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Synthesis of Carbamate-Modified Cellulose Biocoagulant from Jengkol (*Archidendron pauciflorum*) Peel via Crosslinking Method for Lead Removal from Wastewater

Alpan Sofyan Tanjung, Rifda Adillah, Muhammad Fajrul Rahman, Yulia Eka Putri[*]

Department of Chemistry, Faculty of Mathematics and Natural Sciences, Andalas University
Limau Manis, Kecamatan Pauh, Kota Padang, Sumatera Barat, 25175, Indonesia

[*]*Corresponding author: yuliaekaputri@sci.unand.ac.id*

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ABSTRACT. Heavy metal pollution poses a serious environmental and health threat. An effective and environmentally friendly solution to mitigate this issue is the use of cellulose-based coagulants derived from jengkol (*Archidendron pauciflorum*) peel waste modified with carbamates. Cellulose isolated from jengkol peel was successfully modified with carbamate derived from urea through a crosslinking method. The formation of cellulose and carbamate crosslinks was confirmed by Fourier transform infrared (FTIR) spectroscopy, which showed new absorption peaks corresponding to amide ($-NH_2$) and nitrile (CN) functional groups. Morphological observations using a scanning electron microscope (SEM) showed changes in the shape of cellulose fibers after modification with carbamate, where carbamate cellulose exhibited a denser fiber morphology with larger pore sizes. Elemental analysis revealed the presence of nitrogen in carbamate-crosslinked cellulose, indicating that the cellulose has successfully bonded with the carbamate derived from urea. The decrease in heavy metal lead (Pb) concentration in wastewater samples was achieved by adding 1.5 g of carbamate-modified cellulose, resulting in 96.87% Pb removal. These results show that the cellulose-based biocoagulant from jengkol peel waste, modified with carbamate via crosslinking, has been successfully synthesized and significantly reduced the concentration of the heavy metal Pb.

INTRODUCTION

Heavy metals are metallic elements with a relatively high density, having an atomic density greater than 4 g/cm³, at least 5 times that of water, and are toxic or poisonous even at low concentrations (Munir *et al.*, 2021). However, being classified as a heavy metal is more about chemical properties than density. Heavy metals, including lead (Pb), cadmium (Cd), zinc (Zn), mercury (Hg), arsenic (As), silver (Ag), chromium (Cr), copper (Cu), and iron (Fe), are often associated with heavy metal pollution in the environment, especially in waters adjacent to industrial areas (Yang *et al.*, 2018). In particular, the presence of lead in the body can cause various health problems if it accumulates to high concentrations. Pb generates changes in morphology and physiology, damages human cells, causes nervous system illnesses, lowers blood pressure and body weight, lung dysfunction, and cancer (Assiri *et al.*, 2023; Chen *et al.*, 2023; Li *et al.*, 2018; Wei *et al.*, 2020).

A number of studies have been conducted to mitigate the effect of Pb metals in waters by reducing their concentrations via applying various methods, such as ion exchange, membrane filtration, phytoremediation, adsorption, chemical precipitation, etc (Diaconu *et al.*, 2023; Mahmoud and Mostafa, 2023; Xu *et al.*, 2021). One effective approach among these methods is adsorption with coagulants. This method is highly considered due to its simplicity and cost-effectiveness, and it utilizes mechanisms of adsorption, complexation, and coprecipitation. Currently, the use of natural coagulants is preferred because they are biodegradable and do not create new recycling-related problems. Therefore, developing natural coagulants is considered an environmentally friendly and strategic approach to reducing the heavy metal concentrations in water.

