



Removal of Fe(II) from Aqueous Solution Using Characterized Cellulose of *Pentaclethra macrophylla Benth* Pod: Adsorption, Thermodynamic, and Kinetic Studies

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ABSTRACT

The study describes the removal of iron(II) ions from aqueous solutions using characterized cellulose derived from *Pentaclethra macrophylla Benth* pod, as well as the adsorption isotherm, thermodynamic, and kinetic analyses of Fe(II) ion on raw and isolated cellulose. Cellulose was isolated from the pod of *Pentaclethra macrophylla Benth* via delignification and bleaching. X-ray diffraction (XRD), scanning electron microscopy (SEM), Fourier Transform Infrared Spectrometry (FTIR), Brunauer–Emmett–Teller (BET) analysis were used to characterize the raw and isolated cellulose. The adsorbents were further characterized using adsorption isotherms, kinetics, and thermodynamic models. The isolated cellulose has better thermal stability, crystallinity, and porosity than the raw. The crystallinity index of isolated cellulose was estimated at 32.4%. From the findings, Fe(II) removal was around 95.33% at 30°C, 93% at 150 minutes contact time, 87.34% at the initial concentration of metal ions of 50 mg/g, and 78.20% at a pH of 8. The isotherms data were consistent with all models, but the Freundlich and Temkin models fit better than the Langmuir isotherm. This is the basis that the R² in both the Freundlich and Temkin models were at the range of 0.9109–0.9992. The ΔG° values for Fe(II) in cellulose increased with increasing temperature and were all less than -5 kJ/mol. Thus, it implies physical adsorption and exothermic processes. The adsorption of Fe(II) with cellulose followed a pseudo-second-order adsorption. The results suggest that *Pentaclethra macrophylla Benth* pod has a high possibility of being used as an effective and economical adsorbent for the removal Fe(II) ion.

Keywords: Adsorption, Cellulose, Iron(II) ion, *Pentaclethra macrophylla Benth*, Pod

Introductions

Excessive heavy metal pollution is a major global issue. Water pollution from heavy metals poses a serious risk to the global eco system. The need for innovative methods to remove heavy metals from industrial wastewater is driven by restrictive environmental protection legislation and public environmental concerns. The removal of highly poisonous and harmful metal ions from water is accomplished using conventional methods such as coagulation, reverse osmosis, electrochemical treatment, flocculation, membrane technology, ion exchange, precipitation, adsorption/sorption, etc [15]. However, these approaches are expensive, inaccessible and not environmentally benign. The use of adsorption to remove pollutants from diluted solutions is seen as a viable approach. This is due to its efficacy, as well as its safety, subtlety, environmental friendliness, non-destructiveness, and low cost [9]. Activated carbon is the most widely used adsorbent for purifying water because of its large surface area, high porosity, and unique chemical characteristics [16].

Recently, scientists all over the world have been studying low-cost adsorbents made from agricultural wastes. Cellulose, in both its native and derivatized forms, is the most abundant biomass material found in nature and makes for great adsorption materials. Because of their biodegradability, biocompatibility, low accumulation tendencies, low cost, and most importantly, recyclability, agricultural material, both modified and unmodified, has emerged as a new class of adsorbents for the adsorption of metal ions [46] For their use in separation technologies, many low-cost polymers have been grafted with different vinyl monomers. These include chitosan, chitin, pectin, starch, cellulose, bacterial cellulose, lignin, and hemicelluloses [20].

The *Pentaclethra macrophylla Benth* tree is widely distributed along the coasts of West and Central Africa, including in Nigeria's Southern and Middle Belt Areas. A member of the Mimosoideae subfamily of the leguminosae family [8]. Pollution in Nigeria's eastern region is caused in part by the careless disposal of biomass waste, specifically the pods and husks of the *Pentaclethra macrophylla Benth* plant. According to the literature, the unfermented seed oil of pentaclethra macrophylla benth contained fatty acid [30], essential mineral elements (phosphorus, calcium, silicon, magnesium and other metal) [28]. vitamins (thiamin, riboflavin, and niacin) [35], sugars (stachylose, galatose, and fructose) [34].

As a function of equilibrium time, adsorbent dose, temperature, adsorbate concentrations, and pH in a batch system, the biosorption of Cu(II), Co(II), and Fe(II) ions from aqueous solutions by various biomasses has been studied [51].

Several researchers used different low-cost adsorbents from agriculture wastes, such as coconut coirpith [21], sawdust, rice husk [15], banana pith [24], cottonseed hulls [25], apple wastes [3], sugarcane bagasse pith [22], and activated carbon [13] for the removal of copper and cadmium from water and wastewater. There is still a need to design appropriate adsorbents for the removal of iron from aqueous solutions, despite the fact that many researchers have adopted numerous low-cost adsorbents.

The purpose of this study was to provide evidence that iron (II) may be effectively removed from aqueous solutions using cellulose from the *Pentaclethra macrophylla benth* pod. In this investigation, isolated cellulose was characterized using FTIR, SEM, XRD, and BET. The isolated cellulose was used in the removal of Fe(II), in simulated water, and thereafter compared with the removal capacity of raw *Pentaclethra macrophylla benth*. Adsorption, thermodynamic, and kinetic studies were used to ascertain the removal efficiency of cellulose.

Material and Methods

Materials

The natural pentaclethra macrophylla benth pod, Potassium hydroxide (KOH), hydrochloric acid (HCl), ethanol, toluene, acetic acid, sodium chlorite, NaOH and other chemicals were analytical grade. All the chemicals were used as received. Deionized water was used in all experiments

Sample collection and Preparation

The *pentaclethra macrophylla benth* pod was gathered in Aku, Igbo Etiti Local Government Area, Enugu State, and transported to the department of Industrial chemistry laboratory, University of Science and Technology, Enugu. It was carefully sorted to eliminate foreign material from the sample. To prepare for pulverisation, the sample was rinsed with distilled water, sun-dried for 2-3 weeks, and then chopped with a cutter. To increase the surface area and improve future treatment, the sun-dried chopped pod sample was crushed into fine powder and sieved to particle sizes of 0.07 mm.

Dewaxing of *Pentaclethra Macrophylla Benth* Pod

The dewaxing technique used was consistent with Nsude et al. (2022b). The powdered sample (100g) was extracted for 6 hours with 375 ml of toluene and ethanol (2:1) using a soxhlet extractor to remove chlorophyll pigments and waxes. After removing the boiling chips, the filtrate (toluene-ethanol combination) was discarded. The residue (dewaxed) was dried at room temperature, weighed, and stored in a sealed plastic bag for further analysis.

Bleaching of the Cellulose Residue *Pentaclethra Macrophylla Benth* Pod

The resulting sample residue was bleached for 30 minutes at 70 °C using an aqueous solution of sodium hypochlorite (7.5%). The resulting holocellulose was extensively cleaned and filtered. The resultant holocellulose was then treated with 17.5% w/v sodium hydroxide at 80 °C for 30 minutes. The cellulose pulp was thoroughly rinsed with water. The cellulose pulp was whitened further by employing a 1:1 aqueous solution of sodium hypochlorite (3.5 % w/v) for 5 minutes at 100 °C, followed by washing until the filtrate was clear. Excess water was manually squeezed out using a calico cloth, and the alpha-cellulose pulp was oven-dried at 50 °C [42].

Characterizations

Scanning Electron Microscope

The morphological feature of the cellulose was observed using scanning electron microscope (SEM, FEI, Quanta 200, USA), transmission electron microscope (TEM, FEI, Tecnai G20, USA).

