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Design and Evaluation of a Biochar-Integrated Supramolecular Fibrous Framework for Efficient Microplastics Removal from Aqueous Environments

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Abstract*—Current wastewater treatment plants are unable to capture the microplastics (MPs), highlighting the need for sustainable, low-cost, and eco-friendly remediation methods. This study focuses on the design of supramolecular fibrous frameworks produced via the self-assembly of chitin (Ct) and cellulose (Cel) fibers, combined with municipal solid waste (MSW) derived biochar (BC), for MP removal. MSW-BC was prepared via the pyrolysis at 550 °C. Ct was dispersed in 5% acetic acid and combined with Cel and BC at a 10:1:1 ratio. All three components were combined and homogenized for 30 min. Following blending, the mixture was frozen at −19 °C for 24 h. The frozen mixture was oven-dried at 70 °C for 72 h. Fourier transform infrared (FTIR) spectroscopy confirmed successful integration of the three components, revealing characteristic O–H/N–H stretching (3300-3250 cm^{-1}), C–H stretching (2890 cm^{-1}), amide I and II bands (1650-1625 cm^{-1} and 1550 cm^{-1}), and C–O–C/C–O stretching vibrations (1157 and 1010 cm^{-1}), indicative of strong hydrogen bonding and intermolecular interactions between the biopolymer matrix and BC surface. MP removal performance was assessed on packed-bed column experiments using a 50 mL polyethylene (PE) MP solution (1 mg/L). Under optimum conditions (10 cm bed height, 2.5 mL/min flow rate), the quartz-only control removed 42.14% of MPs, while the quartz/Ct-Cel-BC column achieved 95.30% removal. Therefore, incorporating BC may have improved physical entrapment due to its porous structure and fibrous framework. Removal efficiency increased with greater bed height and lower flow rate, which may be due to longer residence times and greater contact time between MPs and the adsorbent surface. Overall, the integration of MSW-BC into a Ct-Cel supramolecular fibrous framework yielded a multifunctional, cost-effective adsorbent with excellent PE MP removal efficiency.

Keywords: *Biochar, Chitin, Cellulose, Supramolecular Fibrous Framework, Microplastics, Column Experiment*

I. INTRODUCTION

Microplastics (MPs) are plastic particles ranging from 1 µm to 5 mm and have become widely distributed contaminants in aquatic environments due to increasing plastic production and improper disposal [1],[2]. MPs enter drinking water sources through surface runoff, atmospheric deposition, wastewater effluent, and landfill leachate [3],[4]. Due to their high surface area and affinity for co-contaminants such as toxic trace metals and organic pollutants increase their mobility and toxicity. MPs are increasingly recognized as a risk to ecological and human health. They enter the environment through different pathways. Aquatic organisms ingest MPs directly or indirectly through the food chain. It results in physical blockage, decreased feeding efficiency, and impaired reproduction, whereas adsorbed contaminants can be transferred across trophic levels [2],[5]. Current removal technologies, including coagulation, flocculation, sedimentation, and membrane filtration, achieve only 25-85% removal, perform poorly for particles less than 100 µm, and are limited by fouling and high operating costs [4],[5]. Therefore, a novel solution for the simultaneous removal of MPs and associated contaminants from water systems is in demand. Recently, biomass-derived materials have emerged as promising alternatives for

removing MPs. For example, chitosan nanofibers sponges self-assembled with cellulose (Cel) to form chitosan-cellulose supramolecular frameworks, achieving up to 99.9% MP removal through physical entrapment and surface interactions, overcoming the size limitations of conventional filtration while retaining high permeability [1],[6]. Municipal Solid Waste (MSW) derived biochar (BC) provides a high surface area, well-developed porosity, and oxygen-containing functional groups that enhance MP adhesion [7],[8]. However, very few studies have examined its integration into supramolecular fibrous frameworks for MP removal [1],[16]. In particular, the integration of MSW BC with self-assembled Ct-Cel fibrous matrix, which could pair the adsorption capacity of BC with the structural and filtration advantages of the fibrous framework, remains unexplored. To address this gap, this study aims to develop a cost-effective, eco-friendly, and locally applicable solution for MP removal that aligns with sustainable waste management and environmental remediation goals. The objective of this study was to fabricate a novel chitin (Ct), Cel, and BC (Ct-Cel-BC) composite from low-cost biomass by integrating the adsorption capacity of BC, the filtration matrix of Cel, and the electrostatic/hydrogen-bonding properties of Ct. The efficiency of the fabricated Ct-Cel-BC composite in removing polyethylene (PE) MP from aqueous environments was evaluated under controlled laboratory conditions using a fixed-bed column system. The outcomes of this research will provide foundational knowledge for future large-scale applications and help address MP pollution in the aqueous environment.

