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Adsorptive Removal of Cd(II) from Aqueous Solution Using Arecanut Husk-Derived Biochar under Different Experimental Conditions

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Abstract

Heavy metals occur naturally in soil due to the weathering of parent materials. At trace levels, they are nontoxic. Cadmium is a heavy metal that is highly toxic at low levels. This study investigates the potential of arecanut husk-derived biochar as a low-cost and eco-friendly adsorbent for removing cadmium ions (Cd^{2+}) from aqueous solutions. Biochar production was carried out using a pyrolysis process. The results demonstrated that adsorption capacity (q_e) was strongly influenced by biochar dosage, contact time, solution pH, and temperature, on cadmium removal efficiency. Optimal performance was observed at a biochar dosage of 60–80 mg, contact time of 60 minutes, pH range of 6–8, and a temperature of 50°C. Under these optimal conditions, the maximum adsorption capacity was found to be 1.73 mg/g and removal efficiency was achieved up to 89.8%. FTIR analysis revealed the involvement of functional groups, including C-H, –COOH, and C=O, in the adsorption process through coordination and complexation with cadmium ions. XRD analysis confirmed the successful immobilization of Cd^{2+} via the formation of cadmium-containing crystalline phases, without altering the biochar's amorphous carbon structure. These findings indicate that chemisorption mechanisms, including surface complexation and mineral precipitation, primarily govern cadmium adsorption. To the best of our knowledge, this research study signifies a novel, acceptable, innovative method that is eco-friendly, cost-effective, and utilizes readily available materials, such as biochar derived from arecanut husk, which helps as a promising, sustainable, and efficient adsorbent for the remediation of cadmium-contaminated water.

Keywords: *bioadsorbent, biochar, heavy metal, cadmium*

1. Introduction

Heavy metal contamination has become a serious issue worldwide [1]. This is mainly due to various human activities and natural geological processes. Toxic metal content in soil and water has drastically increased beyond the permissible limits due to urbanization, industrialization, smelting, mining, overuse of agrochemicals and burning fossil fuels [2]. Through bioaccumulation, direct exposure or by suspended particles, contaminants can directly permeate the water bodies and land, leading to ill effects on natural microbial diversity as well as on other animals [3,4]. Heavy metals pose serious health dangers to both humans and animals when they build up in agricultural soils because they are absorbed by crops and make their way into the food chain. Unlike many organic pollutants, heavy metals are persistent and non-degradable, leading to long-term ecological disruption. This makes their monitoring and remediation a global priority [5,6].

Heavy metals comprise a loosely defined group of elements that include transition metals, metalloids, and certain lanthanides and actinides. Some metals, like iron, manganese, molybdenum, zinc, and copper, are essential micronutrients but can become toxic when present in excess. In contrast, metals such as cadmium (Cd), lead (Pb), mercury (Hg), and arsenic (As) have no known biological function and are extremely toxic even at low concentrations [7]. Cadmium (Cd) is a toxic heavy metal that is not essential for biological functions and creates serious environmental and public health effects due to its presence in the environment. Once they come into contact with aquatic ecosystems, cadmium can be deposited and enter the food chain, affecting various living organisms. The presence of Cd causes kidney dysfunction, liver damage, skeletal disorders, reproductive toxicity, and an increased risk of cancer [8–10].

Heavy metal contamination can be remediated using various methods, such as chemical, biological, and physical techniques. Adsorption is one of them that has drawn interest due to its effectiveness, affordability, and environmental friendliness [11]. It works even at low pollutant concentrations and depends on metal ions adhering to a solid adsorbent's surface [12]. Adsorption efficiency is significantly influenced by variables like pH, contact time, and the adsorbent's physicochemical characteristics. Graphite, clays, zeolites, activated carbon, and biochar are just a few of the many natural and artificial adsorbents that have been studied [13,14]. Due to its large surface area, porosity, and the presence of oxygen-containing functional groups that improve interactions with metal ions, biochar has drawn interest as an efficient adsorbent for heavy metal remediation [15-19].

