Development of Corncob-Based Biochar-Cellulosic Matrix for Heavy Metal Sequestration from Wastewater

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Abstract
Freshwater contamination by heavy metals poses significant threats to human health and the ecosystem. This research explores the use of corncob-based biochar-cellulosic matrix for remediation of heavy metals from wastewater. Biochar-cellulosic composites were prepared in varying ratios (0:1, 1:2, 2:3, and 1:1) of biochar to cellulose. The physicochemical properties of wastewater and heavy metal (lead, copper, zinc, chromium and cadmium) quantification were determined using standard procedures. Acid hydrolysis and alkaline treatment was adopted for cellulose extraction while the biochar was produced through pyrolysis of corncob. The functional groups of the matrix were characterized using FTIR spectroscopy. The result showed that extraction of cellulose from corncob had a yielded of 33.65 %. Treatment of wastewater with the matrix resulted in significant improvements in water quality parameters, with reduction in turbidity from 423.0 NTU to 78.0 NTU, total dissolved solid (TDS) decreased from 60.6 to 35.5 mg/l, conductivity reduced from 49.2 to 39.3 nS/cm, while maintaining pH levels between 7.6-8.3 and temperature ranged between 28.0-29.5 °C. Wastewater treatment with the matrix also demonstrated significant reduction in heavy metal concentration. Lead decreased by 94.74 %, copper concentration was reduced by 58.33 %, zinc concentration decreased by 48.11-64.15 %. Furthermore, chromium concentration in wastewater reduced by 77.78 % while 42.86-85.71 % reduction was observed for cadmium. These findings demonstrated that corncob-based biochar-cellulosic matrix, particularly at a 1:1 ratio, offers a promising, sustainable solution for wastewater remediation, effectively removing heavy metals while improving overall water quality parameters.

Keywords: Cellulose, Biochar, Heavy metals, Wastewater, Corncob

Introduction

The increasing demand for sustainable and efficient wastewater treatment methods has led to the exploration of various materials and technologies. Among these, biochar has gained significant attention due to its potential for environmental remediation. Biochar, a carbon-rich material derived from the pyrolysis of organic matter, has been widely studied for its ability to adsorb contaminants from water (Lehmann and Joseph, 2015). Corncob, an agricultural waste product, is an abundant and renewable resource that can be converted into biochar through pyrolysis. The use of corncob for biochar production not only provides a sustainable waste management solution but also enhances the economic value of agricultural residues (Rattanaphaiboon *et al*., 2022). The effectiveness of biochar in wastewater treatment is largely dependent on its surface area, porosity, and functional groups. For instance, biochar produced at 500°C has shown high adsorption capacities for lead (Pb) and chromium (Cr), making it a promising material for heavy metal remediation (Lee *et al*., 2013). These properties can be tailored by optimizing the pyrolysis conditions, such as temperature and residence time (Drané *et al*., 2024). Higher pyrolysis temperatures generally increase the surface area and porosity of biochar, enhancing its adsorption capacity (Shakya and Agarwal, 2023). Incorporation of cellulosic materials into the biochar matrix can further improve its structural integrity and adsorption efficiency (Divyangkumar *et al*., 2024). Moreover, the modification of biochar with cellulosic materials can enhance its mechanical strength and stability, making it more effective in dynamic water treatment systems (Basu, 2018). This study was therefore aimed at developing a matrix with enhanced adsorption properties for wastewater remediation. This study highlights the potential of corncob-based biochar-cellulosic matrices as a viable solution for wastewater remediation, paving the way for future advancements in this field.

Materials and Methods

Biochar Production

The corn cobs were cleaned to remove dirt, debris, and foreign materials. They were then dried in an oven at 60°C for 24 hours. The corn cobs were then loaded into a pyrolysis reactor. The pyrolysis temperature was then set to 500 °C and left to run for 2 hours. The pyrolysis reactor was then left to cool and the biochar removed from the reactor. The biochar was then pulverized and stored in an airtight bag to prevent moisture absorption.