Fourier Transform Infrared

The IR spectra were obtained from the FTIR-8400S Fourier Transform Infrared spectrophotometer at NARICT Zaria using an ATR disc. It was used to identify the functional groups,

X-ray Diffraction

The crystalline structures of cellulose samples were determined by XRD technique. XRD analysis was carried out using a Bruker D8 ADVANCE Powder XRD instrument with CuK- α radiation of $\lambda = 1.5404$ nm and the X-ray diffractometer was operated at a voltage of 40 kV

and a current of 30 mA. XRD data were collected within the range of scattering angles (2θ) of 10 to 40° at room temperature.

Crystallinity index (CrI) was calculated using the formula as stated in Equation 1:

$$\text{CrI (\%)} = [(I_{200} - I_{\text{CAM}}) / I_{200}] \times 100 \quad (1)$$

Where I_{200} and $I_{\text{Cr-non}}$ are the maximum peak intensities of crystalline and amorphous regions, respectively (Saravanan et al. 2015).

BET Analysis for surface area (Quanta chrome Nova4200e model)

The *Pentaclethra Macrophylla Benth* pod sample was properly weighed and placed into the sample cell, then the filled sample cell bulb was placed into the heating mantle, and the clamp was placed around the mantle to hold the sample cell firm. The out gassing temperature was set at 2500°C , and the system was instructed to start degassing for 3 hours and turn on the heater. Thereafter, the heating mantle was turned off and the sample cell was allowed to cool

Atomic absorption spectrophotometer (AAS) MODEL: FS240A analysis

100 ml of well shaken water sample was transferred into a glass beaker of 250 ml volume, 5 ml concentration of nitric acid was next added and boil until volume reduced to 15-20 ml, addition of concentrated nitric acid drop wise continued until the residue were completely dissolved. The mixture is cooled, transferred and made up to 100ml using metal free distilled water; a calibration graph is obtained by feeding the standard solutions of suitable concentration. The samples are aspirated by feeding them through the capillary and the readings were noted.

Batch Adsorption Studies

The method adopted was in line with Azouaou et al. (2010). A weighed amount of raw and cellulose was introduced into stoppered reagent bottles containing various concentrations of 100 mL aqueous solutions of Fe(II) ions. The suspensions were shaken at room temperature ($25 \pm 1^\circ\text{C}$) using a mechanical shaker for a prescribed time at 150rpm. The solutions were filtered through Whatman 42 filter paper, and the residual concentration of Fe(II) ion was determined by the AAS method at 217 nm. The effects of concentration (50–250 mg/L), contact time (30–150 min), temperature ($30\text{--}50^\circ\text{C}$), solution pH (2–10), and adsorption dose

(0.1–0.5 g) were studied. Blank solutions were treated similarly (without adsorbent), and the concentration recorded at the end of each operation was taken as the initial concentration.

Adsorption Capacity

The adsorption capacity that is the metal ions adsorbed onto the resin was calculated using mass balance relation as in equation 2

$$Q_e = \frac{(C_o - C_e)V}{M} \quad (2)$$

Where: Q_e (mg/g) is the adsorption capacity of the adsorbent; C_o and C_e (mg/l) are the initial and final concentration of the metal ions in solution phase, V is the volume of the aqueous solution (l)

M is the weight of the adsorbent (g), (Ekpete et al., 2011)

The percentage of ions removed was calculated as thus 3

$$\% \text{ Removal} = \frac{(C_o - C_e)}{C_o} \times 100 \quad (3)$$

C_o

Result and Discussion

Morphological Analysis of Raw and Cellulose from *Pentaclethra macrophylla* Benth Pod

The morphology of *Pentaclethra macrophylla* Benth Pod at different chemical treatment stages was analysed using scanning electron microscopy (SEM).

Raw powder and isolated cellulose were viewed in SEM micrographs at different magnifications and processing stages (900x and 1000x). As the image size increased, so was the clarity of the micrograph, and it was also noted that the image appeared tied together and intertwined [34].

The lignocellulosic structure of the raw materials may not be readily apparent due to the presence of wax, hemicelluloses, pectin, and lignin, among other impurities and components. This observation is congruent with the view held by [2] in their morphological study of cocoa pod husks, which attributes the layered or cemented structure to impurities due to wax, hemicellulose, pectin, and lignin.

X-ray Diffraction and Crystal Structure Analysis of Cellulose *Pentaclethra macrophylla* Benth Pod

As reinforcement in polymer composites, cellulose's mechanical and thermal properties are determined by its degree of crystallinity.

Nsude et al. (2022) [29] has previously reported the XRD analysis and the crystallinity of the raw powder and extracted cellulose composites of *Pentaclethra macrophylla* Benth pod. The crystalline area of raw powder is represented by the strong peak at $2\theta = 22.5^\circ$ (I200),

whereas the amorphous zone is shown by the peak at $2\theta = 16.4^\circ$ (Iam). Isolated cellulose after alkaline treatment and bleaching of raw powder has a crystalline region of around $2\theta = 22.5^\circ$ (I200), while the amorphous region has a value of around $2\theta = 16.5^\circ$ (I200) (Iam). This result was in agreement with that found by Akinjkun et al. (2021), who determined that the crystalline area of untreated cellulose in cocoa pod husk or eggshell was $2\theta = 23^\circ$ (I200) while the amorphous zone was $2\theta = 15^\circ$. (Iam),.

Isolated cellulose showed additional peaks at 38°C and 58°C , which are consistent with the crystallographic planes of the cellulose 1 polymorph reported by Shaikh et al., (2021).

The crystallinity index (CrI) was estimated at 30% for raw cellulose and 74% for isolated cellulose. This indicates decisively that progressively more aggressive chemical treatments improve the crystallinity of isolated cellulose.

ATR-FTIR Analysis of Raw and Isolated Cellulose from *Pentaclethra macrophylla* Benth Pod

Table 1 shows the functional group analysis of both raw *Pentaclethra macrophylla* Benth pod powder and the isolated cellulose of PMBP biomass. This FTIR analysis has been reported in Nsude et al. (2022) [29].

Table 1: Summary of Functional Group Analysis of *Pentaclethra macrophylla* Benth Pod

	Functional Groups	Absorption(s) frequency(cm^{-1})
1	OH hydroxyl group	3446 - 3250
2	C- H stretch	2960-2850
3	C-C=O stretching of carboxylic acid or ester (lignin)	1750-1735
4	C=C stretching of aromatic ring (lignin)	1470-1350
5	CH ₂ symmetrical bending (lignin)	1430-1330
6	C-O stretching of acetyl (lignin)	1245-960
7	β -glycosidic linkage	1000-980
8	Si-O-C- stretching	1250-1100
9	-Si-C- symmetric stretching	800-750
10	-Si-O-Si-	720-700

The O-H band is characterised by a broad peak between 3550 and 3200 cm^{-1} . All spectra show a peak at around 1000-980 cm^{-1} , which can be interpreted as the β -glycosidic connections of the glucose ring in cellulose [20], [37] Raw powder and isolated cellulose both contain silicon compounds, as indicated by a band in their spectra at 720-700 cm^{-1} , which has been attributed to the -Si-O-Si-. Carbohydrate C-H rocking vibrations (2960–2850 cm^{-1}) and water O-H bending vibrations (34, 35) account for the existence of organic molecules in the samples, whereas other peaks detected are shown in Table 1. Total FTIR analysis showed that *Pentaclethra macrophylla* Benth pod was predominantly composed of cellulose, with minor amounts of hemicelluloses and lignin. lignin.