Rice husk, coconut shell, and arecanut husk are examples of agricultural leftovers that have been successfully converted into biochar, which helps with waste management and pollution prevention [20]. Arecanut (Areca catechu) is widely cultivated in many South Asian countries, providing husks, fibres, and shells. Its lignocellulosic wastes have high carbon content and can be converted into biochar through pyrolysis, creating value-added materials for environmental applications [21]. Therefore, investigating arecanut husk biochar as a low-cost and sustainable adsorbent can contribute to both agricultural waste valorization and wastewater treatment technologies [22]. In this study, we aim to analyze the adsorption efficiency and removal efficiency of cadmium by arecanut husk biochar under varied environmental conditions. The main processes that assist in immobilizing cadmium and reducing its bioavailability include electrostatic attraction, ion exchange, surface complexation, and precipitation [23]. Although biochar derived from agricultural residues has been generally investigated for heavy metal removal, limited information is available regarding the adsorption behavior of cadmium ions onto arecanut husk-derived biochar. Previous studies have mainly focused on dye adsorption and material characterization of arecanut biomass-derived adsorbents. Therefore, a knowledge gap exists regarding the optimization of operational parameters and adsorption mechanisms governing Cd^{2+} removal using arecanut husk biochar [24]. This study states that the gap by evaluating the influence of adsorbent dosage, contact time, pH, and temperature, while elucidating adsorption mechanisms through FTIR and XRD analyses.

2. Materials and Method

2.1. Preparation of biochar from arecanut husk

2.1.1. Collection and pre-treatment of raw material

Arecanut husks (*Areca catechu*), a plentiful agricultural waste, were collected from local sources in Sri Lanka. To get rid of dust, dirt, and other surface contaminants, the biomass was carefully cleaned using distilled and deionized water. Cleaned husks were placed in an oven at 105 °C for 4 hours to eliminate the moisture content and stored in a desiccator until further use.

2.1.2. Pyrolysis process for biochar production

Cleaned and dried husks were kept in a closed porcelain crucible to limit exposure to oxygen, and then pyrolysis was done in a muffle furnace at 500 °C for 3 h. At the end of pyrolysis, the furnace was allowed to cool to room temperature, while keeping the crucible lid sealed.

2.1.3. Post-pyrolysis treatment

The cooled biochar was crushed with a mortar and pestle and sieved through a 200 µm mesh to obtain a uniform particle size biochar. Samples were demineralized by treating with HCl (1 M), followed by repeated washing with distilled water until a neutral pH was achieved. The prepared biochar was stored in airtight polyethene containers inside a desiccator to prevent moisture absorption [25].

2.2. Preparation of solutions

2.2.1. Cadmium ion stock solution

A stock solution of cadmium ions (1000 mg/L, 500 mL) was prepared by dissolving 3.4200 g of cadmium sulfate hydrate ($3CdSO_4 \cdot 8H_2O$, AR grade) in 500 mL of distilled water using an analytical balance. Desired working solutions were obtained through serial dilution.

2.2.2. Calibration standards for Cd^{2+} analysis by atomic absorption spectrometry

Cadmium stock solution was serially diluted with distilled water to create calibration standards (0.5 – 6 mg/L) for quantitative analysis. Atomic absorption spectrometry (AAS) was performed with a blank (distilled water only) for baseline correction. Before use, all operational and standard solutions were freshly prepared.

2.2.3. Effect of adsorbent dosage

Using 20 mL aliquots of 6 mg/L Cd^{2+} solution, the impact of biochar dose on cadmium ion elimination was investigated. Centrifuge tubes containing the Cd^{2+} solution was filled with precisely weighed doses of 20-120 mg biochar. The suspensions were kept at room temperature (30 ± 2 °C) for contact durations of 15, 30, and 60 minutes after being vortexed for one minute at 20 rpm. After keeping the samples in distinct time periods, mixtures were centrifuged at 3500 rpm for 3 minutes, and supernatants were filtered off through PTFE syringe filters (0.45 µm) before AAS analysis.

2.2.4. Effect of pH

Using 0.1 M NaOH or HCl, the starting pH was adjusted to a range between 3.0 and 8.0 to evaluate the impact of solution pH on adsorption. Biochar (60 mg) was added to the Cd^{2+} solution (20 mL of a 6 mg/L) at each pH value. The Cd^{2+} solutions were shaken and allowed to come into contact with biochar for 15, 30 and 60 minutes at room temperature. Samples were centrifuged and filtered as previously mentioned once they reached the equilibrium.

2.2.5. Effect of temperature

Batch adsorption tests at 20–50 °C were used to examine the impact of temperature. For every 20 mL Cd^{2+} solution (6 mg/L, pH ~6.0), 60 mg of biochar was added. After being vortexed, the tubes were kept at the appropriate temperature in a temperature-controlled system. Supernatants were separated and their residual cadmium concentration was measured when equilibrium was reached at predetermined contact durations (15–60 min).

