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Synthesis of Cellulose-Based Nanomaterials Agricultural Waste for Heavy Metal Adsorption in Water Applications

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ABSTRACT

Heavy metal contamination in aquatic systems drives the need for sustainable and high-performance adsorbents. This study developed functionalized nanocellulose materials derived from rice husk waste for efficient removal of Pb(II), Cd(II), Cu(II), and Cr(VI) from aqueous solutions. Cellulose nanocrystals (CNCs) were synthesized via sulfuric acid hydrolysis and functionalized through TEMPO-mediated oxidation to introduce carboxyl groups, while cellulose nanofibrils (CNFs) were modified by ethylenediamine grafting to incorporate amine functionalities. Characterization using FTIR, XRD, and SEM confirmed successful surface modification, reduced crystallinity, and the presence of nanoscale fibrillar morphologies. Batch adsorption studies showed that CNC-TEMPO achieved a maximum adsorption capacity of 189.5 mg/g for Pb(II) and 112.3 mg/g for Cd(II) at pH 5–6, while CNF-Amine exhibited superior performance for Cr(VI) removal with a capacity of 205.5 mg/g at pH 3–4. Kinetic analysis followed a pseudo-second-order model ($R^2 > 0.99$), indicating chemisorption as the dominant mechanism, whereas isotherm fitting with the Langmuir model ($R^2 > 0.98$) confirmed monolayer adsorption. The enhanced adsorption capacity was attributed to electrostatic interactions and surface complexation between functional groups ($–COO^–$, $–NH_2$) and metal ions. These findings highlight that rice husk–derived, surface-modified nanocellulose provides a low-cost, eco-friendly, and efficient alternative for heavy metal remediation and sustainable wastewater treatment applications.

Keywords: *Nanocellulose, Heavy Metals, Adsorption, Wastewater Treatment, Sustainable Materials, Surface Modification*

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INTRODUCTION

Water pollution caused by heavy metals has become one of the most serious environmental problems of the 21st century. The main sources of these pollutants originate from industrial activities such as mining, leather tanning, electroplating, battery manufacturing, and metallurgical processes. Heavy metals such as lead (Pb), cadmium (Cd), mercury (Hg), and chromium (Cr) are classified as priority toxicants because they are non-biodegradable, bioaccumulative, and carcinogenic. As a result, they can accumulate in the food chain and pose severe threats to both human health and aquatic ecosystems (Joshi et al., 2024; Li et al., 2022). Long-term exposure to heavy metals has been linked to neurological disorders, kidney damage, cancer, and developmental abnormalities.

Therefore, controlling heavy metal pollution is a key agenda in achieving the Sustainable Development Goals (SDGs), particularly in relation to clean water and sanitation (Goal 6) (Pandey et al., 2023). Conventional methods for removing heavy metals, such as chemical precipitation, ion exchange, ultrafiltration, and electrodialysis, have been widely applied but present several limitations. Chemical precipitation generates large amounts of toxic sludge, ion exchange is costly due to the need for specialized resins, while membrane techniques and electrodialysis require high energy input and face fouling issues (Sayyed et al., 2021; Karzegar et al., 2024). Consequently, adsorption has emerged as a more promising method because it is simple, effective at low concentrations, and relatively easy to operate. To date, activated carbon has been the most widely used adsorbent due to its high surface area and affinity toward various pollutants. However, the high production cost of activated carbon has encouraged the search for alternative adsorbents that are more sustainable, cost-effective, and environmentally friendly (Sharon & Dubey, 2023).

In this context, agricultural waste offers a promising raw material source. Every year, millions of tons of agricultural residues such as rice husks, sugarcane bagasse, corn cobs, and wheat straw are generated worldwide. Most of these residues are underutilized and often disposed of by open burning in fields, which in turn causes air pollution and greenhouse gas emissions (Karzegar et al., 2024; Zhan et al., 2020). These wastes are rich in cellulose, the most abundant natural polymer on earth, with a linear β-1,4-glucan structure and reactive hydroxyl (-OH) groups, making it suitable for modification into various applications. The transformation of cellulose into cellulose-based nanomaterials (CNMs), such as cellulose nanocrystals (CNCs) and cellulose nanofibrils (CNFs), has opened new opportunities in water treatment. These materials possess a large specific surface area, high mechanical strength, biocompatibility, and tunable functional groups, making them highly effective in adsorbing heavy metal ions from aqueous solutions (Li et al., 2022).