Environmentally friendly coagulants are synthesized from biologically natural resources, such as plants (Kalibbala *et al.*, 2023). One potential natural coagulant is jengkol peel. Jengkol peel is commonly found in the

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environment as waste and is not utilized for other purposes. It contains active compounds derived from secondary metabolites, including terpenoids, alkaloids, saponins, tannins, flavonoids, and glycosides (Hidayah *et al*., 2019). Among these compounds, glycosides have great potential as natural coagulants because they can form derivatives such as cellulose (Ramos-Vargas *et al*., 2020). Cellulose has the characteristic physicochemical property of strong sorption capacity, making it a suitable adsorbent both in its natural state and after modification. It can be used as a natural adsorbent for various materials, including water, organic substances, metal ions, dyes, etc. (Acharya *et al*., 2021). Previous studies have shown that unmodified cellulose has a relatively low adsorption capacity for heavy metals due to the limited number of available active sites, and this limitation can be effectively addressed by chemical modification (Daochalermwong *et al*., 2020). Therefore, it is necessary to modify its structure and morphology to increase the number of absorption sites, thereby enhancing its absorption capacity.

Modifying cellulose with carbamates is an effective approach to create a specific structure and morphology of cellulose. In this study, crosslinking is used to synthesize carbamate-modified cellulose, yielding products with high thermal stability and flexibility. These properties enhance cellulose's resistance to water, making it suitable for application in waters contaminated with heavy metals (Dayarathne *et al*., 2022). Therefore, we synthesized a natural coagulant by crosslinking cellulose modified with carbamates, using jengkol peel waste as a source of cellulose and urea as a source of carbamate. The ability of the carbamate-modified cellulose product in lowering the concentration of the heavy metal Pb was evaluated by determining the initial and final concentrations of Pb in artificial wastewater samples.

RESEARCH METHODS

Jengkol peel waste was obtained from a local traditional market in Padang city, West Sumatra, Indonesia. The other substances used are high-purity NaOH, NaOCl, HCl, HNO_3, and methanol (all purchased from Merck), urea, and distilled water.

Characterization using X-Ray diffraction (XRD/ PAN Analytical X-pert PRO), Fourier Transform Infrared Spectroscopy (FTIR/JEOL JSM 6950), and Atomic Absorption Spectroscopy (AAS/Shimadzu AA7000) was measured at the central laboratory of Universitas Andalas. Scanning Electron Microscopy (SEM) equipped with Energy Dispersive X-ray Spectroscopy (SEM-EDX/HITACHI FLEXSEM 100) at Sepuluh Nopember Institute of Technology.

Isolation of Cellulose from Jengkol Peel Waste

A Total of 1 kg of jengkol peel waste was washed thoroughly with distilled water to remove dirt or impurities. Then, it was cut into small pieces approximately 1 × 1 cm and dried in the sunlight for 3 days to remove the water content. After that, the small pieces of jengkol peel were dried in an oven at 60 °C for 4 hours. The dried jengkol peel was ground and sieved using a 60-mesh sieve to produce a fine powder. A 60-gram sample of fine powder was added to 90 mL of 2% sodium hydroxide solution and diluted with 810 mL of distilled water. The mixture was vigorously stirred at 80 °C for 2.5 hours. After heating, allow the mixture to cool to room temperature, then filter to separate the solid cellulose fibers from the liquid. Solid cellulose was washed thoroughly with distilled water until the wash water was neutral (pH 7). The cellulose powder was dried in an oven at 100 °C for 2 hours. Then, the powder was transferred to a new beaker and added to 150 mL of 1% NaOCl, stirred continuously at 95 °C for 1 hour. The mixture was heated to around 70 – 80 °C and maintained this temperature for an hour. The mixture was cooled to room temperature and then filtered through Whatman No. 41 filter paper. The wet powder was dried in an oven at 50 °C for 24 hours and labeled as JC (jengkol cellulose) (Desianna *et al*., 2017).

Modification of Cellulose to Carbamate-Modified Cellulose by Crosslinking Method

The isolated cellulose was subsequently converted into carbamate-modified cellulose through a crosslinking process. Fifteen grams of cellulose were mixed with 15 grams of urea, then heated in an oven at 135 °C for 1 hour. Following this, the mixture was washed with methanol, and its pH was adjusted to reach a pH of 3. The product was labeled as JC-C (jengkol cellulose-carbamate) (Harlin, 2019)

Sampling of Lead Sample from Wastewater.