Cellulose Extraction from Corn Cob

The cellulose extraction was carried according to the method of …….. (Ref) . Corn cobs were first cleaned, dried, and ground into a fine powder which was then subjected to acid hydrolysis using dilute sulfuric acid (H_2SO_4) at a concentration of 2 % (v/v) with a solid-to-liquid ratio of 1:10 (w/v) at 120 °C for 60 minutes in an autoclave. The resulting slurry was then cooled and filtered. The solid residue was washed thoroughly with distilled water and its pH level measured using a pH meter until neutral pH was achieved. The acid-treated material was then subjected to bleaching using 4 % NaOH at 70°C for 2 hours with a solid-to-liquid ratio of 1:10 (w/v). The bleached pulp was again washed with distilled water until neutrality and oven dried at 60°C for 24 hours. Cellulose was calculated as the ratio of the dry weight of extracted cellulose to the initial dry weight of corn cob powder multiplied by 100 (Sharma *et al.*, 2020).

Cellulose yield calculation

$$Cellulose\ yield = \frac{dry\ weight\ of\ extracted\ cellulose}{initial\ dry\ weight\ of\ corn\ cob\ powder} \times 100 \qquad \text{………… 1}$$

Biochar-cellulose matrix preparation

Preparation of cellulose solution

5 g Five grams (5 g) of cellulose was weighed using an analytical balance was gradually added into a 250 mL beaker containing100 mL of distilled water . while stirring with a magnetic stirrer at 600 rpm. The stirring was done for 2 hours at room temperature to ensure complete dissolution.

Incorporation of biochar into cellulose matrix

A series of biochar-cellulose mixtures with ratio 1:0, 1:2, 2:3 and 1:1 was prepared by weighing 2.5g, 3.33g and 5g of biochar powder was added to the cellulose solution in small increments (approximately 1 g at a time). Using a magnetic stirrer at 5000 rpm for 5 minutes after each addition to ensure even distribution. Continue this process until all the biochar is incorporated.

Casting the matrix

20 mL of the biochar-cellulose mixture was measured using a graduated cylinder. The measured mixture was poured into a glass Petri dish, a stainless-steel spreading tool was used to distribute the mixture uniformly across the dish. The Petri dishes were placed in a water bath at 60°C (±1°C). The samples were left to dry for until constant weight was achieved. The dried matrices were allowed to cool at room temperature.

Water absorbing capacity

The biochar-cellulosic matrix samples were cut into 2 cm × 2 cm pieces and dried in an oven at 105°C for 24 hours. After cooling in a desiccator for 30 minutes, the initial weight (W_0) of each sample were recorded using an analytical balance. The samples were immersed in distilled water at room temperature (23 ± 2°C) for 24 hours. After immersion, samples were removed and gently blotted with filter paper to remove surface water. The final weight (W_t) was recorded immediately.

The water absorbing capacity was calculated using the equation.

$$WAC = \frac{Weight\ of\ Hydrated\ Residue - Initial\ Dry\ Weight}{Initial\ Dry\ Weight} \times 100 \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots 2$$

Solubility test

A 1 g sample of the matrix was weighed and placed in a glass beaker containing 100 mL of distilled water at room temperature (25±2°C). The mixture was gently stirred at 200 rpm for 30 minutes using a magnetic stirrer. After stirring, the solution was filtered through Whatman No. 1 filter paper, and the undissolved material was collected and dried in an oven at 105°C for 3 hours until constant weight was achieved.

The solubility percentage was calculated using the equation

$$Solubility\ (\%) = \frac{Initial\ Weight - Final\ Weight\ of\ Insoluble\ Fraction}{Initial\ Weight} \times 100 \ \ldots\ldots\ldots 3$$

Moisture content

The moisture content of the biochar-cellulosic matrix samples was determined using the oven-drying method. Approximately 2 g of each sample was weighed accurately in pre-dried aluminum dishes. The samples were then placed in a convection oven at 105°C for 24 hours to ensure complete moisture removal. After drying, the samples were cooled in a desiccator to room temperature and reweighed. The moisture content was calculated as the percentage of weight loss relative to the initial sample weight. This process was repeated in triplicate for each sample to ensure accuracy and reproducibility of results. The average moisture content was then calculated and reported for each biochar-cellulosic matrix composition.

$$Moisture\ content\ (\%) = \frac{(W2 - W3)}{(W2 - W1)} \times 100 \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots 4$$

Where; W1= Weight of the empty container

W2= Weight of the container with the wet sample
W3= Weight of the container with the dried sample

Treatment of Wastewater with Biochar-cellulosic Matrix
Wastewater which was gotten from was treated with the varying ratio (1:0, 1:2, 2:3 and 1:1) of biochar cellulosic matrix. 1g of each sample was weighed and added into 200ml of wastewater sample, then left to stand for 2 hours, after which the wastewater samples were decanted.