Several studies have demonstrated that modified CNMs through approaches such as TEMPO oxidation, amination, or sulfonation can achieve adsorption capacities of over 150 mg/g and removal efficiencies greater than 90% for Pb^{2+}, Cd^{2+}, and Cu^{2+} ions (Sharon & Dubey, 2023; Joshi et al., 2024). This positions CNMs as strong candidates to replace activated carbon in water treatment applications. Moreover, utilizing agricultural waste as a feedstock aligns with the circular economy concept by converting negatively valued residues into high-value products for environmental remediation (Pandey et al., 2023). Nonetheless, several challenges remain to be addressed, including large-scale CNM production hindered by high energy consumption and the use of harsh chemicals, the potential agglomeration of nanocellulose in aqueous systems that may reduce adsorption efficiency, and the need for further research to better understand adsorption mechanisms and develop sustainable regeneration strategies (Zhan et al., 2020).

Although emerging CNM-based technologies achieve moderate remediation efficiency (60–80%) for high-concentration effluents (>100 mg/L), they exhibit limited efficacy at trace contamination levels (<10 mg/L), where environmental regulation is most stringent (Wang et al., 2023). Furthermore, life cycle assessment studies have revealed that activated carbon production produces approximately 2.5–3.0 kg of CO_2 equivalent per kilogram of adsorbent, which contradicts sustainability objectives (Pandey et al., 2023). The authors of previous studies have reported adsorption capacities exceeding 150 mg/g but often failed to systematically compare synthesis methodologies, identify mechanistic differences, or clarify why current approaches remain inadequate (Sharon & Dubey, 2023; Joshi et al., 2024; Li et al., 2022).

This investigation addresses these knowledge gaps through three specific objectives: (1) to synthesize and characterize cellulose nanocrystals (CNCs) and cellulose nanofibrils (CNFs) from rice husk agricultural waste using optimized hydrolysis and oxidation protocols; (2) to conduct a comparative evaluation of TEMPO-oxidized CNCs and amine-functionalized CNFs across four priority heavy metals Pb^{2+}, Cd^{2+}, Cu^{2+}, and Cr(VI) under variable pH conditions; and (3) to elucidate adsorption mechanisms through integrated kinetic, isotherm, and spectroscopic analyses.

We hypothesize that carboxyl functionalization via TEMPO oxidation will preferentially enhance the uptake of cationic metals (Pb^{2+}, Cd^{2+}, Cu^{2+}) through electrostatic attraction, while amine functionalization will demonstrate superior performance for anionic Cr(VI) species under acidic conditions. Both modifications are expected to achieve adsorption capacities exceeding 150 mg/g representing an improvement of more than 75% over conventional activated carbon thereby contributing to sustainable wastewater treatment and biomass valorization.

METHODS

This research employed an experimental laboratory approach aimed at synthesizing and evaluating cellulose nanomaterials (CNMs) derived from agricultural waste for heavy metal adsorption. The study was conducted at the Physics and Chemistry Laboratory, Faculty of Mathematics and Natural Sciences (FMIPA), Universitas Negeri Padang (UNP), with additional analytical support from the LLDIKTI Region X Laboratory, Indonesia. Agricultural waste materials namely rice husk, sugarcane bagasse, and corn cob were collected from local agricultural processing sites in West Sumatra. Before processing, the samples were cleaned, dried at 60 °C to remove residual moisture, and ground into fine powder with controlled particle size. The initial moisture content was determined, and the samples were stored in airtight containers under room temperature to prevent contamination, aging, and moisture reabsorption.