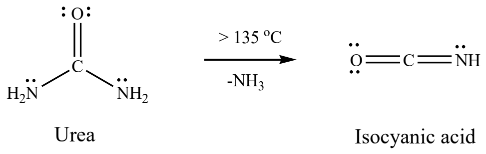
Water samples were collected from the Batang Arau River in Padang City, West Sumatra, Indonesia. Samples were taken from the downstream area at five collection points, each approximately 300 meters apart. The sampling bottle was submerged to about 30 cm below the water surface. The sample was transferred into a 200 mL bottle through filtration using filter paper. The sample was allowed to fill the bottle completely to ensure there were no air bubbles, and the bottle was immediately closed securely. All collected sample bottles were transferred to a beaker. 100 mL of the collected sample was transferred into a beaker, and 5 mL of concentrated HNO_3 was added while stirring thoroughly. After cooling, the solution was filtered through Whatman 41 filter paper and transferred to a vial for Pb metal concentration analysis by AAS. The cellulose product, in varying quantities of 1 g and 1.5 g, was added to separate beaker glasses, each containing 100 mL of river wastewater sample. These beakers were then placed in a jar test apparatus and stirred for 1 minute at 60 rpm. Following this, the mixtures were allowed to sit for 4 hours. The resulting solution was filtered using Whatman 41 filter paper and subsequently dried in an oven at 105 °C for 3 hours (Dadebo and Gelaw, 2024).

RESULTS AND DISCUSSION

Modification Reaction

The cellulose isolated from jengkol peel waste was activated with sodium hydroxide during isolation to enhance the accessibility of its hydroxyl groups. Figure 1 illustrates the detailed reaction mechanism involved in the conversion of cellulose to carbamate-modified cellulose. During the reaction of cellulose and urea at a temperature of 135 °C, urea will decompose to form ammonia (NH_3) and isocyanic acid (HNCO). The isocyanic acid then reacted with the hydroxyl groups (-OH) on the cellulose backbone, typically at the C-2, C-3, or C-6 positions of the glucose units in the cellulose chain (Antonino *et al.*, 2021). The nucleophilic hydroxyl groups attacked the electrophilic carbon of isocyanic acid, forming a carbamate linkage. The final product, carbamate-modified cellulose, has carbamate groups attached to the cellulose backbone, increasing its water solubility and imparting a fiber-like morphology.