II. METHODOLOGY

A. Preparation of Ct-Cel-BC composite

BC was prepared by carbonizing a 1:1:1 (w/w/w) mixture of coconut shells, coconut husks, and wood collected from a pyrolyzer unit at Karadiyana, in a muffle furnace at 550 °C. Then, the BC was allowed to cool, crushed and sieved using a 100 µm sieve. The Ct obtained from Xi'an Qinghekang Biotechnology Co. Ltd. was dispersed in 5% acetic acid and magnetically stirred for 24 h. It allowed exfoliation into a finer nanofiber form and activates amine/amide groups for self-assembly [9]. Medical-grade cotton was mechanically disintegrated to obtain Cel fibers and expose hydroxyl groups for hydrogen bonding [1]. Ct, Cel, and BC were combined in a 10:1:1 mass ratio, allowing the Ct and Cel fractions to dominate the hydrogen-bonded fibrous network while BC was embedded as a dispersed adsorptive filler [10]. The mixture was homogenized in a blender for 30 min, allowing activated –OH, $–NH_3^+$, and –NHCO– groups to form an intermolecular hydrogen-bond network without a chemical cross-linker. The mixture was allowed to freeze at -19 °C for 24 h, and then oven-dried at 70 °C for 72 h to obtain the final porous Ct-Cel-BC composite [1].

B. Column setup and filtration experiments

A glass column (3 cm in diameter and 35 cm in height) was packed with a pre-washed quartz support layer (sieved using a 1 mm sieve), followed by the Ct-Cel-BC composite bed, and a 0.5 cm height quartz layer on top to secure the bed [11],[12]. The height of the Ct-Cel-BC composite bed was varied to (2, 5 and 10 cm) to evaluate its effect on MP retention.

Pristine PE MP (size ∼100 µm Microscrub®) was purchased from Micro Powders, Inc., Tarrytown, New York, USA. It was selected as the model MP as it is considered one of the most widely produced and environmentally detected polymers [3],[9]. A PE MP suspension of 100 µm particles at a fixed initial concentration of 1 mg/L was introduced at the top of the column and allowed to percolate downward with the controlled flow rates of 2.5, 5, and 10 mL/min.

C. Analytical methods

The Fourier transform infrared (FTIR) spectrometer (PerkinElmer, L1600400, UK) was used to obtain FTIR spectra of individual raw materials (Ct, Cel, and BC) and the final Ct-Cel-BC composite. Retained MP particles in the effluent were filtered using glass fiber filter papers, stained with Nile Red, and counted under UV light [13]. Removal efficiency (RE) was calculated using equation 1,

$$RE\ (\%) = [(N_0 - N_e) / N_0] \times 100 \quad (1)$$

where N_0 is the initial MP count in the fed MP suspension, and N_e is the residual MP count in the column effluent [11]. All glassware was cleaned with filtered deionised water to minimise background contamination, and procedural blanks were processed alongside the experimental samples. Each experimental condition was performed in triplicate (n=3). The mean and standard deviation (SD) of the removal efficiency were calculated for each condition using Origin 2018, and these mean ± SD values, with error bars indicating ± SD, were used in the figures.

III. RESULTS AND DISCUSSION

A. FTIR characterization of the Ct-Cel-BC composite

FTIR spectra of Ct, Cel, BC, and the synthesized Ct-Cel-BC composite are presented in Fig. 1. The BC spectrum showed a broad O–H stretching band at 3470 cm^{-1}, an aromatic C=C/conjugated carbonyl band in the 1600-1650 cm^{-1} region, and a C-O stretching band at 1020 cm^{-1} [12]. The Cel spectrum exhibited a sharp O-H band at 3335 cm^{-1}, a C-H band at 2890 cm^{-1}, and glycosidic C-O-C/C-O bands at 1160 and 1020 cm^{-1} [14]. The Ct spectrum showed a broad N–H/O–H band at 3260 cm^{-1}, the Amide I (C=O) band at 1630 cm^{-1}, and the amide II band at 1550 cm^{-1} [14].

