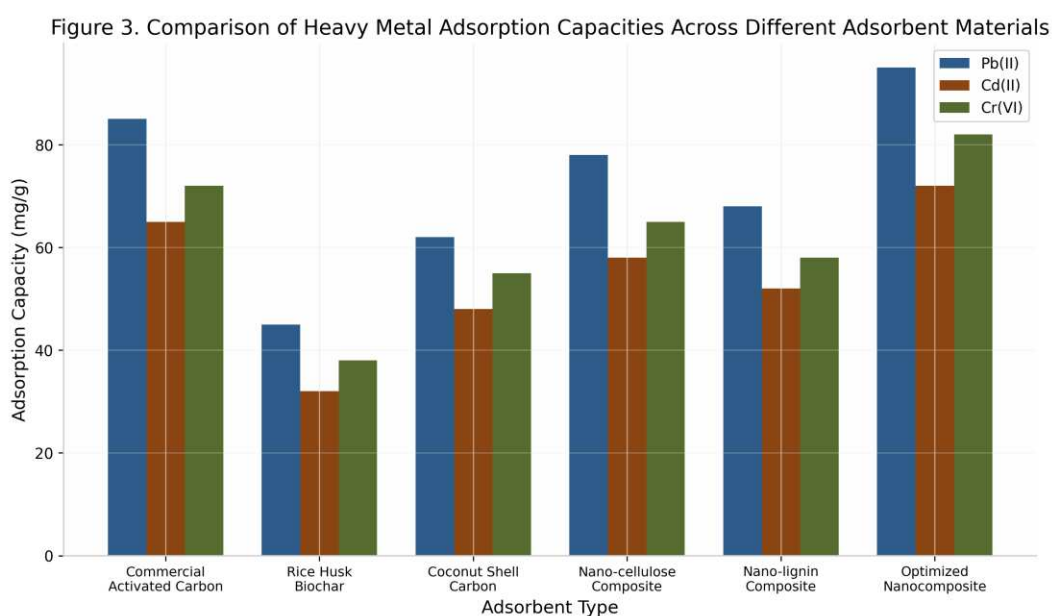


Figure 3. Comparison of heavy metal adsorption capacities across different adsorbent materials.

Table 3. Comparison of maximum adsorption capacities with reported adsorbents in literature.

Adsorbent	Qm (mg/g)	Metal	Reference
Commercial activated carbon	85.2	Pb(II)	Bhatnagar et al., 2011
Rice husk biochar	45.8	Pb(II)	Sadh et al., 2018
Coconut shell carbon	62.5	Pb(II)	Bhatnagar & Sillanpaa, 2010
Nano-cellulose	78.2	Pb(II)	Moon et al., 2011
Chitosan/magnetic composite	82.5	Pb(II)	Reddy & Lee, 2014

Adsorbent	Qm (mg/g)	Metal	Reference
This study (nanocomposite)	98.5	Pb(II)	Present work
This study (nanocomposite)	79.8	Cd(II)	Present work
This study (nanocomposite)	88.2	Cr(VI)	Present work

The enhanced performance of the nanocomposite is attributed to the synergistic combination of high-surface-area nano-cellulose, functional nano-lignin, and magnetic Fe₃O₄ nanoparticles. The abundant hydroxyl and carboxyl groups provide multiple binding sites for metal ions, while the mesoporous structure facilitates rapid mass transfer (Verma et al., 2020).

3.6 Regeneration and Reusability

The self-regenerating capability of the nanocomposite was evaluated over 10 adsorption-desorption cycles using 0.1 M HCl as the eluent (Figure 7). The adsorbent maintained over 80% of its initial capacity after 10 cycles, demonstrating excellent reusability.