1. Urea pyrolysis reaction



2. The formation of carbamate-modified cellulose

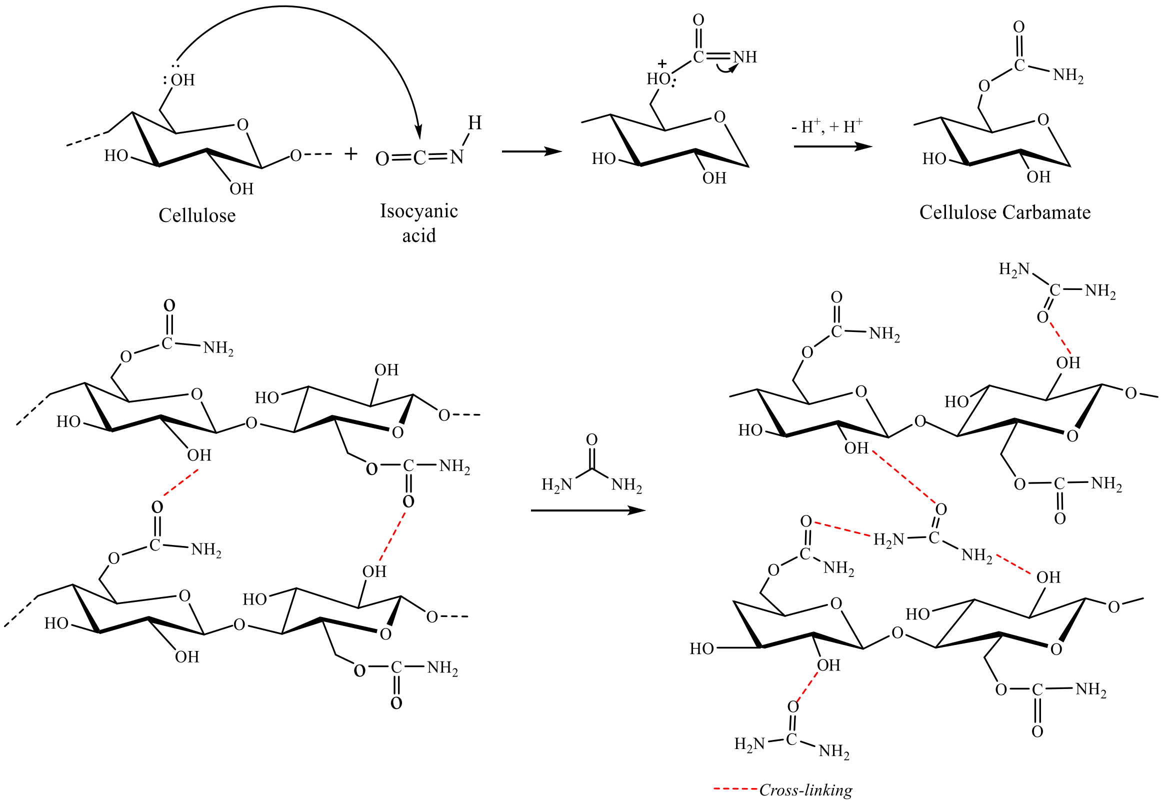


Figure 1. Proposed mechanism for the modification reaction via crosslinking method.

FTIR Spectrum of the Cellulose Products

Characterization of interactions in cellulose, carbamate-modified cellulose, and carbamate-modified cellulose after Pb absorption using FTIR spectra reveals distinct spectral patterns. The functional groups shown in Figure 2 highlight a new absorption peak in carbamate-modified cellulose at wavenumbers 1621 cm^{-1}, indicating the presence of the scissoring mode of the NH_2 groups (Liang *et al*., 2020; Tabaght *et al*., 2023). The absorption peak at 1452 cm^{-1} was assigned to C–N stretching modes (Onija *et al*., 2012). The increased absorption intensity of the C–N stretching modes in the cellulose carbamate spectrum suggests a higher nitrogen content, indicating the formation of bonds between cellulose and urea (Zhang *et al*., 2013). The O–H group spectra of carbamate-modified cellulose show a higher absorption intensity compared to cellulose. This increase is due to the reaction between urea and hydroxyl groups, which disrupts intra- and intermolecular hydrogen bonds in the cellulose chain (Willberg-Keyriläinen *et al*., 2018). The FTIR spectrum of carbamate-modified cellulose synthesized after Pb absorption shows the absence of C–N vibration specific vibrational frequencies at wave numbers 1452 – 1455 cm^{-1} associated with carbon-nitrogen (C–N) bonds in a molecule. Additionally, the absorption peak of the amide functional group shifts to a higher wavenumber, specifically to 1634 cm^{-1}. The amide functional group has a lone pair of electrons that can bond with the Pb ion to form a coordination complex. This coordination affects the interaction of functional groups in the carbamate-modified cellulose-Pb structure, resulting in the elimination of the C–N vibrational mode and a shift in the NH_3 absorption peak (Liang *et al*., 2020).