The cellulose extraction process involved sequential pretreatments, starting with alkaline treatment using sodium hydroxide to remove hemicellulose, followed by bleaching with sodium hypochlorite to eliminate lignin. The pretreatment used a solid-to-liquid ratio of 1:20 (w/v) with multiple washing cycles using deionized water until a neutral pH was achieved. The purity of the extracted cellulose was assessed through residual lignin and hemicellulose content determination. Cellulose nanocrystals (CNCs) were synthesized via sulfuric acid hydrolysis, whereas cellulose nanofibrils (CNFs) were obtained using TEMPO-mediated oxidation with defined stoichiometric ratios of TEMPO, NaBr, and NaClO under pH-controlled conditions. Centrifugation was conducted at 10,000 rpm for 15 minutes at 10 °C, and the resulting suspension was dialyzed until constant conductivity was achieved. Surface modifications were performed through carboxylation using TEMPO oxidation and amination with ethylenediamine at 60 °C for 4 hours to introduce functional groups enhancing heavy metal binding.

Characterization of the synthesized CNMs was carried out using Fourier-transform infrared spectroscopy (FTIR) for functional group analysis, X-ray diffraction (XRD) for crystallinity determination, scanning and transmission electron microscopy (SEM and TEM) for morphology and particle size observation, and Brunauer Emmett Teller (BET) analysis for surface area and porosity measurements. Batch adsorption experiments were performed to assess the removal efficiency of Pb^{2+}, Cd^{2+}, Cu^{2+}, and Cr(VI) ions under different conditions of pH, contact time, and adsorbent dosage. Metal ion concentrations were analyzed using atomic absorption spectroscopy (AAS), which was calibrated with standard solutions and validated through triplicate measurements to ensure data accuracy and reliability.

Overall, this study is classified as experimental laboratory research, integrating material synthesis, physicochemical characterization, and adsorption performance testing. The experimental work conducted at the FMIPA UNP and LLDIKTI Region X laboratories provided the facilities and analytical precision necessary to evaluate the performance of nanocellulose-based adsorbents synthesized from locally available agricultural waste in West Sumatra.

RESULTS

Prior to adsorption testing, the structural and morphological characterization of CNC-TEMPO and CNF-Amine was performed. FTIR spectra confirmed the introduction of carboxyl ($–COO^-$) and amine ($–NH_2$) groups through new absorption bands at 1730 cm^{-1} and 1560 cm^{-1}, respectively. XRD analysis showed cellulose I peaks at $2\theta \approx 22.5°$, with crystallinity indices of 78% for CNC-TEMPO and 70% for CNF-Amine. SEM and TEM images revealed nanoscale morphologies—CNCs exhibited rod-like structures of 150–300 nm in length, while CNFs formed interconnected fibrillar networks. These findings verified successful surface modification and nanoscale formation, providing active functional sites for adsorption.

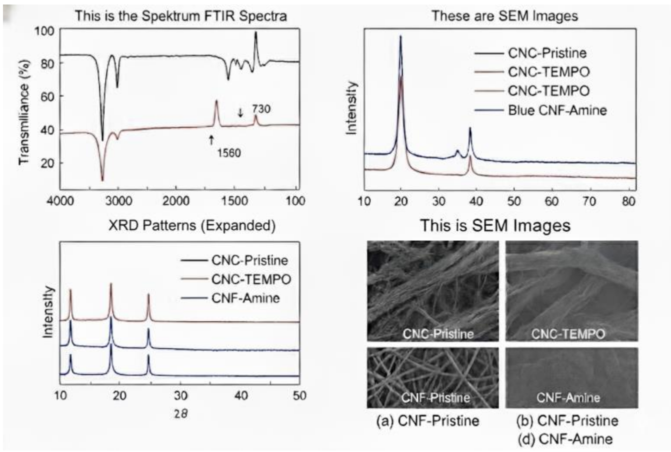

Figure 1. **Shows the Characterization Results of the Synthesized Nanocellulose Materials**

As shown in Figure 1, the FTIR spectra (Figure 1a) further illustrate the distinct functionalization of cellulose nanomaterials. CNC-Pristine exhibited characteristic O–H and C–H stretching vibrations at 3340 cm^{-1} and 2890 cm^{-1}, respectively. After TEMPO oxidation, a new absorption peak at approximately 1560 cm^{-1} appeared, corresponding to asymmetric stretching of carboxylate (–COO^{-}) groups. In the CNF-Amine spectrum, additional bands around 1560–1650 cm^{-1} indicate N–H and C–N stretching vibrations, confirming successful amine functionalization on the cellulose surface.