2.3. Characterization of biochar

2.3.1. Fourier transform infrared spectroscopy

Fourier-transform infrared spectroscopy (FTIR) was employed to identify the functional groups involved in Cd^{2+} adsorption onto the biochar surface. For FTIR analysis, the biochar samples were finely ground, sieved to a particle size of 200 µm, and oven-dried at 60 °C for 4 h prior to measurement. The dried samples were placed directly onto the attenuated total reflectance (ATR) crystal for scanning. To prepare cadmium-loaded biochar, 60 mg of biochar was equilibrated with 20 mL of Cd^{2+} solution (6 mg L^{-1}, pH ~5.0) at 30 ± 2 °C. After adsorption, the biochar was separated by filtration, washed with deionized water to remove residual cadmium ions, oven-dried at 60 °C, and subjected to FTIR analysis. Spectra were recorded using a PerkinElmer Spectrum 2 FTIR spectrometer operating in ATR mode over the wavenumber range of 4000–400 cm^{-1}, with a spectral resolution of 4 cm^{-1} and 32 accumulated scans. Air scans were collected and used as the background for all measurements.

2.3.2. Powder X-ray diffraction

XRD analysis was conducted to investigate the structural characteristics of the biochar before and after Cd^{2+} adsorption. Prior to analysis, the biochar samples were ground, sieved, and packed into glass sample holders. Cadmium-loaded biochar was prepared following the same adsorption procedure used for the FTIR analysis and subsequently dried at 60 °C to remove residual moisture. The XRD measurements were performed using a Rigaku SmartLab diffractometer equipped with Cu Kα radiation. Diffraction patterns were recorded over a 2θ range of 5°–80° with a step size of 0.01° and a scanning speed of 12° min^{-1}.

2.4. Data analysis

All adsorption experiments were conducted in triplicate, and average values were reported. The adsorption capacity (q_e) was calculated using standard adsorption equations (1) - (2):

$$q_e = \frac{(C_0 - C_e) \times V}{m} \quad (1)$$

$$Removal\ efficiency\ (\%) = \frac{C_0 - C_e}{C_0} \times 100 \quad (2)$$

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 adsorbent mass (g).

3. Results and Discussion

3.1. Effect of adsorbent dosage on Cd^{2+} removal at different contact times

At three distinct contact times 15, 30, and 60 minutes the effect of biochar dose on the adsorption of cadmium ions (Cd^{2+}) was methodically examined. Figure 1 illustrates the correlation between the adsorption capacity (q_e, mg/g) and adsorbent dosage. To ensure uniformity in comparison, all tests were conducted using a fixed solution volume (20 mL) and an initial Cd^{2+} concentration of 6 mg/L.

In adsorption studies, this decreasing tendency with increasing adsorbent dosage is a well-established phenomenon. The constant amount of Cd^{2+} ions in the solution are not enough to occupy all the accessible

surface area, even though greater dosages offer more active binding sites. As a result of the total adsorption being dispersed over a greater adsorbent mass, the calculated q_e falls. The adsorption efficiency per mass of biochar may also be further decreased by particle aggregation at larger dosages, which can block active sites and decrease effective surface area [11,26].

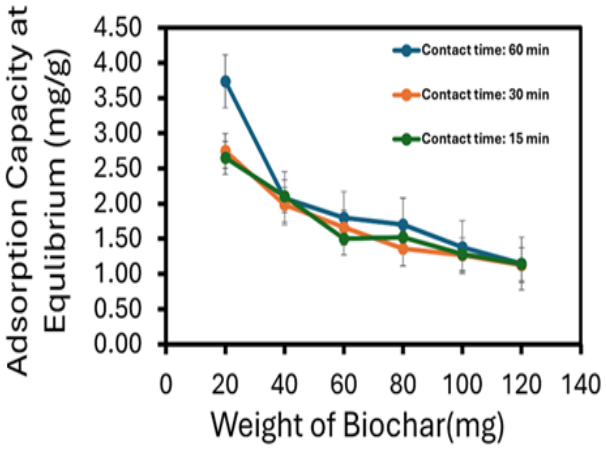


Figure 1. Graph of adsorption capacity vs. weight of biochar.