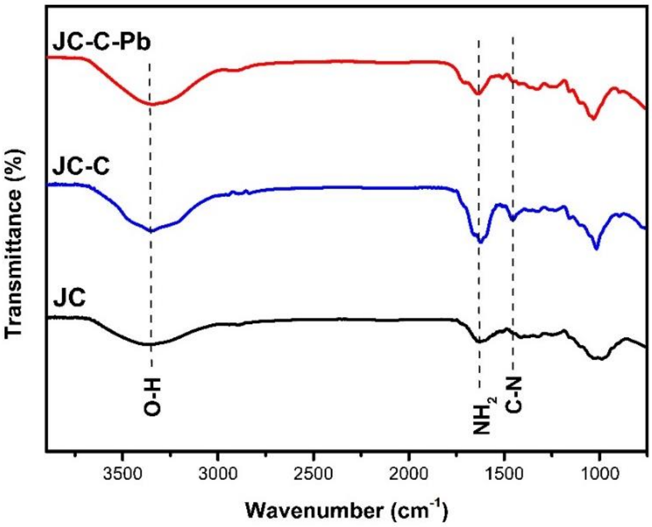
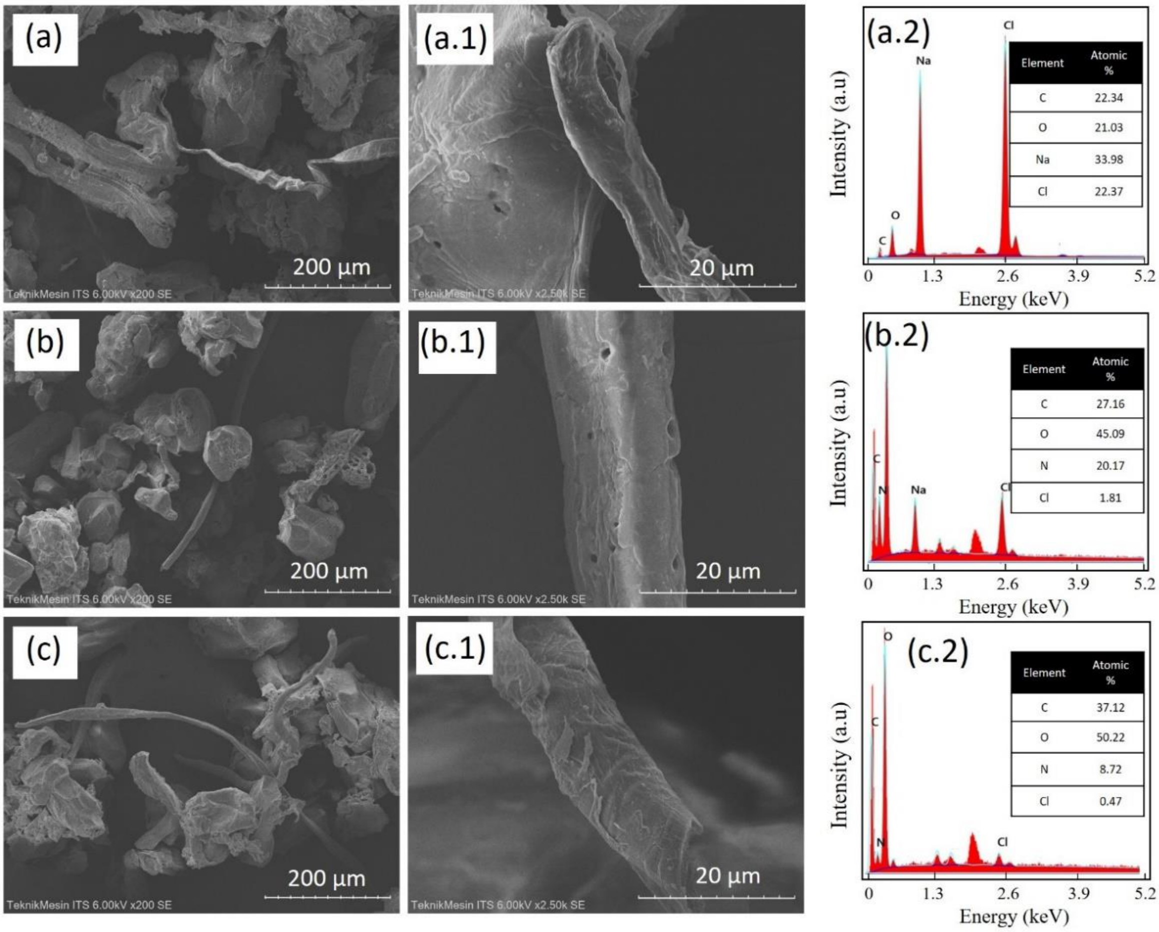