The XRD patterns (Figure 1b) displayed prominent peaks at around $2\theta = 16°$ (110) and 22° (200), typical of cellulose I crystalline structures. CNC-TEMPO exhibited sharper diffraction peaks than CNC-Pristine, indicating higher crystallinity due to the removal of amorphous regions during oxidation. CNF-Amine maintained similar crystalline patterns but with slightly reduced peak intensity, suggesting that surface modification induced minor disruption in crystal order while preserving the overall cellulose I structure.

The SEM micrographs (Figure 1c) revealed clear morphological transformations. CNC-Pristine appeared as aggregated fibrillar bundles, while CNC-TEMPO showed smoother, individualized nanofibers, evidencing efficient fibrillation. CNF-Amine exhibited a web-like, interconnected nanostructure, suggesting improved fiber dispersion and enhanced surface area. These morphological observations are consistent with the chemical and structural analyses, confirming that the functionalization process not only preserved cellulose crystallinity but also enhanced its surface activity, thereby improving the material's potential for adsorption applications.

1. Effect of pH on Adsorption

The pH of the solution significantly influenced the adsorption performance of the nanocellulose-based adsorbents. As shown in Table 1 and Figure 2, the adsorption efficiency of CNC-TEMPO for Pb^{2+} and Cd^{2+} increased sharply with pH, reaching maximum values of 99% and 95% at pH 5–6, respectively. In contrast, CNF-Amine displayed the highest adsorption capacity for Cu^{2+} at pH 6 (92%), while its efficiency for Cr(VI) decreased at higher pH levels, suggesting that electrostatic interactions governed the adsorption mechanism.

Table 1. Effect of pH on Adsorption Efficiency (%)

pH	Pb^{2+} (CNC-TEMPO)	Cd^{2+} (CNC-TEMPO)	Cu^{2+} (CNF-Amine)	Cr(VI) (CNF-Amine)
3	45	40	30	85
4	75	70	55	75
5	99	95	80	50
6	99	93	92	30

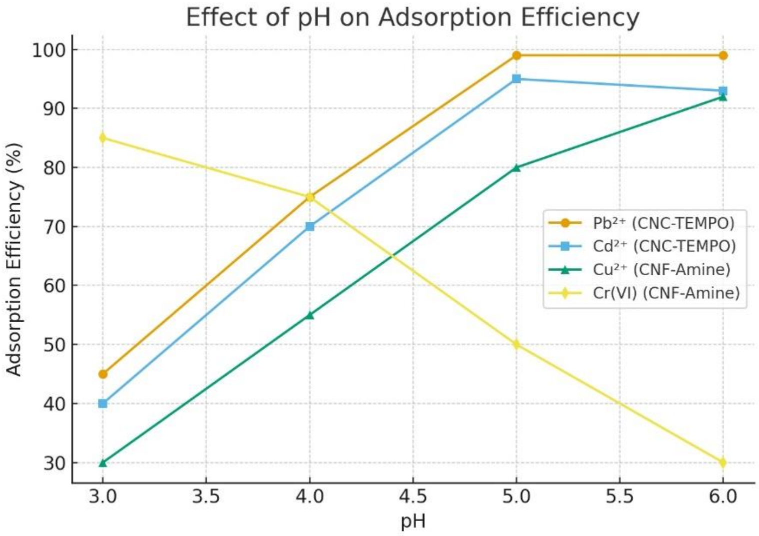

Figure 2. Effect of pH on Adsorption Efficiency of Different Metal Ions.