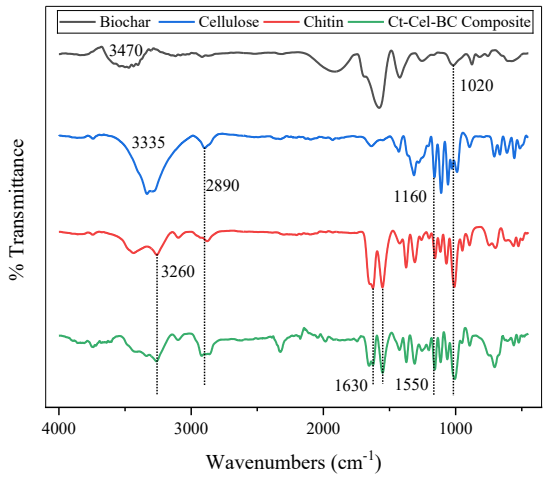


Figure 1. FTIR spectra of chitin, cellulose, biochar and the Ct-Cel-BC composite.

All characteristic functional group bands from the three materials were retained in the Ct-Cel-BC composite. It confirmed that Ct, Cel, and BC were successfully combined into a single fibrous structure. The O–H/N–H stretching envelope broadened and shifted relative to the individual Ct (3260 cm^{-1}) and Cel (3335 cm^{-1}) bands, consistent with new intermolecular hydrogen bond formation during self-assembly [14]. The Ct amide I band shifted from 1626-1630 cm^{-1} in pure Ct to 1625-1650 cm^{-1} in the composite, indicating that carbonyl groups of Ct interacted with hydroxyl groups of Cel and surface functional groups from BC, while the amide II band remained unchanged at 1550 cm^{-1}. It demonstrated that the core amide functionality of Ct was preserved rather than chemically changed. The polysaccharide ring structures and oxygenated surface groups of all three components were confirmed to remain intact within the composite, as the C–O–C/C–O region resolved into bands at 1157 and 1010 cm^{-1}, matching the Cel and BC reference bands [7],[15]. These results confirmed that the fabrication procedure yielded the intended Ct-Cel-BC supramolecular fibrous composite, assembled via intermolecular hydrogen bonding rather than chemical cross-linking [1],[16].

B. Filtration performance

Removal efficiency was evaluated at flow rates of 2.5, 5, and 10 mL/min, at bed heights of 2, 5, and 10 cm.

1) Effect of bed height

The quartz/Ct-Cel-BC bed height significantly affected the system's removal efficiency. Overall, at all flow rates, removal efficiencies increased as bed height increased from 2 to 10 cm. At 10 mL/min, Removal efficiencies were 35.2 ± 13.73% (2 cm), 67.9 ± 4.46% (5 cm), and 70.5 ± 7.93% (10 cm). At 5 mL/min, removal efficiency enhanced from 64.1 ± 4.60% to 86.5 ± 2.78% and to 87.9 ± 3.92%, with the increase of bed height from 2 to 5 to 10 cm, and at 2.5 mL/min, removal efficiency escalated from 74.4 ± 3.38% at 2 cm to 90.5 ± 2.13% at 5 cm and to 95.30 ± 1.15% at 10 cm. Increasing the bed height may increase the probability of entrapment and trapping of MP particles in quartz/Ct-Cel-BC media, thereby improving MP removal performance. This observation makes good physical sense and highlights a real strength of the Ct-Cel-BC composite material. A higher bed heights contains a greater total amount of the Ct-Cel-BC fibrous material, meaning there are more fibers and more capture sites available for the MP particles to encounter as they pass through the column and PE MP solution takes longer to pass all the way through the column at any given flow rate, giving each MP particle more time to come into contact with the fibrous material and become trapped or sorbed onto it before it reaches the outlet.

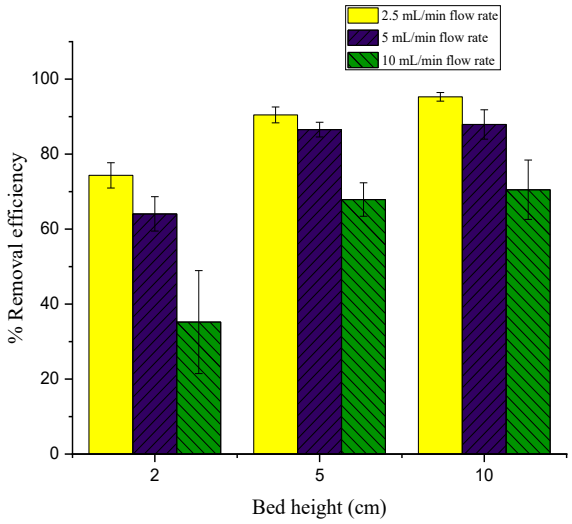


Figure 2. Effect of bed height on microplastic removal efficiency using a Ct-Cel-BC packed-bed column. Bars represent mean ± SD (n=3).