The overall removal efficiency of Cd^{2+} rose with dosage in spite of this drop in q_e. For instance, for dosages ≥80 mg, the C_e readings decreased to almost zero or even negative (below detection limit) within 60 minutes, indicating almost total removal of Cd^{2+} from solution. This implies that even if q_e drops with dosage, the system removes the most cadmium overall, which is crucial for real-world wastewater treatment applications.

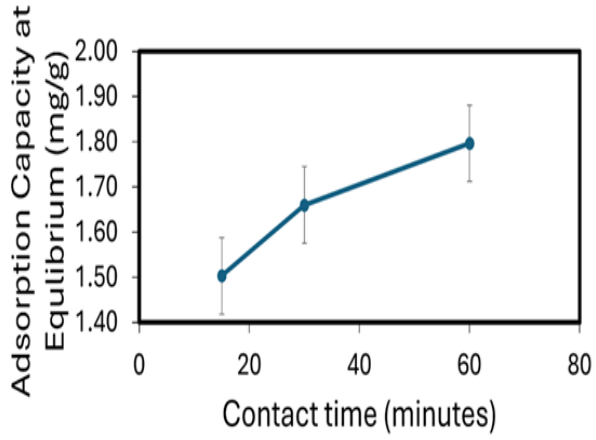


Figure 2. Graph of adsorption capacity vs. contact time.

There are noticeable variations when the impact of contact time is considered. Rapid binding onto readily accessible exterior surface sites dominates adsorption for the shortest time (15 minutes). Because a little amount of biochar quickly absorbs a significant portion of the available Cd^{2+} ions, the q_e values at low dosages are relatively high. Higher C_e values, however, show that equilibrium has not yet been reached, indicating that the process is incomplete. A considerable amount of the Cd^{2+} ions remain in solution due to the brief contact time, which restricts diffusion into the biochar's interior pores.

Adsorption continues to advance at 30 minutes, as evidenced by lower C_e values than at 15 minutes. This suggests that a larger percentage of accessible sites may take part in adsorption because Cd^{2+} ions were able to permeate deeper into the biochar's porous structure. At this point, the q_e values show a balance between the slower intraparticle diffusion process and the early fast surface adsorption. Interestingly, the q_e starts to plateau after a dosage of 60 mg, suggesting that the system is getting close to saturation.

The best adsorption took place at the 60-minute contact period, when equilibrium was almost or completely reached. C_e values were low at this point, suggesting that the majority of the Cd^{2+} ions had been effectively eliminated from the solution. However, because of the increased dilution of adsorption capacity across larger amounts of biochar, the q_e values were lower than those for shorter durations at the same dosages. Nearly every available adsorption site had interacted with Cd^{2+} ions by the time the system reached a point where the concentration gradient driving the adsorption process was reduced [26].

The findings show that adsorption efficiency is significantly influenced by both adsorbent dosage and contact time. Although the capacity per gram is diminished, greater dosages guarantee maximum removal of total Cd^{2+}, while smaller dosages produce higher qe values. Similar to this, contact duration has a significant impact on the amount of adsorption; in the conditions under study, 60 minutes is enough to achieve equilibrium. When combined, the results show that the best balance between effective adsorbent use and efficient Cd^{2+} elimination is achieved with a dosage of 60–80 mg and a contact time of 60 minutes.

These findings are in line with previously published work on the adsorption of heavy metals by biochar and other inexpensive carbonaceous adsorbents. The typical two stage adsorption mechanism, a delayed diffusion-controlled adsorption inside micropores after an initial fast absorption on the exterior surface is highlighted by the time dependent behavior. However, when developing treatment systems, the dosage-dependent trend emphasizes how crucial it is to take into account both adsorption capacity and overall removal efficiency.

3.2. Effect of pH on Cd^{2+} removal

One of the most important variables affecting adsorption processes is pH, which controls the adsorbent's surface chemistry as well as the speciation of metal ions in solution. While maintaining constant other parameters, batch adsorption studies were conducted by altering the starting pH of cadmium solutions from 3.0 to 8.0. 20 mL of a 6 mg/L Cd^{2+} solution and 60 mg of biochar were used in each system, and contact periods were 15, 30, and 60 minutes.