Brunauer–Emmett–Teller of Analysis for surface area *Pentaclethra macrophylla* Benth Pod

BET analysis is a method used to determine the adsorption property of a solid biomass material. Table 2 shows the BET analysis of the raw and isolated cellulose

Table 2: Analysis for Surface Area Raw and Cellulose of *Pentaclethra macrophylla* Benth Pod

	Properties	Raw	Cellulose
1	SpecificSurface area BJH (m ² /g)	164.73	324.12
2	pore volume (m ² /g)	0.081	0.159
3	Average pore Diameter or size BJH (nm)	2.013	2.133
4	BET summary surface area (m ² /g)	159.133	288.090
5	BET pore Diameter (nm)	2.140	2.940
6	Correlative Coefficient	0.989	0.996

Table 2 reveals that isolated cellulose has better properties than raw powder, as reported by Nsude et al. (2022) [29]. In comparison to isolated cellulose, the raw material has a smaller specific surface area. Variable chemical treatments applied to the raw material are correlated with the observed increase in a specific surface area. Because of this increase in surface area, isolated cellulose should be more bioavailable than raw cellulose. The findings of Sieradzka et al. (2020), who investigated the pyrolysis of biomass wastes, are in line with these observations. Table 2 shows that the isolated cellulose has greater values for pore volume, average pore diameter, BET summary surface area, and BET pore diameter than the raw cellulose. According to Farooq et al. (2012) [14], the adsorption capacity is proportional to the BET surface area; thus, larger particles with a greater BET surface area should result in greater metal removal. This finding suggests that *Pentaclethra macrophylla* Benth pod cellulose can be used effectively as a metal remover in polluted environments.

Effect of pH on the Adsorption of Fe²⁺ Using *Pentaclethra macrophylla* Benth Pod as Adsorbent

The pH of the aqueous solution is an important controlling parameter in the heavy metal adsorption process [6]; [5]. Figure 1 shows the effect of different pH on the adsorption of Fe(II) ions with raw and isolated cellulose from *Pentaclethra macrophylla* Benth pod as an adsorbent. For the raw, the percentage of Fe(II) ions adsorbed increased as the pH moved from an acidic pH to an alkaline pH of 8, and decreased at pH 10. This trend of adsorption was observed with isolated cellulose with a higher percentage of adsorption. This is consistent

with Kukwa et al.'s (2020) research on the pH variation and metal removal of rice husk used as an adsorbent.

This implies that alkaline media tend to support adsorption more than acidic media. However, the low efficiency at pH 10 and low solubility are attributed to the precipitation of Fe(II) ions at pH values greater than 8. At low pH values, the adsorbent's surface would be repulsively associated with hydroxium ions (H_3O^-) to the surface functional groups, reducing the percentage adsorption of Fe(II) ion. This can further be inferred that there was a corresponding increase in the deprotonation of the adsorbent surface, leading to a decrease in H^+ ions on the adsorbent surface, when the pH of the adsorbing medium was increased from pH 2 to 10, [36]. This creates more negative charges on the adsorbent surface, which favours the adsorption of positively charged species and the positive sites on the adsorbent surface [32].

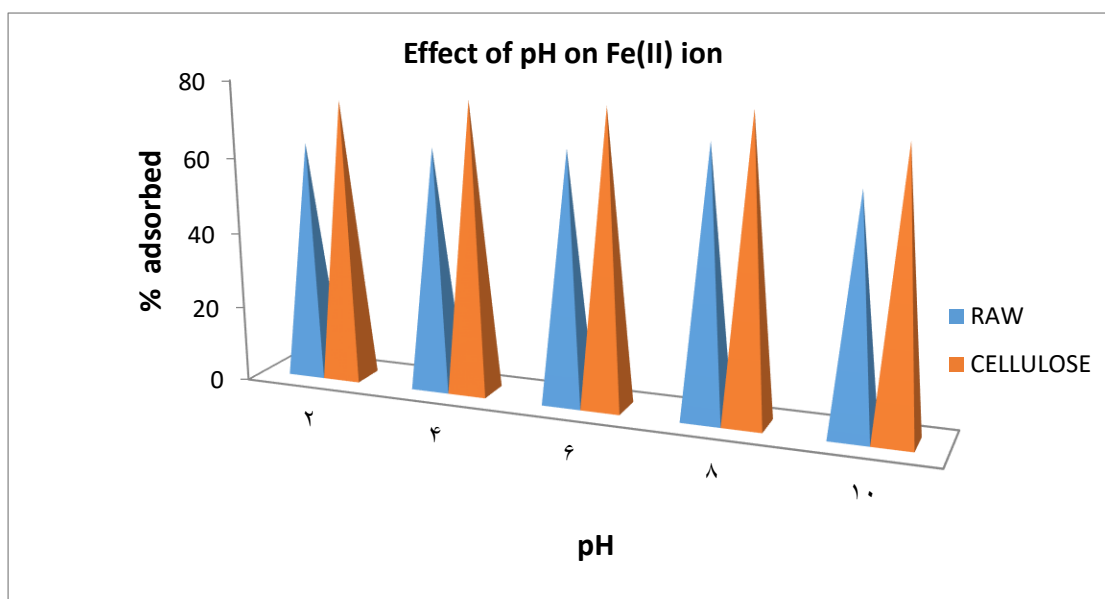


Figure 1: Effect of pH on the Adsorption of Fe^{2+} Using *Pentaclethra macrophylla* Benth Pod Biomass Adsorbent

Effect of Dosage on the Adsorption of Fe^{2+} Using *Pentaclethra macrophylla* Benth Pod Biomass wastes Adsorbent

Adsorbent dosage is an important parameter in the adsorption of metal ions from aqueous solutions owing to its effects on the amount of metal ions removed per unit mass of adsorbent. Figure 2 shows an increase in the adsorption percentage of heavy Fe(II) ion removal as the

adsorbent increases from 0.1 g to 0.5 g. The maximum percentage of Fe^{2+} adsorption was found to be about 70.427% for raw and 89.69% for isolated cellulose at 0.5 g of dosage. This implies that the isolated cellulose was a better adsorbent than the raw material.

It can be inferred that the percentage adsorbed of Fe(II) ions increased with the increasing mass of isolated cellulose. This is due to the greater availability of the exchangeable sites, or surface area, at the higher dose of the adsorbent. This effect has corresponded with the results of previous studies [52] [50].

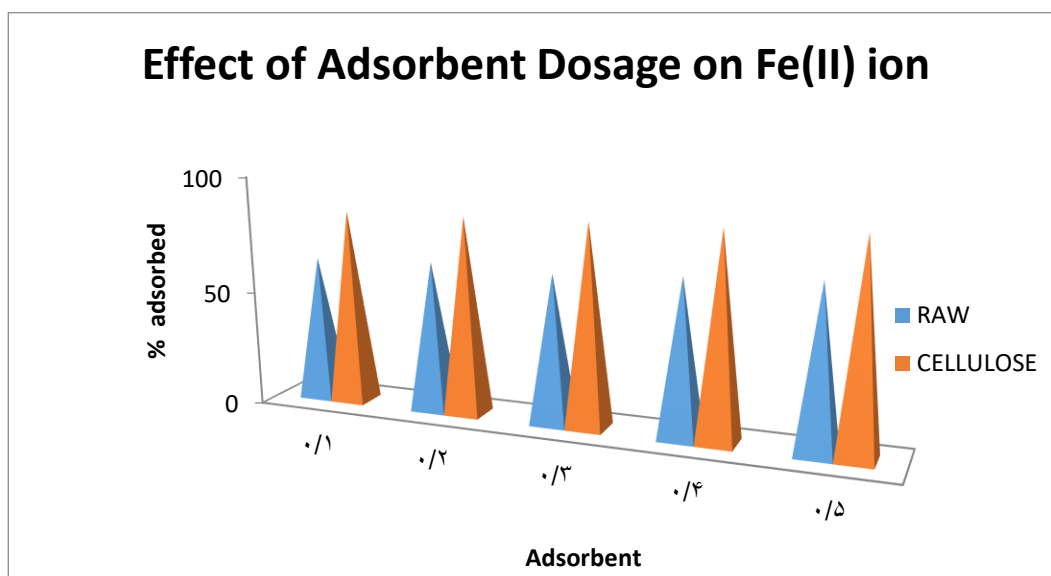


Figure 2: Effect of Dosage on the Adsorption of Fe^{2+} using *Pentaclethra macrophylla* Benth Pod Adsorbent

Effect of Temperature on the Adsorption of Fe^{2+} Using *Pentaclethra macrophylla* Benth Pod Biomass wastes Adsorbent

The effect of temperature on the adsorption of Fe^{2+} on raw and isolated cellulose is shown in Figure 3. An increase in temperature from 30 °C to 50 °C was found to result in a steady decrease in the removal efficiency of the adsorbent for the Fe(II) ion. This is probably due to the effect of temperature on the interaction between the adsorbent surface and the metal ions in solution [11]; [32].