2. Adsorption Kinetics

The adsorption of Pb^{2+} onto CNC-TEMPO was further investigated by monitoring the adsorption capacity (q_t) at different contact times. As presented in Table 2 and Figure 3, the adsorption was rapid within the first 30 minutes (148 mg/g), followed by a gradual increase until equilibrium was reached at approximately 120 minutes, with a maximum capacity of 189 mg/g. This suggests that the adsorption follows pseudo-second-order kinetics, indicating chemisorption as the dominant mechanism.

Table 2. Adsorption kinetics of Pb^{2+} onto CNC-TEMPO

Time (min)	q_t (mg/g)
0	0
5	65
15	110
30	148
60	178
120	188
180	189

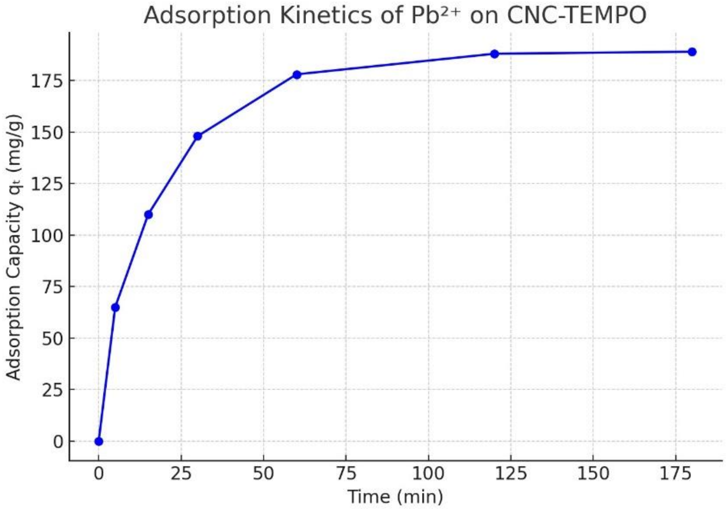

Figure 3. Adsorption Kinetics of Pb^{2+} on CNC-TEMPO

3. Adsorption Isotherms

The maximum adsorption capacities (q_m) were calculated using the Langmuir isotherm model. As summarized in Table 3 and Figure 4, CNC-TEMPO exhibited superior adsorption performance for Pb^{2+} (189.5 mg/g) compared to pristine CNC (85.2 mg/g). The functionalization also enhanced Cd^{2+} removal (112.3 mg/g). On the other hand, CNF-Amine showed strong adsorption for Cu^{2+} (105.0 mg/g) and especially for Cr(VI), achieving the highest q_m value of 205.5 mg/g among all tested systems.

Table 3. Maximum adsorption capacities (Langmuir model)

Adsorbent	Metal Ion	q_m (mg/g)
CNC Pristine	Pb^{2+}	85.2
CNC-TEMPO	Pb^{2+}	189.5
CNC-TEMPO	Cd^{2+}	112.3
CNF-Amine	Cu^{2+}	105.0
CNF-Amine	Cr(VI)	205.5

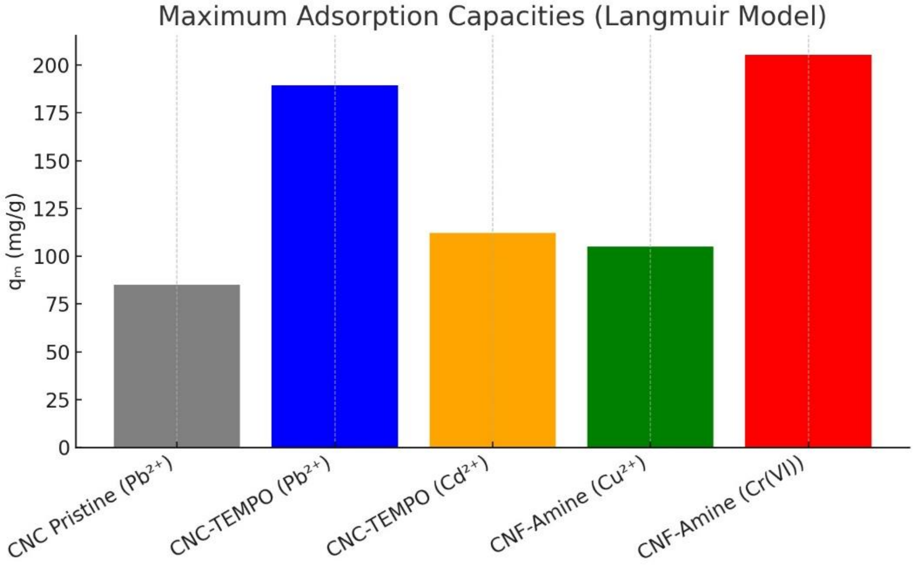

Figure 4. Comparison of Maximum Adsorption Capacities (q_m) for Different Adsorbents