2) Effect of flow rate

The flow rate of the PE MP solution affected the removal efficiency. At a bed height of 10 cm, removal efficiency rose from 70.5 ± 7.93% at 10 mL/min to 87.9 ± 3.92% at 5 mL/min, and to 95.30 ± 1.15% at 2.5 mL/min. At the 5 cm bed height, removal efficiency rose from 67.9 ± 4.46% to 86.5 ± 2.78% and to 90.5 ± 2.13% over the same flow rates, and at the 2 cm bed height, removal efficiency rose from 35.2 ± 13.73% to 64.1 ± 4.60% and to 74.4 ± 3.38%. In each instance, decreasing the flow rate increased MP removal efficiency, confirming that increasing the flow rate negatively impacts MP removal efficiency. Reducing the flow rate increases the time of PE MP solution to retain inside the quartz/Ct-Cel-BC column, giving the PE MP particles more opportunity to reach and become caught by the fibers of the composite. A lower flow rate reduces the force of the PE MP solution in pushing against particles that have already made contact with the fibers, making it less likely that a trapped particle is knocked loose and swept back out of the column. Importantly, even at the fastest flow rate tested (10

mL/min), the composite still achieved 70.5 ± 7.93% removal efficiency with a 10 cm bed height and 67.9 ± 4.46% with a 5 cm bed height. At higher flow rates, the increased velocity is expected to reduce the contact time available for PE MP particles to interact with the hydrogen-bonded fibrous surfaces of the composite, to push particles deeper into the bed before they can be captured, and to erode or dislodge particles that were only loosely and physically entrapped. This shows that the Ct-Cel-BC composite responds very favorably to increased contact time, and that near-complete removal is readily achievable with this material under realistic, moderate operating conditions. This decline reflects the reduced contact time between the PE MP suspension and the composite bed at increasing flow rates, which leads to less opportunity for adsorption and entrapment within the porous matrix [13].

However, these findings were obtained under controlled laboratory conditions using a single MP type (~100 µm PE) at a fixed concentration of 1 mg/L. They represent laboratory performance and proof of concept, not proven practical treatment potential, and should not be extrapolated to real wastewater containing mixed polymer types, irregularly shaped particles, dissolved organic matter, and competing contaminants.

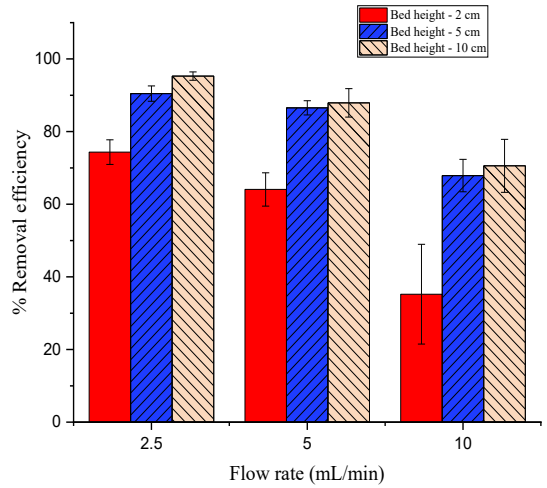


Figure 3. Effect of flow rate on microplastic removal efficiency using a Ct-Cel-BC packed-bed column. Bars represent mean ± SD (n=3).

IV. CONCLUSION AND RECOMMENDATIONS

A biochar-integrated supramolecular Ct-Cel-BC fibrous framework was successfully fabricated via hydrogen bond-driven self-assembly of Ct, Cel, and MSW-derived BC without any chemical cross-linker. FTIR analysis confirmed retention of all characteristic functional groups from the three materials, along with broadening of the O–H/N–H envelope and a shift in the chitin amide I band. It provides direct evidence of intermolecular hydrogen bonding of composite components. The fixed-bed column system enables evaluation of PE MP removal at different bed heights. The results indicated that MP entrapment by the Ct-Cel-BC composite was significantly improved with increasing bed height and decreasing flow rate. Overall, the system exhibited improved retention efficiency with longer residence times and greater filtration depths, suggesting that hydraulic conditions are important for the efficient removal of MPs. These findings reflect laboratory performance with a single MP type (~100 µm PE, 1 mg/L) and demonstrate proof of concept rather than proven practical treatment potential in real wastewater.

Future studies are needed to evaluate how PE and other MPs perform at different concentrations. It is important to evaluate reusability across multiple filtration cycles and to test this composite material in real-world wastewater and landfill leachate scenarios to validate its practical use in a sustainable MP remediation approach.

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