Figure 2. FTIR spectrum of the cellulose products.

Morphology Observation and Elemental Analysis of the Cellulose Products

Morphological observations through SEM images revealed changes in all the observed cellulose products, as shown in Figure 3. The morphology of cellulose isolated from jengkol peel, as shown in Figure 3(a), exhibits a mixture of fibrils and thin sheets, and magnification of the particles, as shown in Figure 3(a.1), confirms the presence of a rough and porous surface. The pores on the cellulose surface result from the delignification process, where lignin compounds are removed from lignocellulose to form cellulose. These surface characteristics are consistent with the morphological changes commonly observed after alkaline delignification, in which NaOH cleaves ether and ester linkages between lignin, hemicellulose, and cellulose, leading to the removal of these components and producing a more open, fibrillated structure (Aloud *et al*., 2023). Modification of cellulose with carbamate changes the size of the cellulose fibers to increase in size, as shown in Figure 3(b), and magnification of the particles, as shown in Figure 3(b.1), reveals a rougher and more porous surface. Changes in the particle's surface indicate the formation of crosslinks between cellulose and carbamate, resulting in larger fibrils and thicker sheets with increased surface texture. The absorption of the heavy metal Pb by carbamate-modified cellulose significantly alters the surface morphology of the particles, as shown in Figure 3(c). The fibrils and sheets' texture becomes smoother, and surface pores disappear (Figure 3(c.1)). These changes occur because the heavy metal Pb binds to carbamate-modified cellulose both on the surface and within the pores; as a result, the surface was covered, and the pores were filled with Pb (Koprivica *et al*., 2023)

The EDX spectrum confirms the presence of elements in the resulting cellulose product, and the corresponding atomic percentages are provided in each figure. As shown in Figure 3(a.2), the main components of cellulose are carbon and oxygen. Modification of cellulose with carbamate introduces nitrogen (N) with high intensity, confirming the incorporation of nitrogen bound to the carbonyl group in the carbamate-modified cellulose structure. Additionally, as shown in Figure 3(b.2), the EDX spectrum of carbamate-modified cellulose also shows increased intensity for the carbon and oxygen spectra due to the addition of a carbonyl functional group from the urea bound to the cellulose. The presence of Cl and Na elements is attributed to the use of NaOH and NaOCl solutions in the delignification process (Shaikh *et al*., 2021). Similar findings have been reported in previous studies, where residual Na and Cl were detected on treated biomass surfaces as a result of incomplete ion removal during alkaline and bleaching treatments, despite subsequent washing and neutralization processes (Bichang'a *et al*., 2024; Ali *et al*., 2020; Susi *et al*., 2018; Kumar *et al*., 2014). The EDX spectrum of carbamate-modified cellulose after Pb adsorption does not show a distinct Pb signal (Figure 3(c.2)), which can be attributed to the very low concentration of Pb in the sample. Nevertheless, the presence of Pb can be inferred indirectly by comparing the nitrogen peak intensity of the carbamate-modified cellulose before and after Pb adsorption. A noticeable decrease in nitrogen peak intensity is observed after Pb adsorption, suggesting the involvement of nitrogen-containing functional groups in the coordination interaction with Pb^{2+} ions (Hamza *et al*., 2021). This observation is further supported by the decrease in the nitrogen atomic percentage from 20.17% to 8.72%, as shown in the inset table. In addition, the EDX spectrum also shows a noticeable decrease in the intensity of Na and Cl after Pb adsorption. This reduction is attributed to the ion-exchange and displacement processes that occur during the interaction with Pb^{2+}. Residual Na^+ and Cl^- originating from the alkaline and bleaching treatments are known to be weakly adsorbed on the highly hydrophilic cellulose surface. When Pb^{2+} interacts with the carbamate functional groups, multivalent metal ions replace these loosely bound species, causing Na^+ to be exchanged and Cl^- to be partially desorbed from the surface. This phenomenon further supports the occurrence of Pb uptake by carbamate-modified cellulose (Daochalermwong *et al*., 2020).