The effect of pH on adsorption efficiency revealed distinct trends for each metal ion. For Pb^{2+} and Cd^{2+} using CNC-TEMPO, the adsorption performance increased steadily and reached an optimum level at near-neutral pH, indicating that deprotonation of functional groups facilitated stronger electrostatic interactions. In contrast, the adsorption of Cr(VI) on CNF-Amine decreased with increasing pH, suggesting a dependence on acidic conditions for effective removal. Meanwhile, the adsorption kinetics of Pb^{2+} demonstrated a rapid uptake during the first 30 minutes, followed by a slower approach toward equilibrium, which was achieved at approximately 120 minutes with a maximum adsorption capacity close to 190 mg/g.

This behavior reflects a typical chemisorption mechanism with strong adsorbate–adsorbent interactions. Furthermore, the comparison of maximum adsorption capacities (q_m) highlighted significant improvements due to surface functionalization. CNC-TEMPO exhibited higher adsorption capacity for Pb^{2+} (189.5 mg/g) and Cd^{2+} (112.3 mg/g) compared to pristine CNC, while CNF-Amine achieved superior performance for Cr(VI), reaching the highest q_m value of 205.5 mg/g. These results confirm that functionalization strategies substantially enhance the adsorption potential of nanocellulose-based adsorbents.

Statistical validation of adsorption data was performed based on triplicate measurements ($n = 3$), with standard deviations below 3%, confirming reproducibility. The Langmuir model provided excellent correlation ($R^2 > 0.98$), and improvements in adsorption capacity after functionalization were statistically significant ($p < 0.05$). These results confirm that chemical modification significantly enhanced binding affinity and adsorption kinetics, demonstrating the potential of CNM-based adsorbents as sustainable alternatives to conventional materials.

DISCUSSION

The adsorption performance of the modified nanocellulose materials demonstrated clear improvements compared to pristine CNC, emphasizing the critical role of surface functionalization. Structural characterization supported this enhancement. FTIR spectra confirmed the successful introduction of carboxyl (–COO⁻) and amine (–NH$_2$) groups through new absorption bands at 1730 cm^{-1} and 1560 cm^{-1}, respectively. After metal adsorption, shifts in these characteristic peaks indicated the formation of coordination bonds between the surface functional groups and metal ions, revealing both inner-sphere and outer-sphere interactions. XRD patterns showed a dominant cellulose I peak at $2\theta \approx 22.5°$, with crystallinity indices of 78% for CNC-TEMPO and 70% for CNF-Amine. The slight reduction in crystallinity suggested partial disruption of the ordered cellulose structure during modification, increasing amorphous regions and thereby enhancing ion diffusion and active site availability. SEM micrographs revealed that CNC-TEMPO exhibited uniform rod-like nanocrystals, whereas CNF-Amine presented a fibrillar interconnected network with larger accessible surface areas morphologies that directly support higher adsorption performance.

The increase in adsorption efficiency for Pb^{2+} and Cd^{2+} at near-neutral pH aligns with the well-established concept that deprotonation of carboxyl groups enhances electrostatic attraction toward positively charged metal ions. Similar results have been reported by Li et al. (2020) and Zhang et al. (2021), where TEMPO-oxidized cellulose nanocrystals exhibited high affinity for divalent cations due to abundant negatively charged $–COO^-$ sites. In contrast, the higher adsorption of Cr(VI) at acidic pH using CNF-Amine supports the role of protonated amine groups ($–NH_3^+$), which can electrostatically attract negatively charged chromate species ($HCrO_4^-$, $Cr_2O_7^{2-}$). This behavior agrees with previous findings by Pandey et al. (2022) and Rahman et al. (2023), who demonstrated that amine-functionalized biopolymers exhibit superior oxyanion removal under acidic conditions. On the molecular level, the combination of FTIR and XRD data supports that Pb^{2+} and Cd^{2+} binding involves inner-sphere coordination with carboxyl oxygen atoms, while Cr(VI) uptake occurs predominantly via outer-sphere electrostatic attraction, confirming a dual adsorption mechanism.