Heavy Metal Analysis
Heavy metals analysis was carried out on the wastewater samples which were treated with the developed biochar-cellulosic composite. The wastewater samples were digested by adding 5ml of 99% concentrated HCl to 50 ml of each wastewater sample and placed on a hot plate and left to vaporize to give 25 ml. This step was repeated for all samples including the blank sample. The wastewater samples were then filtered using a filter paper and funnel into a wash bottle. The samples were then aspirated into a flame atomic absorption spectrometer, where absorbance was measured and compared against known metals concentration.

Water quality parameters analysis
Water quality parameters were measured using standard analytical methods. The pH was determined using a calibrated digital pH meter (Model XYZ) with automatic temperature compensation. Total Dissolved Solids (TDS) and electrical conductivity were measured using a multiparameter meter (Model ABC), which was calibrated using standard KCl solution prior to measurements. Temperature readings were taken simultaneously using the temperature sensor integrated within the multiparameter meter. Turbidity was measured using a calibrated turbidity meter (Model DEF??) and expressed in Nephelometric Turbidity Units (NTU). The probes were rinsed with distilled water and dried between measurements to prevent cross-contamination.

Fourier-transformed infrared spectroscopy (FT-IR)
The developed matrices were prepared for FT-IR by crushing them into powder which were then embedded in KBr pellets and analysed using Agilent 620 FTIR spectrophotometer. The spectra were recorded in the absorption band range of 4000 – 400cm-1 $^{-1}$, with a spectral resolution of 4 cm^{-1}.

RESULT AND DISCUSSION
Result
Cellulose yield
The cellulose weight gotten after extraction was 84.13 g from 250 g of corn cob sample. The cellulosic yield from corncob was 33.65%

FT-IR result
The FT-IR result is presented in Table 1. The FT-IR spectra for four samples Sample A, Sample B, Sample C and Sample D were obtained and analyzed for characteristic absorption bands. The key peaks and their assignments are summarized in Table 1.

The FT-IR spectra of all four cellulose samples revealed common absorption bands corresponding to key functional groups in cellulose. The presence of strong absorption peaks at 849 cm^{-1} and 1103 cm^{-1} across all samples indicates that the β-(1→4)-glycosidic linkages are preserved, confirming the cellulose structure. The broad absorption band around 3280 cm^{-1}, associated with O-H stretching vibrations, suggests that all samples have extensive hydrogen bonding, characteristic of cellulose's hydroxyl groups. This also reflects the hydrophilic nature of cellulose.

However, notable differences were observed between the samples. The 1423 cm^{-1} peak, indicative of crystalline cellulose, was more intense in Sample A (67.95) compared to Sample D (65.45) and Sample C (62.95), suggesting that Sample A has a higher degree of crystallinity. This could imply that Sample A has undergone less processing or is more structurally ordered than the other two samples.

Additionally, Sample B, C and D exhibited a distinct peak at 2117 cm^{-1}, which was absent in Sample A. This peak could correspond to the presence of impurities or chemical modifications, such as carboxyl groups (C≡O), possibly introduced during the extraction or processing of the cellulose. The high intensity of this peak in Sample B (90.58), Sample C (92.03) and Sample D (91.31) suggests that these samples may have been subjected to similar treatment processes or contain similar impurities. The O-H bending peak at 1640 cm^{-1}, which indicates absorbed water, was slightly higher in Sample A (77.86) compared to Sample B (70.63), Sample C (72.52) and Sample D (73.67). This suggests that Sample A may have a higher moisture content, which could be due to differences in storage conditions or sample handling. Overall, the FTIR analysis confirms that all four samples are primarily composed of cellulose, but varying crystallinity, moisture content, and potential impurities distinguish them.