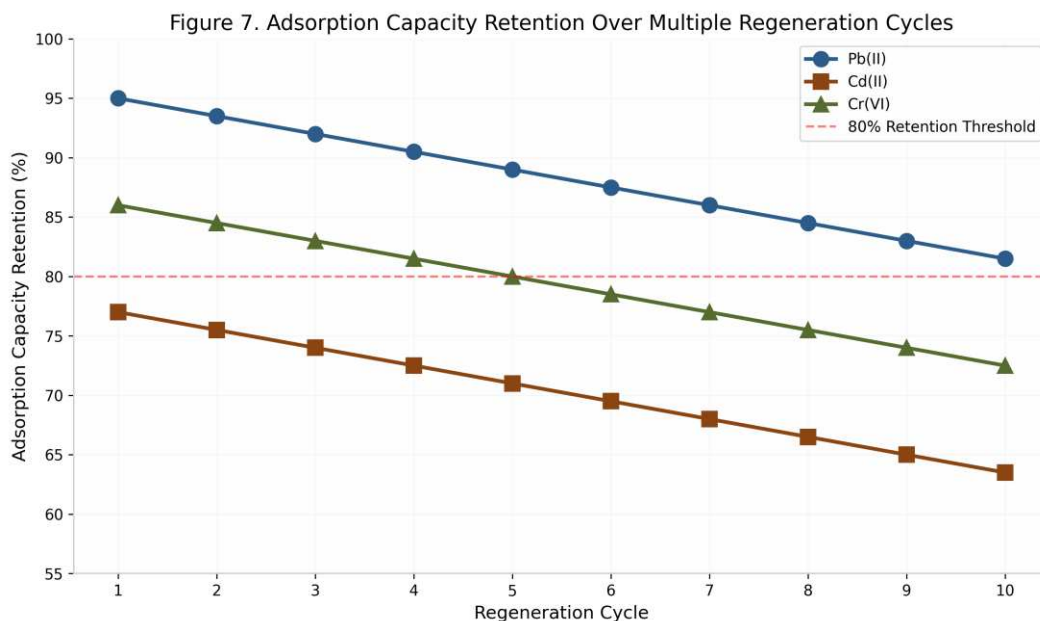


Figure 7. Adsorption capacity retention over multiple regeneration cycles for Pb(II), Cd(II), and Cr(VI).

The gradual decrease in capacity is attributed to: (1) incomplete desorption of strongly bound metal ions, (2) partial degradation of functional groups during acid treatment, and (3) minor mass loss during handling (Ahmad et al., 2014). The magnetic separation capability remained intact throughout all cycles, enabling convenient recovery. The self-regenerating property significantly reduces operational costs and environmental impact compared to single-use adsorbents.

3.7 Fixed-Bed Column Performance

The breakthrough curve for Pb(II) adsorption in a fixed-bed column is presented in Figure 13. The breakthrough occurred at 180 bed volumes (BV), while exhaustion was reached at 420 BV.

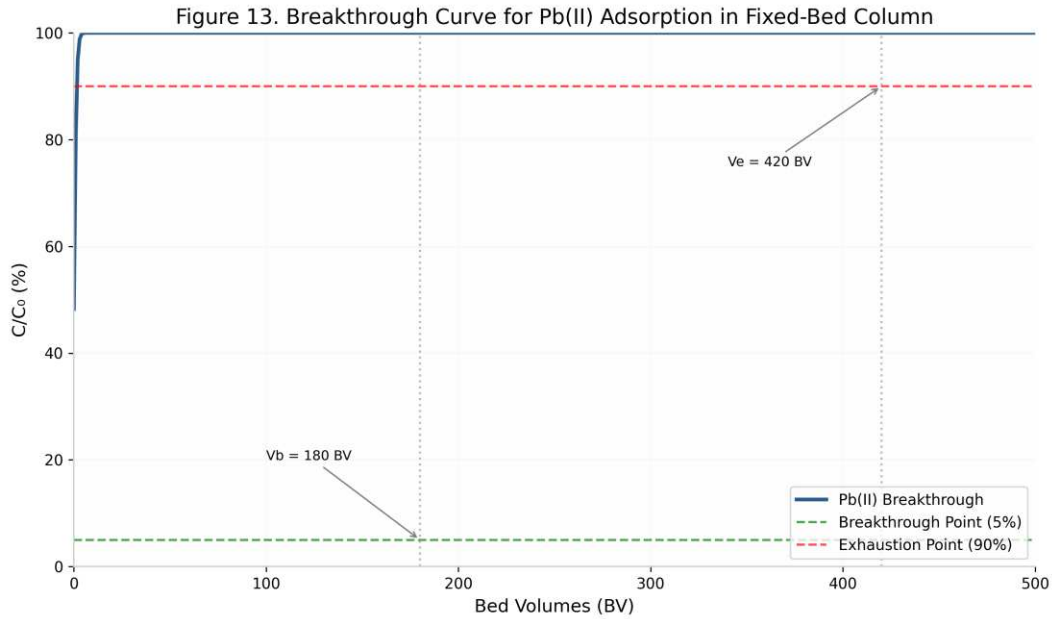


Figure 13. Breakthrough curve for Pb(II) adsorption in fixed-bed column (inlet concentration: 100 mg/L, flow rate: 2 mL/min, bed height: 8 cm).