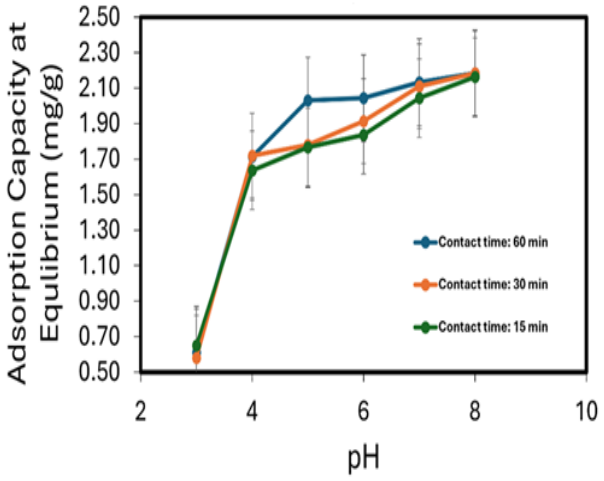


Figure 3. Graph of adsorption capacity vs pH.

The findings indicate that pH has a significant impact on cadmium adsorption (Fig. 3). With Ce levels as high as 4.0–4.3 mg/L during all contact durations, adsorption was significantly inhibited at acidic conditions (pH 3), suggesting low removal effectiveness. A significant drop in C_e was noted as the pH rose from 3 to 6, which was correlated with an increase in adsorption capacity (q_e). For instance, C_e dropped below detection limits at pH 6 in 60 minutes, suggesting almost total cadmium elimination. Adsorption peaked which shows a plateau between pH 6 to pH 8, indicates that maximal uptake had been reached and no significant further improvement taken place [27].

The adsorption of heavy metals onto biochar is characterized by this behavior. The presence of H^+ ions suppress metal binding at low pH levels by competing with Cd^{2+} ions for accessible surface sites. Furthermore, protonation of the surface functional group (–COOH) reduces their ability to coordinate with Cd^{2+}. Proton competition diminishes and these groups become deprotonated as the pH increases to moderate levels (pH 5–7), which improves complexation and electrostatic interaction with metal ions. The adsorption capacity stabilizes above pH 7, which could be explained by equilibrium being established and binding sites becoming saturated. Overall, the results show that, in line with findings for other lignocellulosic biochar, the ideal pH range for cadmium adsorption onto arecanut husk biochar is between 6 and 8 [16,26,27].

3.3. Effect of temperature on Cd^{2+} removal

Another factor that has a huge impact on adsorption is temperature, which reflects the process's kinetics and thermodynamics. Adsorption tests using a fixed Cd^{2+} concentration solution (6 mg/L and 60 mg) with 60 mg of biochar were carried out at temperatures between 20 and 50 °C. The C_e values derived from evaluating contact periods of 15, 30, and 60 minutes are shown in Table 1. A distinct trend can be seen in the data (Fig. 4): cadmium uptake was enhanced by rising temperatures. After 60 minutes at 20 °C, the residual C_e was 2.07 mg/L, which indicates a modest adsorption capability. On the other hand, C_e dropped to 0.80 mg/L at 50 °C in the same settings, suggesting a significantly higher removal efficiency. At 20 °C, the computed q_e values were around 1.48 mg/g, and at 50 °C, it increased progressively to 1.76 mg/g.

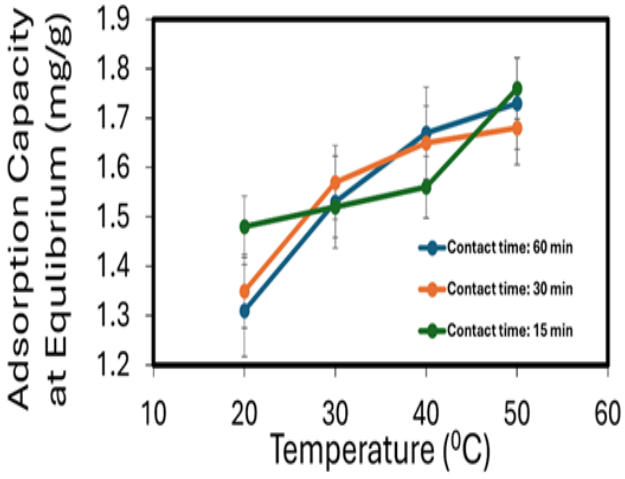


Figure 4. Graph of adsorption capacity vs temperature.