At 30 °C, the isolated cellulose had the highest percentage adsorption of 95.33%, which was higher than the raw, with a percentage adsorption of 70.25% (see Figure 4.7).

The tendency for the adsorption process to slow down might be because the attractive forces between the adsorbent and the adsorbate ions are getting weaker. An increase in temperature

from 30 °C to 50 °C caused a proportional decrease in the amount of Fe(II) ions adsorbed onto the surface of the adsorbent. At high temperatures, the thickness of the boundary layer was expected to decrease due to the increased tendency of the Fe(II) ion to escape from the surface of the adsorbent into the solution phase, and hence there was bound to be weak adsorption interaction between the adsorbent and the adsorbate. This decrease in adsorption capacity with an increase in temperature indicates that the adsorption processes were exothermic in nature, which is consistent with Vilar et al. (2012) and Ojedokun & Bello (2016) [31].

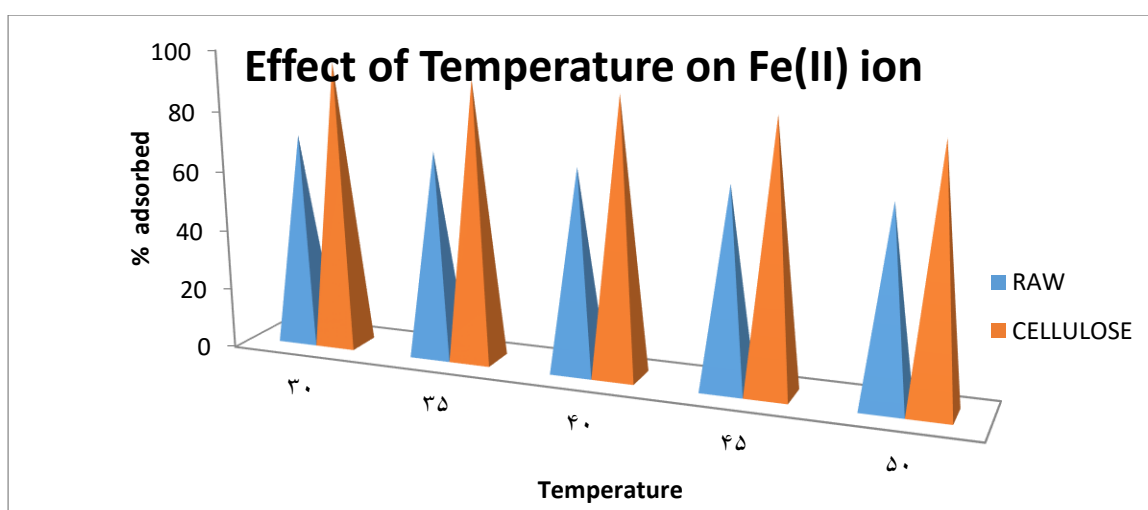


Figure 3: Effect of Temperature on the Adsorption of Fe²⁺

Effect of contact time on the Adsorption of Fe²⁺ Using *Pentaclethra macrophylla* Benth Pod Biomass wastes Adsorbent

The contact time of an adsorption system is important because it can be used to control the movement of metal ions from the solution to the surface of the adsorbent and back again [4]. The adsorption of metal ions onto raw and cellulose-rich *Pentaclethra macrophylla* Benth pods is depicted in Figure 3. The amount of adsorbed Fe(II) ions was shown to rise over time. The cellulose has the largest percentage removal of Fe(II) ions (93%), while the raw has the lowest (73.74%) (Figure 4).

The gradual increase in adsorption over the course of several hours suggests that many free surface sites were available for adsorption at the outset of the process, but that the remaining free sites were occupied slowly over time due to repulsive forces between the solute

molecules on the adsorbent surface and the solution phase. This is because, as time passes, more and more metal ions migrate from the solution through the adsorbent's border layer and end up at the adsorbent's active sites. Since the adsorption kinetics is dependent on the adsorbents' surface area, the initial faster rate could be because of the availability of the uncovered surface area of the adsorbents. This phenomenon was consistent with Wongjunda and Saueprasearsit [55], (2010) and Kukwa et al. (2020) [23], who researched metal adsorption using rice husk ash, modified rice husk ash.

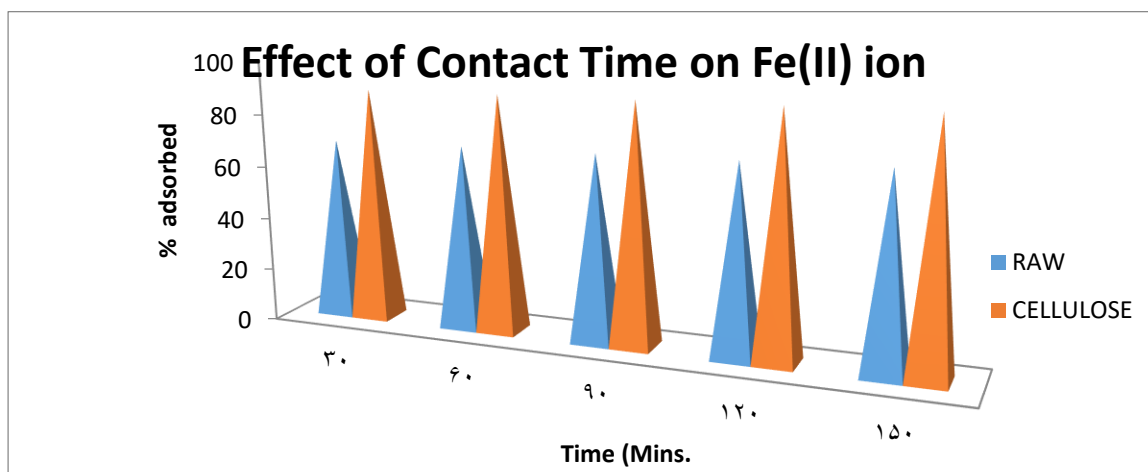


Figure 4: Effect of contact time on the adsorption of Fe^{2+} *Pentaclethra macrophylla Benth* pod adsorbent

Effect of Varying Concentration of Fe^{2+} at *Pentaclethra macrophylla Benth* Pod Adsorbent

The rate of adsorption is a function of the initial concentration of metal ions. Figure 4 indicates that higher adsorption was found to take place at lower concentrations. This may be due to the interaction of all metal ions present in the solution with binding sites. The highest percentage removal of $\text{Fe}(\text{II})$ ions was 67.26% for raw, and 87.34% for cellulose at 50 ppm, whereas, low for other concentrations. At higher concentrations, more $\text{Fe}(\text{II})$ ions were left unadsorbed in solution due to saturation of adsorption sites [5]. Augustine et al. (2007) [4] associated the lower metal ion concentrations with the higher percentage uptake due to the larger surface area of the adsorbent available for adsorption. This is also in line with Ekpete et al. (2011) [11], who associated an increase in concentration of the metal ions with a decrease in percentage removal, leading to less saturation of the adsorption sites. At a higher concentration of metal ions, the ratio of the initial number of moles of metal ions to the

adsorption sites available was higher, resulting in a lower adsorption percentage (Mohan & Gandhimathi, 2009) [26].