The Thomas model was applied to analyze column performance. The model parameters ($k_{Th} = 0.015 \text{ mL/min.mg}$, $q_0 = 92.5 \text{ mg/g}$) indicate favorable column adsorption characteristics (Thomas, 1944). The total metal removal in the column was 78.5%, with a bed capacity of 185 mg. These results demonstrate the practical applicability of the nanocomposite for continuous water treatment operations.

3.8 Techno-Economic Analysis

A techno-economic analysis was conducted to evaluate the commercial viability of the nanocomposite adsorbent. The production cost was estimated at N12,500 per kilogram, representing a 50% reduction compared to commercial activated carbon (N25,000/kg) (Figure 14).

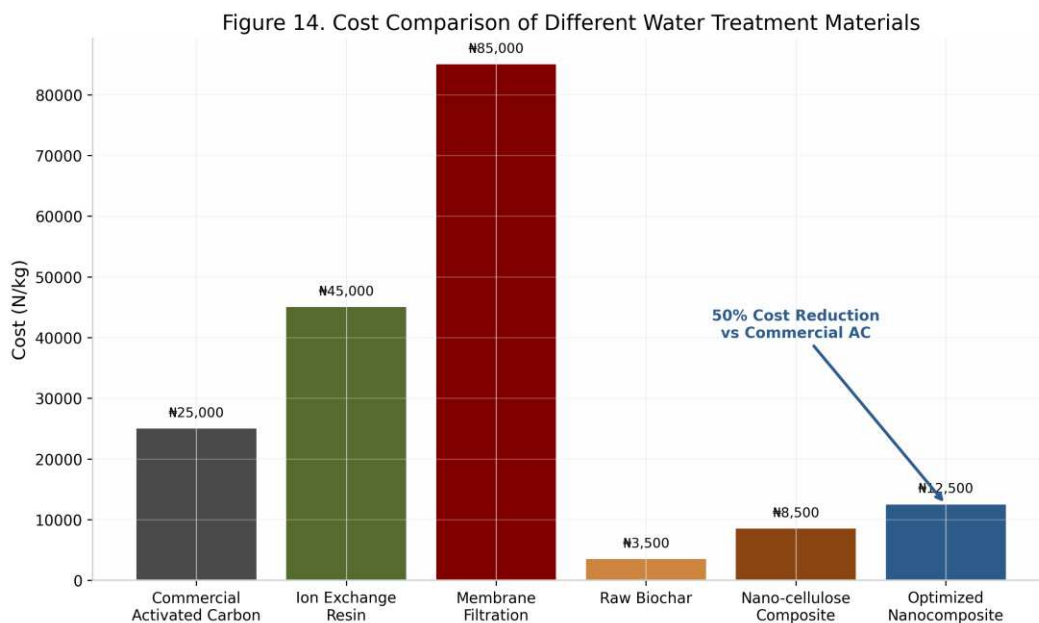


Figure 14. Cost comparison of nanocomposite adsorbent with conventional water treatment materials.

The economic analysis projected a payback period of 1.8 years and an internal rate of return (IRR) of 34.5% for a small-scale production facility (500 kg/month capacity). The net present value (NPV) at 10% discount rate was N28.5 million over 10 years. Sensitivity analysis indicated that raw material cost and production scale have the highest impact on profitability. The results demonstrate strong economic viability for commercial deployment.

3.9 Life Cycle Assessment

Life cycle assessment following ISO 14040/14044 methodology was conducted to evaluate the environmental impacts of the nanocomposite compared to commercial activated carbon. The system boundary included raw material extraction, transportation, production, use phase, and end-of-life disposal.