The mobility and diffusion of Cd^{2+} ions into the pores of biochar are enhanced by higher temperatures, which give them more kinetic energy. Additionally, higher temperatures might make surface functional groups more flexible, which would enable greater interactions with metal ions. At shorter contact times, the effect of temperature was most noticeable. Higher temperatures achieved the equilibrium at less time, as seen by the significant difference in C_e between 20 °C and 50 °C after just 15 minutes. The effect of temperature, however, decreased after 60 minutes, suggesting that adsorption sites were getting close to saturation regardless of temperature. These findings demonstrate that cadmium

adsorption onto arecanut husk biochar is thermodynamically favored at higher temperatures. [15,28].

Table 1. Final concentration at equilibrium obtained from AAS for temperature variation.

Temperature (°C)	C_e (mg/L)		
	60 min	30 min	15 mi
20	2.07	1.95	1.55
30	1.4	1.29	1.45
40	0.99	1.05	1.32
50	0.8	0.95	0.73

3.4. Characterization of biochar by FTIR

To investigate the surface chemistry of biochar made from arecanut husks and to pinpoint the functional groups involved in cadmium binding, Fourier Transform Infrared (FTIR) spectroscopy was utilized. ATR mode was used to record spectra in the 4000–400 cm^{-1} range, and band assignments were determined using existing literature. Figure 5 illustrates the pure biochar's multiple distinctive peaks. It was determined that C–H vibrations, most likely from leftover cellulose were responsible for a faint band at about 1400–1500 cm^{-1}. A noticeable absorption at about 1700 cm^{-1} suggested the presence of ketone or carboxylic acid groups due to C=O stretching vibrations. Ether or ester connections were represented by peaks between 1200 and 1000 cm^{-1}, which were attributed to C–O stretching.

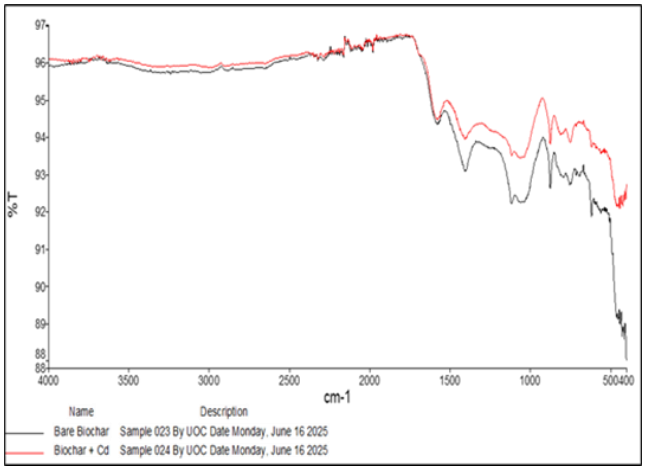


Figure 5. FTIR spectrum image for biochar & biochar loaded with cadmium Ion.

There were noticeable spectrum alterations following the adsorption of Cd^{2+}. Coordination between carbonyl groups and cadmium ions was shown by the change of the C=O stretching peak from about 1700 cm^{-1} to approximately 1685 cm^{-1}. The C–O stretching region also displayed modest fluctuations and decreased intensity, which further suggests that oxygenated groups play a role in metal binding. The consumption of active sites during adsorption is shown in the general decrease in peak intensities, especially in the C=O region. These findings demonstrate that, while the overall structure of the biochar stayed intact, Cd^{2+} uptake mostly happens through chemisorption processes involving complexation with carbonyl and carboxyl groups on the surface of the biochar.

3.5. Characterization of biochar by XRD

Additional information about the biochar's structural characteristics was revealed by X-ray diffraction (XRD) research. The pure biochar's XRD pattern revealed a large hump in the 2θ range of 15° to 30°, which is typical of amorphous carbon domains created when lignocellulosic biomass is pyrolyzed. Small crystalline peaks were also seen, such as signals at 40° and 50–75°, which are indicative of mineral phases like calcite that are incorporated in the matrix of biochar. A heterogeneous surface structure that can support ion-exchange interactions as well as physical adsorption is suggested by the coexistence of crystalline minerals and amorphous carbon.