Table 1: FT-IR result for developed biochar-cellulosic composite

Wavenumber (cm^{-1})	**Sample A (0:1) intensity**	**Sample B (1:1) intensity**	**Sample C (1:2) intensity**	**Sample D (2:3) intensity**	**Functional Group Assignment**
849	62.64	54.29	56.15	57.69	C-O-C stretching β-glycosidic linkages
1028	20.33	22.15	21.35	21.28	C-O stretching (Carbohydrates)
1103	46.37	45.68	45.44	45.83	C-O-C stretching β-glycosidic linkages
1319	64.55	59.20	60.07	61.27	CH_2 bending (Crystallinity)
1423	67.95	-	62.95	65.45	CH2 bending (Crystallinity)
1640	77.86	70.63	72.52	73.67	O-H bending (Water)
2117	-	90.58	92.03	91.31	Biochar functional group
2884	72.22	67.46	68.16	69.28	C-H stretching (Aliphatic)
3280	42.70	42.34	42.73	42.59	O-H stretching (Hydrogen bonding)

Functional properties of the developed corncob-based biochar matrices

Table 2 shows the water absorbing capacity (WAC), solubility, moisture content and swelling capacity of the developed matrices. The water absorbing capacity of the 1:1 matrix proved to be the highest with a value of 300 % with the 0:1 matrix having a value of 232 %. The solubility of the developed matrix was highest in the 2:3 matrix with a value of 0.79 %, moisture content of the 1:2 matrix was the highest with 9.9 % being the highest value among the developed matrices. The swelling capacity of the 1:2 matrix was also the highest with a value of 24.33%.

Table 2: Physical Properties of the Developed Matrices

Parameters	A (0:1)	B (1:2)	C (2:3)	D (1:1)
WAC (%)	232	255	270	300
Solubility (%)	0.57	0.63	0.79	0.73
Moisture Content (%)	9.1	9.9	9.6	9.7
Swelling capacity (%)	22	24.33	18.33	16

Physicochemical result of treated wastewater

Table 2 shows the tested physicochemical result of the wastewater before and after treatment with the developed biochar-cellulose matrix. The pH result for all the water samples was within the accepted range of NSDWQ standard. Turbidity for all the samples were above the NSDWQ standard, however, the biochar-cellulose matrix showed high reduction from the untreated wastewater with the 1:1 ratio having a value of 78.0 NTU compared to the untreated water with 423.0 NTU. Total dissolved solids (TDS) in the wastewater samples were highest in the untreated water sample with value of 60.6 mg/L and the 1:1 sample having a value of 35.5 mg/L. Conductivity for the untreated water sample measured at 49.2 nS/cm while the 1:1 sample had the least value at 39.3 nS/cm.

Table 3: Physicochemical Properties of the Corncob-based Biochar-cellulosic Matrix Treated Wastewater

Samples	**pH**	**Temperature (°C)**	**Turbidity (NTU)**	**TDS (mg/l)**	**Conductivity (nS/cm)**
Raw Water	7.7	28.0	423.0	60.6	49.2
A (0:1)	8.3	29.1	356.0	53.2	44.6
B (1:2)	8.3	29.5	138.0	45.9	42.4
C (2:3)	7.6	29.4	105.0	40.6	40.7
D (1:1)	8.1	29.4	78.0	35.5	39.3
NSDWQ	6.5-8.5	Ambient	5.0	500	1000

NSDWQ: Nigerian Standard for Drinking Water Quality

Heavy metals adsorption

The results in Table shows the percentage adsorption of heavy metals by the developed matrices. For zinc, the 0:1 matrix proved to be more effective removing 64.15% of zinc from the wastewater, 2:3 matrix effectively removed 85.71% of cadmium significantly higher than other matrices, 58.33% of copper was removed by both the 1:2 and 1:1 matrix as they proved the most effective for copper removal from wastewater, lead adsorption by the 1:1 matrix was seen to be highly effective at 94.74% while chromium was significantly adsorbed by the 1:2 matrix.

Table 4: Heavy Metals Adsorption by the Developed Matrices

Sample	**Zinc (%)**	**Cadmium (%)**	**Copper (%)**	**Lead (%)**	**Chromium (%)**
A (0:1)	64.15	57.14	33.33	42.11	40.74
B (1:2)	51.89	57.14	58.33	84.21	77.78
C (2:3)	63.21	85.71	33.33	36.84	72.22
D (1:1)	48.11	57.14	58.33	94.74	75.93

Discussion

The need for effective wastewater treatment solutions is critical, given increasing environmental concerns about heavy metal pollution and water contamination (Bhatnagar & Sillanpää, 2017). Corncob-based biochar combined with cellulosic matrices represents a promising approach to address these issues. Biochar, a carbon-rich material obtained through pyrolysis of biomass, is particularly effective in adsorbing pollutants due to its high surface area and porous structure (Ahmad et al., 2014). Additionally, cellulose provides a hydrophilic, renewable matrix that enhances biochar's structural stability and adsorption potential, making it an ideal candidate for wastewater treatment applications.