(a.2)

Element	Atomic %
C	22.34
O	21.03
Na	33.98
Cl	22.37

(b.2)

Element	Atomic %
C	27.16
O	45.09
N	20.17
Cl	1.81

(c.2)

Element	Atomic %
C	37.12
O	50.22
N	8.72
Cl	0.47

Figure 3. FTIR Spectrum of the cellulose products. SEM images and EDX spectrum of cellulose products, (a, a1, a2) JC, (b, b1, b2) JC-C, and (c, c1, c2) JC-C-Pb.

Effectiveness of Lead Removal

In Table 1, the initial Pb metal concentration in river water samples was 0.032 mg/L. The addition of a biocoagulant to the samples reduced the Pb concentration in the solution. These results indicate that the resulting cellulose product can adsorb Pb due to its high hydroxyl (OH) content. Introducing carbamate groups on cellulose nearly doubled the adsorption capacity for Pb. This increased adsorption is attributed to the presence of polar groups, including hydroxyl (OH), amide ($–NH_3$), and nitrile (CN), which have high electronegativity and bind Pb metal via coordination pathways. Furthermore, the adsorption capacity of the carbamate-modified cellulose product can be further improved by increasing its mass, allowing it to absorb nearly all of the Pb in the river water sample. Thus, adding a biocoagulant results in more Pb being bound to the biocoagulant (Jagaba *et al*., 2021).

Table 1. Confirmative value for Pb removal efficiency in wastewater samples.

Cellulose Product	Pb Concentration(mg/L)		Reduced concentration of Pb (mg/L)	(%) Removal
	Initial Concentration	Final Concentration		
JC-Pb/1	0.032	0.022	0.010	31.25%
JC-C-Pb/1	0.032	0.014	0.018	56.25%
JC-C-Pb/1.5	0.032	0.001	0.031	96.87%

CONCLUSION

The effect of chemical modification of the cellulose structure derived from jengkol peel with carbamate via crosslinking enhances the Pb metal adsorption capacity from river water samples. The success of this crosslinking method is demonstrated by the appearance of new absorption peaks in the FTIR spectrum of carbamate-modified cellulose, corresponding to amide (–NH3) and nitrile (CN) groups. In addition, SEM morphological observations further supported the analysis based on FTIR data, which revealed changes in the carbamate-modified cellulose structure, displaying a rough, porous surface. Moreover, the EDX spectrum confirmed the successful synthesis of the biocoagulant, as evidenced by the presence of nitrogen in the carbamate-modified cellulose product. The ability of the cellulose product to absorb Pb metal increased by 16.18% after modification to carbamate-modified cellulose, achieving a 96.87% reduction in Pb levels. These results highlight a cellulose-based biocoagulant derived from jengkol peel as a promising and sustainable material for Pb removal via crosslinking, offering advantages in green chemistry and abundant raw material availability.

AUTHOR CONTRIBUTION

Conceptualization, project administration, supervision, funding acquisition, writing-review and editing, Y.E.P.; Methodology, investigation (including cellulose isolation and carbamate modification), data curation, formal analysis, writing-original draft preparation, A.S.T; Sampling, characterization analysis, data visualization, writing-original draft preparation, R.A.; Investigation, validation, statistical analysis, figure preparation, writing-review and editing, M.F.R.

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