However, as the initial concentration of Fe(II) ions increased from 50 mg/L to 250 mg/L, the amount of metal ions adsorbed at equilibrium decreased. This occurred as a result of an increase in the driving power of the concentration gradient to overcome the mass transfer resistance of metal ions between aqueous and solid phases and to accelerate the likely collision of metal ions and sorbents, resulting in a greater uptake of metal ion [26].

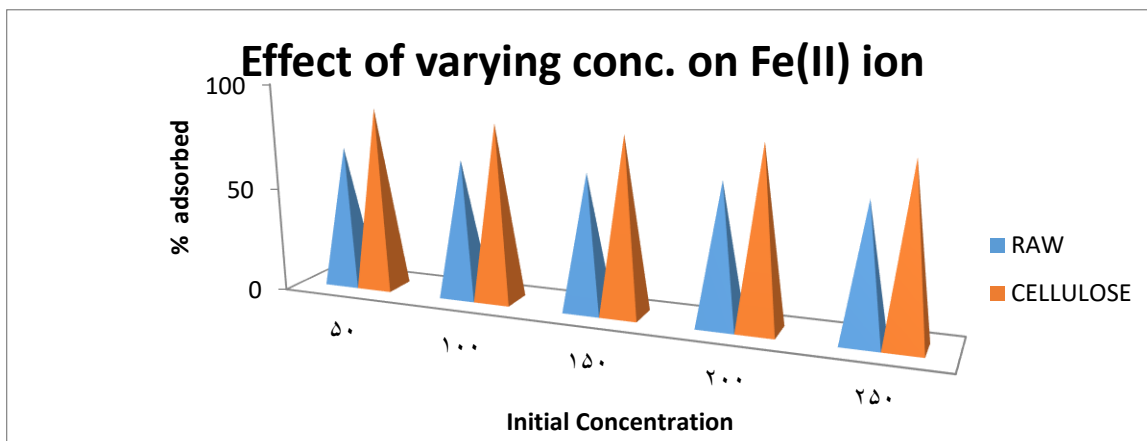


Figure 5: Effect of varying concentration of Fe²⁺ ion on *Pentaclethra macrophylla Benth* Pod Adsorbents

Adsorption Isotherm of Fe(II) Ion on *Pentaclethra macrophylla Benth* Pod

Adsorption isotherms are essential in understanding the dynamic between adsorbate molecules and surface adsorption sites (Table 3). The adsorption mechanisms of raw and isolated cellulose were elucidated using the Langmuir, Freundlich, and Temkin isothermal models. Nonlinear models of the Langmuir isotherm, the Freundlich isotherm, and the Temkin isotherm are represented by Equations (4), (5), and (6), respectively (Augustine et al. 2007; Babas et al., 2022).

$$qe = \frac{K_L q_m C_e}{(1 + K_L C_e)} \tag{4}$$

$$qe = K_F C_e^{1/n}, \tag{5}$$

$$qe = B \ln A C_e, \tag{6}$$

Adsorbate affinity to the adsorbent surface can be determined by employing the variable as follows: q_e = adsorbate amount at equilibrium (mg. g^{-1}), C_e = adsorbate concentration at equilibrium (mg. L^{-1}), K_L = Langmuir isotherm constant (mg. g^{-1}), K_F = Freundlich isotherm constant (mg. g^{-1}), n = adsorption intensity, and A = binding constant (in g^{-1}).

Table 3: Isotherms model constants of of Fe(II) Ion on *Pentaclethra macrophylla Benth* Pod

Isotherm	Isotherms constants	Raw	Cellulose
	R^2	0.8548	0.9792
Langmuir	R_L	0.5731	0.3215
	K_L	0.0149	0.3215
	$q_m, \text{mg. g}^{-1}$	39.683	62.89
Freundlich	R^2	0.9943	0.9959
	$K_F, \text{mg/g}$	1.9279	1.8599
	$1/n$	1.1282	0.7734
Temkin	R^2	0.9348	0.9513
	B	14.5380	16.046
	A	11.7705	4.4558
	K or b(J/mol)	170.420	154.404

The Langmuir constants q_{max} and b were determined from the C_e/Q_e versus C_e plot shown in Figure 6-7 with values of linear regression coefficients. From Table 1, it was observed that the interaction between the *Pentaclethra macrophylla Benth* pod adsorbents and the adsorbate (Fe(II) ions) was generally poor. However, the isolated cellulose has better interaction than the raw adsorbent. q_e can also be interpreted as the total number of binding sites that are available for adsorption, and q as the number of binding sites that are in fact occupied by the metal ions at the concentration [1].

From Table 1, isolated cellulose has the highest amount of Fe(II) ion, based on its maximum adsorbent capacity of 62.89 mg/g, which is better than raw with 39.683 mg/g. Based on the Langmuir model, the isolated cellulose adsorbents have a higher adsorption capacity than the

raw. This could be associated with an increase in the surface area of the isolated cellulose adsorbents. This is in line with [5] who worked on different adsorbents of biomass origin. From the low R_L values (Table 1) and the range of $(0 < RL < 1)$ favourable or $(RL \leq 0)$ irreversible [38]. it is observed that the adsorption of Fe(II) ions in raw and isolated cellulose has R_L values that lie between 0 and 1, which implies that the adsorption is favourable.

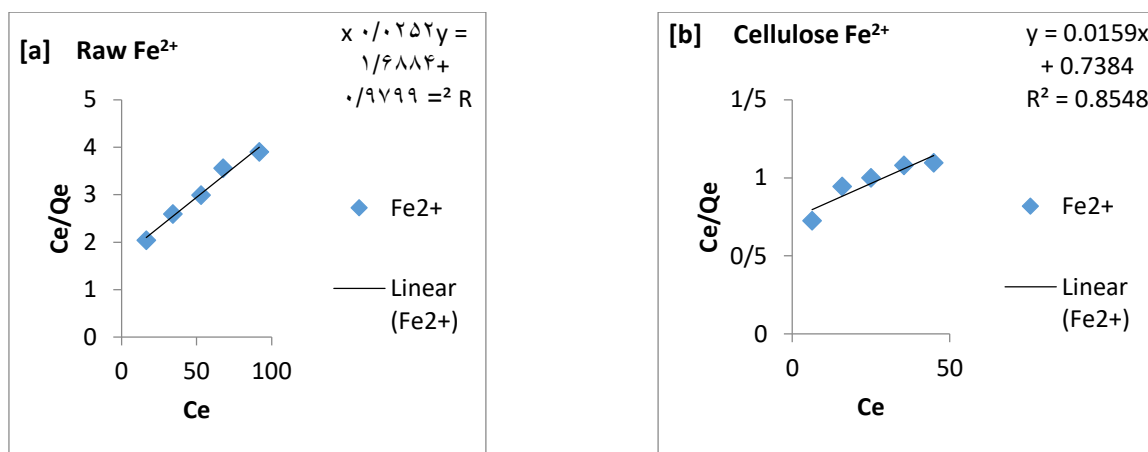


Figure 6-7: Langmuir adsorption of Fe²⁺ using *Pentaclethra macrophylla Benth* pod

The Freundlich adsorption isotherm constants K_f and $1/n$ were determined from the $\log Q_e$ versus $\log C_e$ plot shown in Figure 8–9, with values of linear regression coefficients that show the validity of the Freundlich isotherm model on the data in Table 1. The small value of K_f is a measure of the degree or strength of adsorption, and it indicates increased adsorption (Horsfall et al., 2006; Ekpete et al., 2011).