In the study provided, corncob-based biochar was synthesized and incorporated into a cellulosic matrix to create a composite material for wastewater treatment. This biochar-cellulosic matrix was then assessed for its ability to reduce various physicochemical parameters such as turbidity, total dissolved solids (TDS), and conductivity in wastewater, as well as to remove heavy metals like zinc, cadmium, copper, lead, and chromium. Results indicated that the composite's ratio of biochar to cellulose significantly impacted its efficacy in pollutant removal.

For instance, the sample with a 1:1 biochar-cellulose ratio showed the highest reduction in turbidity (78.0 NTU) and TDS (35.5 mg/L), as compared to untreated water, which had values of 423.0 NTU and 60.6 mg/L, respectively. This decrease in turbidity and TDS suggests that biochar-cellulosic matrices are effective at capturing suspended solids and dissolved contaminants, aligning with findings by Aveling (2020), who observed a similar reduction in water turbidity when using biochar composites.

In terms of heavy metal adsorption, different matrix compositions exhibited varying levels of adsorption efficiency. The 2:3 biochar-cellulose matrix achieved the highest cadmium removal at 85.71%, while the 1:1 matrix was particularly effective at adsorbing lead, with a removal efficiency of 94.74%. This pattern of selective heavy metal adsorption reflects the findings of Enaime et al. (2020), who noted that the adsorption potential of biochar can vary depending on the type of heavy metal and biochar composition.

The adsorption capabilities of biochar-cellulosic matrices can be attributed to several mechanisms. Firstly, the presence of carboxyl and hydroxyl groups on the biochar surface facilitates ion exchange with metal ions in wastewater, enhancing adsorption (Li et al., 2018). The high porosity of biochar further allows for physical adsorption, as contaminants are retained in the biochar's micropores. This aligns with observations by Lyu et al. (2020), who found that biochar's microporous structure plays a crucial role in capturing and retaining pollutants.

Furthermore, Fourier-transform infrared spectroscopy (FT-IR) analysis revealed the functional groups present in the biochar-cellulosic composite. Peaks associated with β-(1→4)-glycosidic linkages and O-H stretching vibrations confirm the preservation of cellulose's structure and the presence of hydrogen bonding, indicating a stable and hydrophilic matrix. The 2117 cm^{-1} peak observed in some samples suggests chemical modifications or impurities, possibly enhancing the composite's reactivity and adsorption potential (Tran et al., 2021).

The functional properties of the developed matrices, such as water absorbing capacity, solubility, moisture content, and swelling capacity, are crucial for their performance in wastewater treatment. The high-water absorbing capacity of the 1:1 matrix (300%) and the high moisture content of the 1:2 matrix (9.9%) suggest that these matrices are highly effective in retaining water and adsorbing contaminants. This is in line with the observations of Aljeboree et al. (2021), who noted that biochar's physicochemical properties enhance its adsorption capabilities.

Compared to traditional adsorbents such as activated carbon, corncob-based biochar-cellulosic matrices offer a more sustainable and cost-effective solution. While activated carbon is highly effective in pollutant adsorption, its production is energy-intensive, and it can be expensive (Yuan et al., 2018). In contrast, corncob-based biochar utilizes agricultural waste, making it an environmentally friendly alternative. Furthermore, the addition of cellulose enhances the mechanical stability and reusability of biochar, potentially lowering treatment costs.

The performance of corncob biochar-cellulosic composites also aligns with other studies on agricultural waste biochars, such as rice husk or bamboo, which have shown promising results in heavy metal adsorption. However, corncob-based biochar-cellulosic matrices exhibit unique properties that make them particularly suitable for industrial wastewater treatment applications due to their superior adsorption capacity for specific metals and ease of modification (Duan et al., 2020).

Conclusion

Corncob-based biochar-cellulosic matrix demonstrate substantial potential as a sustainable solution for wastewater treatment. They effectively reduce physicochemical contaminants and adsorb heavy metals, offering a viable alternative to more costly and environmentally taxing treatment methods. This approach could significantly improve water quality and contribute to sustainable wastewater management.

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