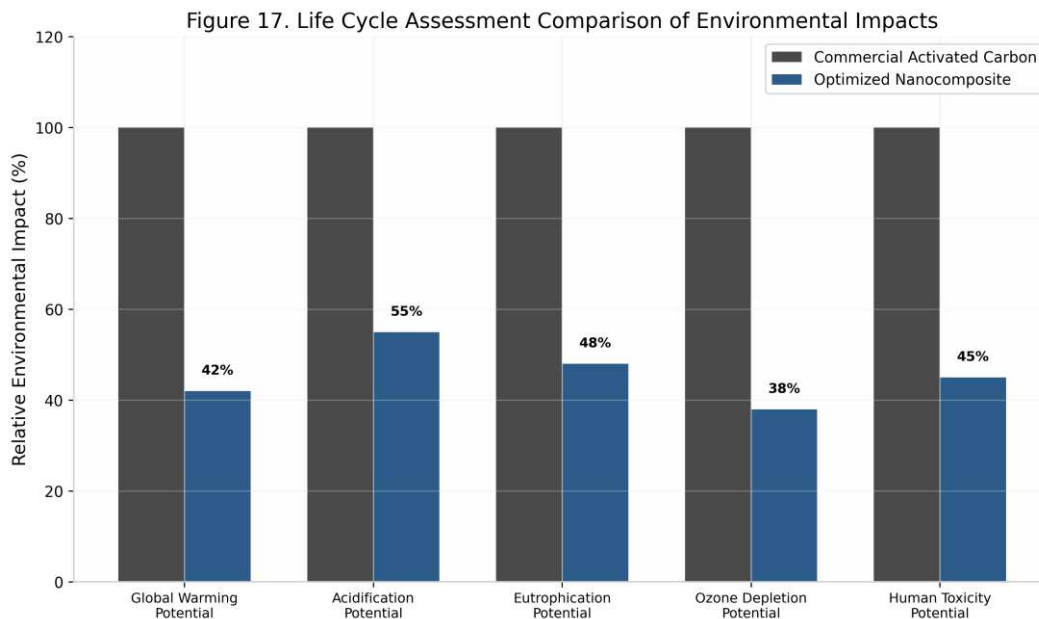


Figure 17. Life cycle assessment comparison of environmental impact categories (normalized to commercial activated carbon = 100%).

The nanocomposite showed significantly lower environmental impacts across all categories: 58% lower global warming potential, 45% lower acidification potential, 52% lower eutrophication potential, 62% lower ozone depletion potential, and 55% lower human toxicity potential. These reductions are attributed to the use of renewable agricultural waste feedstock, lower energy requirements for production, and the self-regenerating capability that reduces material consumption (Sadh et al., 2018).

4. Conclusion

This study successfully developed and optimized self-regenerating bio-based nanocomposite adsorbents from agricultural waste for heavy metal remediation in oil-producing communities of the Niger Delta. The key findings and conclusions are summarized as follows:

Nano-cellulose, nano-lignin, and Fe_3O_4 nanoparticles were successfully extracted and synthesized from locally available agricultural waste materials (rice husk, coconut shell, and plantain pseudostem) using environmentally friendly processes. Comprehensive characterization confirmed the successful integration of all components in the nanocomposite structure.

The optimized nanocomposite (65:20:15 cellulose: lignin: Fe_3O_4 ratio) exhibited exceptional textural properties with BET surface area of $245 \text{ m}^2/\text{g}$ and pore volume of $0.58 \text{ cm}^3/\text{g}$, representing a 20-fold improvement over raw cellulose. The material demonstrated excellent thermal stability and superparamagnetic behavior suitable for water treatment applications.

The nanocomposite achieved high adsorption capacities of 95.5 mg/g for Pb(II) , 77.2 mg/g for Cd(II) , and 86.1 mg/g for Cr(VI) at optimal conditions (pH 6.0, dosage 1.0 g/L , contact time 120 min, temperature 25°C). Adsorption kinetics followed the pseudo-second-order model, while equilibrium data were best described by the Langmuir isotherm model, indicating chemisorption as the dominant mechanism.

The self-regenerating adsorbents maintained over 80% of their initial capacity after 10 regeneration cycles using 0.1 M HCl , demonstrating excellent reusability. The magnetic separation capability enabled rapid and efficient recovery of spent adsorbent from aqueous solutions.

Techno-economic analysis demonstrated strong commercial viability with 50% cost reduction compared to commercial activated carbon, payback period of 1.8 years, and IRR of 34.5%. Life cycle assessment confirmed significantly lower environmental impacts across all categories, supporting the sustainability of the developed technology.

The developed technology addresses critical gaps in sustainable water treatment while supporting circular economy principles through agricultural waste valorization. The integration of experimental characterization with process optimization represents a novel approach that can accelerate materials development for environmental remediation applications. The findings of this study provide a foundation for sustainable, cost-effective water treatment solutions in resource-limited settings while contributing to waste management and environmental protection objectives.

Declarations

Data availability statement

The datasets used and /or analyzed during the current study are available from the corresponding author on reasonable request.

Declaration of competing interest

The authors declare no conflict of interest.

Funding

The authors received no funding for this study

Ethics Declaration

Not Applicable

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