The $1/n$ values in Table 1, Fe(II) ion is present in both raw and isolated cellulose adsorbents and has high adsorption intensity and linearity, with $1/n$ values ranging from 0.7888 to 1.1282. However, the adsorption intensity due to raw Fe(II) ions has weak adsorption bonds and a decreased adsorption capacity, based on their high $1/n$ values. This implies that the isolated cellulose with a lower value of 0.7735 ($1/n < 1$) is a better adsorbent than the raw. These findings are consistent with those of [38], [18], [1], and Ekpete et al. (2011), who investigated various novel adsorbents used in heavy metal removal.

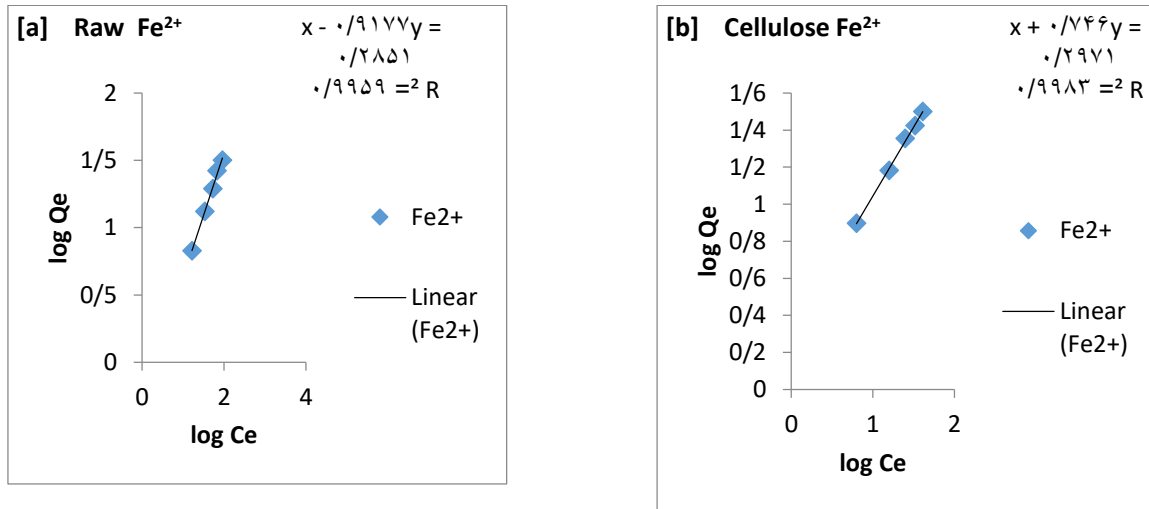


Figure 8-9: Freundlich adsorption of Fe²⁺ using *Pentaclethra macrophylla Benth* pod

The B and b were determined from the slope, whereas the A_T was obtained from the intercept. The heat of adsorption (B) of less than 40 kJ/mol indicates a physical adsorption, and its value of more than 40 kJ/mol represents a chemical adsorption [12]; [48]; [40]. The relationship between adsorbents (raw and isolated cellulose) and the metal ion Fe(II) was within the range of physical adsorption since the heat of adsorption was less than 40 kJ/mol. These findings corroborate the work of [43], who investigated Cd(II) ion adsorption using unmodified and NTA-modified *Dendrocalamus strictus* charcoal powder.

From table 4.12, the heat of adsorption for the modified is greater than for the unmodified. This implies that the modification/treatment processes were effective and produced more adsorbate than the raw adsorbents. It could also be attributed to the larger surface area of the modified adsorbents, as such having more affinity for the metal ions. The R² values for all the adsorbents and metal ions were close to unity, which implies that the model was consistent for both modified and unmodified adsorbents.

An overall consideration of the R² values of the three isotherm models used in this research reveals that the experimental data were consistent with all the models but fit more to the Freundlich and Temkin model of the Langmuir isotherm. All the R² values in both the Freundlich and Temkin models lie within the range of 0.9109–0.9992, whereas the Langmuir model has an R² value in the range of 0.8549–0.974.

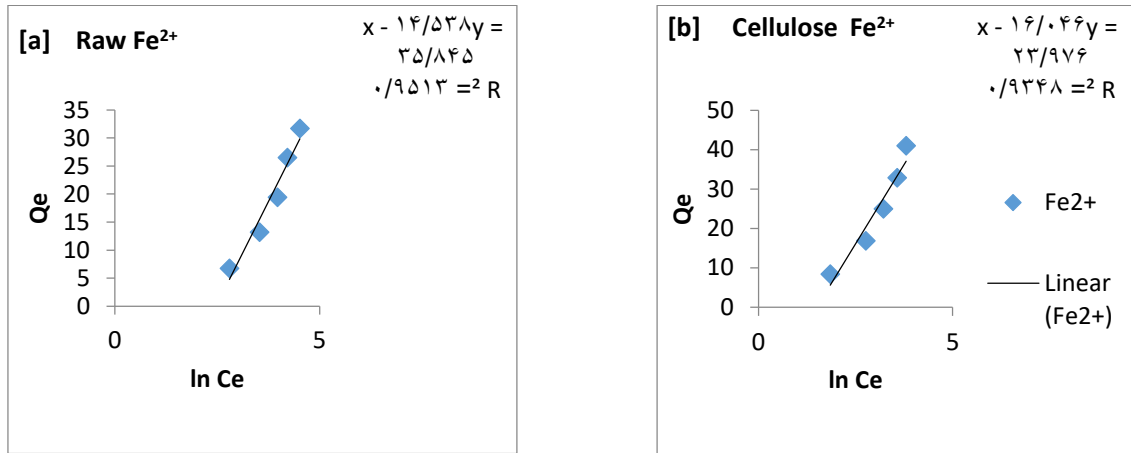


Figure 10-11: Temkin adsorption of Fe²⁺ using *Pentaclethra macrophylla Benth* pod

Thermodynamic Studies of Fe(II) ion on *Pentaclethra macrophylla Benth* pod

The adsorption effectiveness of the Fe(II) ion on raw and isolated cellulose was evaluated using the thermodynamic parameters ΔG° , ΔH° , and ΔS° (Table 4). The enthalpy change ΔH° (kJ mol⁻¹) and entropy change ΔS° (J/mol⁻¹K⁻¹) of Fe(II) ion adsorption were calculated using the slope and intercept of the plot of $\ln(K_L)$ vs $1/T$ (K⁻¹). Using Equation (7), we were able to determine Gibbs free energy, ΔG° , but the enthalpy, ΔH° , and the entropy, ΔS° were estimated with Equation (8).

$$\Delta G^\circ = -RT \ln K_L \tag{7}$$

$$\ln K_L = \Delta S^\circ/R - \Delta H^\circ/T \ (1/T) \tag{8}$$

Table 4 Thermodynamic Adsorption of Fe(II) on *Pentaclethra macrophylla Benth* pod

Adsorbents	ΔH° (- KJmol ⁻¹)	ΔS° (- Jmol ⁻¹ K ⁻¹)	R²	ΔG° (KJ mol ⁻¹)				
				303K	308K	313K	318K	323K
Raw	10.673	26.68	0.977	2.588	2.455	2.322	2.188	2.055
Cellulose	24.928	84.09	0.972	-	-	-	-	-
		6	8	1.053	1.474	1.894	2.315	2.735

K_L is a dimensionless and represents the adsorption equilibrium constant according to the best-fitted model. R is the universal constant of ideal gases, 8.314 J·K⁻¹ mol⁻¹. The negative

values of Gibbs free energy for all three metals show that the adsorption process is spontaneous and that the degree of spontaneity of the reaction increases with increasing temperature [10]. The isolated cellulose adsorbent has higher ΔG° values than the raw adsorbent. This implies that the isolated cellulose contains more adsorbate, which is shown as increased ΔG° values. Table 2 shows that an increase in temperature leads to an increase in the value of ΔG° . These observations were in line with [43] research on the removal of Cd(II) *via* unmodified and NTA-modified *Dendrocalamus strictus* charcoal powder, and [10], who worked on waste rubber tyre as an adsorbent of Pb(II), Cu(II), and Fe(II) ions.