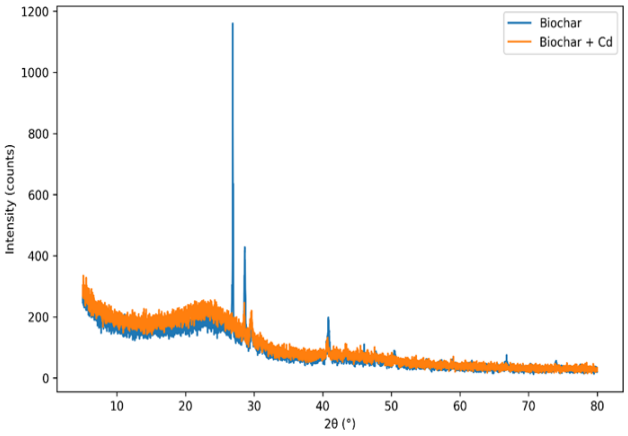


Figure 6. XRD patterns of arecanut husk-derived biochar before and after Cd^{2+} adsorption. The comparison demonstrates changes in diffraction peak intensity while preserving the amorphous carbon structure following cadmium adsorption.

Cadmium-loaded biochar's XRD pattern showed clear alterations. After adsorption, the broad amorphous peak was still discernible, indicating that the biochar's basic carbon backbone was retained. But the peak's intensity increased at 40°, perhaps as a result of crystalline phases containing cadmium forming on the surface of the biochar. This suggests that precipitation or assimilation into mineral phases, in addition to surface complexation, may be involved in cadmium adsorption. The stability of biochar during adsorption and its ability to immobilize cadmium through a variety of methods are highlighted by the preservation of the amorphous carbon structure and the appearance of crystalline signals connected to cadmium [19,20].

Table 2. Comparison of Cd^{2+} adsorption performance of arecanut husk-derived biochar with previously reported biochar adsorbents.

Adsorbent	Target Metal	Removal Efficiency (%)	Key Findings	Reference
Peanut hull biochar	Cd^{2+}	94.9	Rapid Cd^{2+} removal within 15 min; adsorption followed pseudo-second-order kinetics	[26]
Wheat straw biochar	Cd^{2+}	98.0	High Cd^{2+} removal efficiency and strong adsorption performance	[26]
Coconut husk biochar	Cd^{2+}	10.79 –24.88	Adsorption affected by temperature and contact time	[29]
Arecanut husk-derived biochar	Cd^{2+}	89.8	Effective Cd^{2+} removal under optimized pH, dosage, temperature, and contact time conditions; adsorption governed by surface complexation and mineral precipitation	Present Study

4. Conclusion

This study showed how well Areca nut husk biochar works as an inexpensive, environmentally friendly adsorbent to remove cadmium ions (Cd^{2+}) from aqueous solutions. The importance of pH, temperature, and contact duration in affecting adsorption performance was validated by batch adsorption tests. Due to decreased proton competition and the deprotonation of functional groups like -OH and -COOH on the surface of the biochar, the cadmium adsorption capacity rose dramatically from pH 3 to pH 6. The adsorption capacity plateaued above pH 6, suggesting that the adsorption sites were almost saturated. This demonstrates that the ideal pH range for the elimination of Cd^{2+} is between 6 and 8.

The adsorption capability improved with temperature, especially between 20 and 50 degrees Celsius, according to temperature experiments. This suggests that higher temperatures improve the interaction between the surface of the biochar and the cadmium ions. Shorter contact periods resulted in better adsorption, although equilibrium was only reached after 60 minutes, after which temperature had only a slight impact. Under these optimal conditions, the maximum adsorption capacity was found to be 1.73 mg/g and removal efficiency was achieved up to 89.8%. The function of functional groups and mineral phases in cadmium binding was validated by spectroscopic and structural characterizations utilizing FTIR and XRD. While XRD study showed that the amorphous carbon backbone of biochar was preserved along with the production of crystalline phases following cadmium adsorption, FTIR data showed that carbonyl and oxygenated groups actively participated in complexation with Cd^{2+} ions. This offers compelling proof that chemisorption and surface precipitation mechanisms are both involved in adsorption. In conclusion, because of its good adsorption capability, stability, and surface reactivity, areca nut husk biochar shows prominent activity in cadmium remediation. The results highlight its use as an economical and ecologically friendly adsorbent for the removal of Cd^{2+} in wastewater treatment.

Author contributions

Kuganesan Mithula: Data curation, Formal analysis, Investigation, Visualization, Writing – original draft. **Dulith Abeykoon:** Funding acquisition, Project administration, Supervision, Writing – review & editing. **Tharindra Weerakoon:** Conceptualization, Funding acquisition, Project administration, Supervision, Writing – review & editing.

Conflicts of interest

There are no conflicts to declare.

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