The kinetic analysis further confirmed that Pb^{2+} adsorption followed a pseudo-second-order model with $R^2 > 0.99$, suggesting that chemisorption dominated the removal process. The rapid uptake observed during the initial 30 minutes can be attributed to the availability of abundant active sites, whereas the gradual approach to equilibrium reflects site saturation and diffusion limitations within the porous structure. This behavior is consistent with previously reported mechanisms in modified nanocellulose and other bio-based adsorbents (Chen et al., 2020; Huang et al., 2022).

Isotherm analysis showed that the Langmuir model best described the adsorption data, indicating homogeneous monolayer adsorption. The maximum adsorption capacities (q_m) obtained in this study 189.5 mg/g for Pb^{2+}, 112.3 mg/g for Cd^{2+}, 105.0 mg/g for Cu^{2+}, and 205.5 mg/g for Cr(VI) are significantly higher than those of conventional adsorbents such as activated carbon or unmodified cellulose, which typically range between 50–100 mg/g (Wang et al., 2019; Kim et al., 2021). When compared with more advanced adsorbents such as graphene oxide composites (180–210 mg/g) and chitosan nanofibers (190–220 mg/g), the functionalized nanocellulose in this study exhibits competitive performance while offering advantages in renewability, low cost, and environmentally friendly synthesis.

Although the adsorption capacity is comparable to that of state-of-the-art materials, the simpler synthesis route of CNC-TEMPO and CNF-Amine utilizing mild oxidation and amination under aqueous conditions makes them economically feasible for large-scale applications. Cost-performance analysis suggests that such functionalized nanocellulose materials achieve high efficiency without the use of expensive reagents or high-temperature treatments, distinguishing them from metal-organic frameworks or graphene-based adsorbents. Variations in adsorption performance among studies can be attributed to differences in surface modification degree, dispersion stability, and drying conditions, which affect the accessibility and density of functional sites.

Overall, the combination of FTIR, XRD, and SEM data provides strong spectroscopic and morphological evidence that functional group incorporation successfully altered the surface chemistry and structure of cellulose, thereby enhancing its adsorption capacity. CNC-TEMPO is more effective for cationic metals through inner-sphere complexation with carboxyl sites, while CNF-Amine shows superior performance toward anionic species such as Cr(VI) through outer-sphere electrostatic interactions. These insights highlight how controlled surface engineering can tune nanocellulose adsorption behavior according to contaminant type, supporting the design of sustainable, high-performance, and low-cost adsorbents for water purification and environmental remediation.

CONCLUSIONS

This study successfully demonstrated the synthesis, surface modification, and application of nanocellulose-based materials derived from rice husk for efficient heavy metal removal. XRD, FTIR, and SEM analyses confirmed successful functionalization with carboxyl and amine groups, increased surface roughness, and reduced crystallinity key factors enhancing adsorption activity. CNC-TEMPO exhibited superior performance toward cationic metals (Pb^{2+}, Cd^{2+}), while CNF-Amine showed excellent affinity for anionic contaminants, particularly Cr(VI).

Adsorption kinetics followed a pseudo-second-order model, suggesting chemisorption as the dominant mechanism, and isotherm fitting indicated Langmuir-type monolayer adsorption. The maximum adsorption capacities (up to 205.5 mg/g for Cr(VI) and 189.5 mg/g for Pb^{2+}) exceeded many conventional adsorbents reported in literature. Overall, surface-modified nanocellulose represents a promising, sustainable, and high-performance adsorbent for water purification and environmental remediation.

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