It has been reported that, ΔG° values up to -20 kJ/mol are consistent with the electrostatic interaction between sorption sites and the metal ion (physical adsorption), while ΔG° values more negative than -40 kJ/mol involve charge sharing or transfer from the surface of the metal ion to form a coordinate bond (chemical adsorption). The ΔG° values obtained in this study for raw and isolated cellulose for all the metals investigated at the range of temperatures of 303K–323K were all less than -5 kJ/mol, which indicates that physical adsorption was the predominant mechanism in the sorption process [17].

The adsorption enthalpies of both raw have negative values (endothermic), whereas the isolated cellulose in Table 2 have negative values(exothermic in nature). The ΔH° values of the isolated cellulose for Fe(II) ions were higher than the raw. This implies that isolated cellulose contained more adsorbate than the raw at a given concentration of the adsorbent. This observed increase in ΔH° is consistent with (Mustapha et al. 2019), who worked on *Albizia lebbek* pods used in the adsorption of Pb(II), Cu(II), Cd(II), and Zinc(II). The ΔH° values analysis for this study, as shown in Table 4, reveals that isolated cellulose was a better adsorbent for Fe(II), with the ΔH° values of 24.928 KJ/mol than raw with the value of 10.673 KJ/mol.

Table 4 has negative entropy values and indicates less disorder and randomness due to an association between the adsorbent and the adsorbate during adsorption. This negative ΔS° for both raw and isolated cellulose is in line with [33], who worked on the biosorption of different metals using unmodified and modified lignin extracted from agricultural waste. From table 4, the ΔS° values for isolated cellulose adsorbent are higher than the raw. This implies that the loss of randomness is a result of an increase in the adsorbent-adsorbate interface during the adsorption process [56].

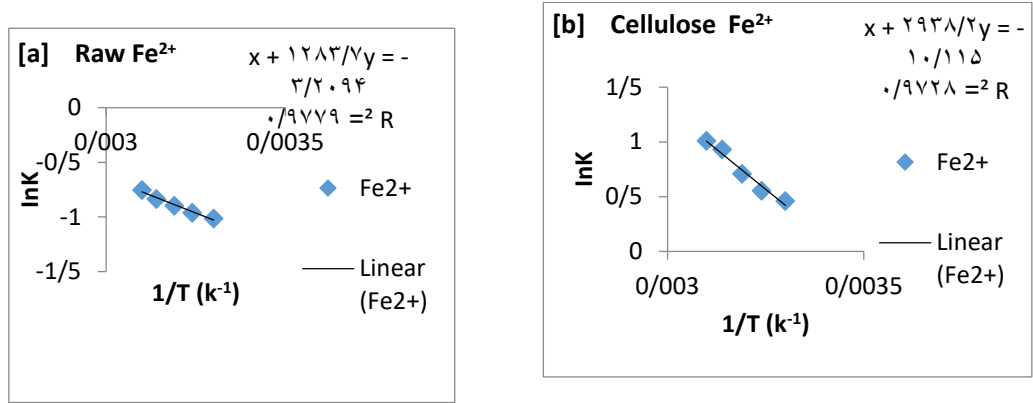


Figure 12-13: Thermodynamic study of Fe(II) on *Pentaclethra macrophylla Benth* pod

Adsorption Kinetic Studies of Fe(II) ion via *Pentaclethra macrophylla Benth* Pod

The kinetics study of Fe(II) ion on raw and cellulose *Pentaclethra macrophylla Benth* pod was investigated using pseudo-first-order, pseudo-second-order, and intraparticle diffusion kinetics models. Plotting kinetic data according to Equations (9) and (10) [14] determines whether the adsorption was first or second order.

$$qt = qe (1 - e^{-k_1t}) t, \tag{9}$$

$$qt = \frac{q^2 ek_2t}{qek_2t + 1}, \tag{10}$$

q_e is the equilibrium amount of adsorbate per unit mass of adsorbent ($\text{mg} \cdot \text{g}^{-1}$), q_t is the equilibrium amount of adsorbate per unit mass of adsorbent at time t ($\text{mg} \cdot \text{g}^{-1}$), and k_1 and k_2 are the rate constants in min^{-1} and $\text{g} \cdot \text{mg}^{-1} \cdot \text{min}^{-1}$, respectively.

For the purpose of demonstrating Fe(II) ion adsorption on raw and cellulose *Pentaclethra macrophylla Benth* pod [56], the intraparticle diffusion model given by Equation (11) was used.

$$qt = K_1 dt^{1/2} + C, \tag{11}$$

In order to define the adsorption kinetics of heavy Fe(II) ion, the kinetic parameters for the adsorption processes were studied for contact times ranging between 1 to 150 minutes, and

models were applied to experimental data. The adsorption kinetics of the Fe(II) ion is shown in Figures 14–15.

Table 5: Kinetic parameters for the adsorption on *Pentaclethra macrophylla Benth* pod

Ions	Adsorbents	First Order			Second Order			Intra Particle Diffusion		$q_{exp.}$ (mg/g)
		k_1 (min ⁻¹)	q_{cal} (mg/g)	R^2	k_2 (g/mg min ⁻¹)	q_{cal} (mg/g)	R^2	kd (mg l ⁻¹ min ^{-1/2})	R^2	
Fe ²⁺	PMBP-R	-0.0017	3.379	0.9975	0.0293	7.5358	0.9994	0.0863	0.9877	7.0809
	PMBP-C	-0.0045	1.3517	0.9981	0.0427	9.4522	0.9998	0.0922	0.9937	9.0809

The rate constant for the pseudo-first-order adsorption process (K_1) was obtained by plotting $\ln(q_e - q_t)$ versus time. The value of K_1 was extrapolated from the slope, whereas the maximum adsorbate of the adsorbent was obtained as the intercept. The graphs for the pseudo-first-order adsorption process for Fe(II) shown in Figure 14–15 were negative graphs. The comparison of the experimentally determined adsorption capacity ($q_{exp.}$) and the calculated maximum adsorbate ($q_{cal.}$) of the pseudo-first-order adsorption shows that the adsorption did not follow the pseudo first order. This is based on the wide range of differences found in the experimental and calculated values of each of the adsorbates in the PMBP adsorbent. In-line with this finding was the research of [41], who researched on the removal of cadmium using modified and unmodified carbohydrate polymers from Chitosan, and also Saini et al. (2019) [43], who worked on a comparative study for the removal of cadmium(II) ions using unmodified and NTA-modified *Dendrocalamus strictus* charcoal powder.

The pseudo-second-order adsorption rate constant (K_2) was studied with a plot of t/q vs. t . The slope was used to calculate the maximum adsorbate of the adsorbent, and the intercept was used to calculate K_2 . The experimented adsorption capacity ($q_{exp.}$) and the calculated maximum adsorbate ($q_{cal.}$) of pseudo second order gave equivalent values and the adsorbents (raw and isolated cellulose). This implies that the adsorption of Fe(II) with the adsorbents followed a pseudo-second-order adsorption process. This is consistent with Shilpi (2013) [49] and Qing (2010) [39], who researched on metal removal using natural biomass with adsorption kinetics that were achieved through pseudo-second order adsorption.

The intra-particle diffusion was observed using the relationship between specific sorption (q) and the square root of time ($t^{1/2}$). Where K_d is the rate of sorption controlled by intra particle diffusivity ($\text{mgg}^{-1} \text{min}^{-1/2}$), and C depicts the boundary layer thickness [5]. This model predicts that the plot of qt versus $t^{1/2}$ should be linear with k_d and C as slope and intercept, respectively, if intra-particle diffusivity is involved in the sorption process.

According to [19] and [5], the intra-particle diffusivity plot for sorption mechanism assumes an intra-particulate diffusivity model if the following conditions are met: (i) High R^2 values are required to determine applicability. (ii) A straight line that passes through the origin for the plot area qt versus $t^{1/2}$. (iii) Intercept $C < 0$.

Figure 18-19 show intraparticle diffusion of Fe(II) ions. From figures 18-19, the straight line did not pass through the origin, and the constant C , that is, the intercept in the graphs, is greater than zero ($C > 0$). This implies that the model does not explain the adsorption of Fe(II) ions using the *Pentaclethra macrophylla Benth* pod adsorbents. However, the presence of high correlation coefficient values of R^2 indicates the multiple adsorption stages and the suggestion that pore diffusion was involved [19].

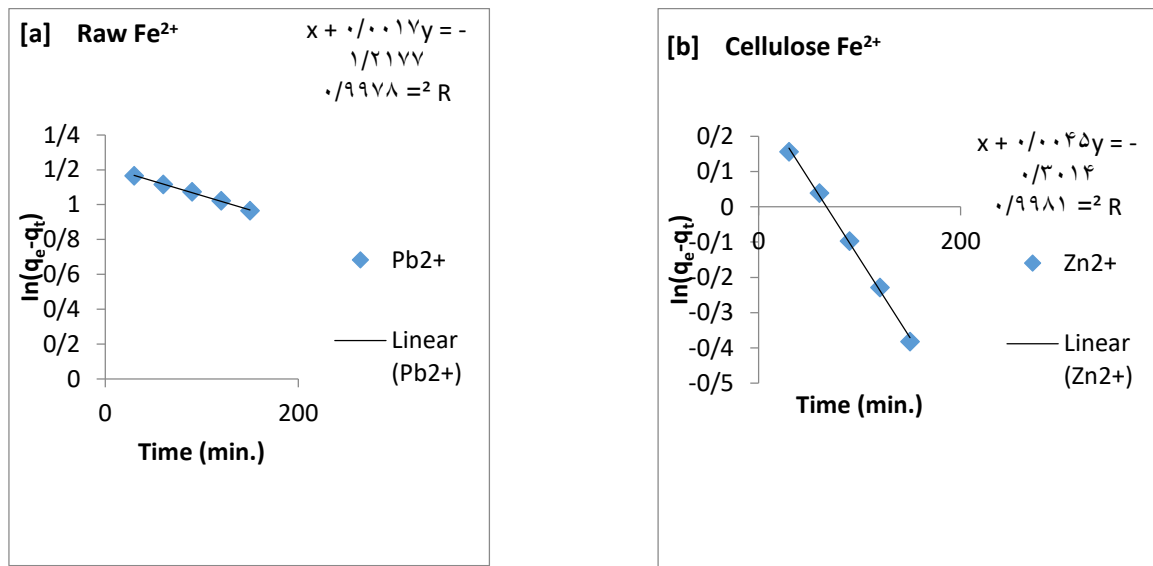


Figure 14-15: Pseudo-first-order plots for Fe(II) ion removal at different times

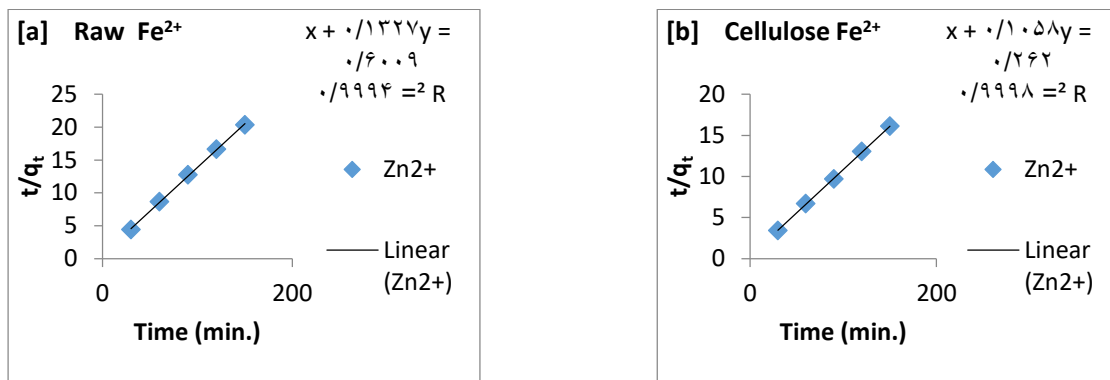


Figure 16-17: Pseudo-second-order plots for Fe(II) ion removal at different times

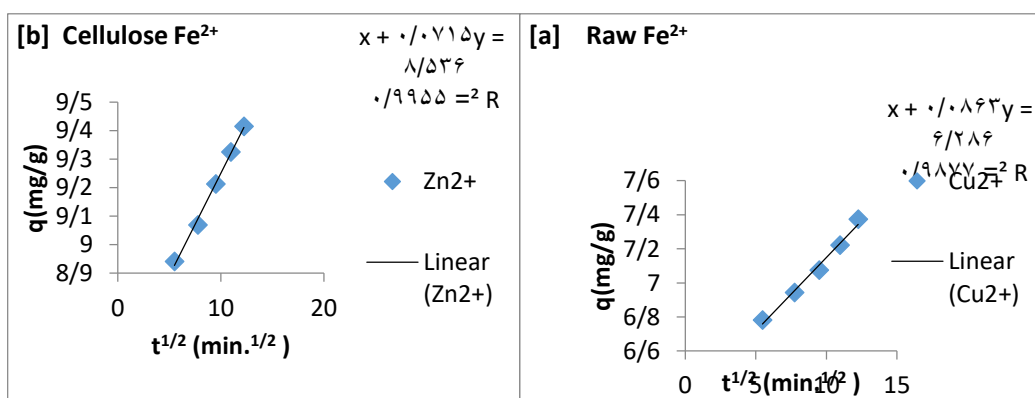


Figure 18-19: Plots for evaluating intra particle diffusion rate constant of Fe (II) ion

Conclusion

Cellulose was successfully isolated from *Pentaclethra macrophylla Benth* pod via delignification and bleaching. XRD, SEM, BET, and FTIR measurements of the isolated cellulose revealed some level of removal of hemicellulose and lignin from the raw *Pentaclethra macrophylla Benth* pod powder. The crystallinity, morphological properties, stability, and function group of both raw and isolated cellulose were compared.

The adsorption isotherms, kinetic and thermodynamic studies show that the cellulose has better Fe(II) ion removal capacity than the raw. The kinetics studies show that pseudo-second-order adsorption best explains the removal of Fe(II) ion, with an exothermic reaction for cellulose.

The result revealed that Fe(II) is successfully removed from an aqueous solution using raw and isolated cellulose. The study was based on the availability, cheapness, and non-toxic nature of *Pentaclethra macrophylla Benth* pod, and the results of the study recommend the use of cellulose for Fe(II) ion removal.

Ethics approval and consent to participate

Note applicable

Consent for publication

Not applicable

Data Availability

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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Competing Interests

The authors declare that they have "no competing interests" in this article.

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Authors' Contributions

NPO and OKJ conducted the experiment on the removal of Fe(II) from aqueous solution using characterized cellulose of *Pentaclethra macrophylla Benth* pod: adsorption, thermodynamic, and kinetic studies. The interpretation of the spectra of both raw and isolated cellulose, the adsorption study and the extrapolation of the data from the model were jointly conducted by NPO and OKJ. Both authors wrote the manuscript, proofread and approved for submission.

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