

Hybrid Chitin-Coffee Ground Biochar Foam for Microplastic Adsorption

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ABSTRACT

Microplastics threaten ecosystems and human health. Based on the existing microplastic adsorption solutions, we hypothesized a sustainable filtration method based on a hybrid chitin-coffee ground biochar foam made of β/α -chitin foam from waste seafood and coffee ground biochar from used coffee grounds to adsorb microplastic particles in water would be efficient. The hybrid chitin-coffee ground biochar foam achieved consistently high adsorption efficiency in seawater and showed strong performance for fluorescent PS microspheres larger than 1 μm in deionized water, river water and seawater. This adsorption performance resulted from the combined effects of electrostatic attraction, hydrogen bonding, hydrophobic, and π - π interactions. This study demonstrates that waste-derived materials were able to offer a low-cost, environmentally friendly, and efficient solution for microplastic adsorption from water.

Keywords: Microplastic; coffee ground biochar; chitin; adsorption; sustainable

INTRODUCTION

Microplastics are small plastic fragments that are smaller than 5 mm, which mainly originate from plastic packaging, old tires, cosmetics, and synthetic textile (1-2). Microplastics enter the water system by weathering and surface runoff and are found in every aquatic system (3). Microplastics can cause negative effects on both the environment and human health. Accumulating a large amount of microplastic in the body can cause long-term health impacts, like metabolic disorders, intestinal flora imbalance, and inflammatory responses (4). Because microplastics can act as vectors for more harmful contaminants, their ingestion by humans may lead to the adsorption of these toxic substances, posing even more

severe health risks (5). At the same time, in ecosystems, many marine organisms often die from being entangled by plastics or cannot digest the microplastic they consume, which severely impacts biodiversity (6-7). Moreover, by consuming these animals, humans are exposed to an even larger amount of microplastic due to bioaccumulation (8).

Many technologies have been developed to address the issue of microplastic pollution in aquatic systems. For relatively large plastic debris (>1 mm), techniques such as filtration and elutriation have proven effective (9-11). In contrast, microplastics under 5 μm are more difficult to remove, because their small sizes prevent them from being captured by standard filters. There are many emerging approaches for removing microplastics, such as membrane bioreactors, coagulation and agglomeration, photocatalysis, and bioaccumulation (12-13). However, these approaches often suffer from inherent limitations on effectiveness, economic costs, and environmental impact.

We hypothesized that utilizing low-cost adsorbents

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derived from waste material may be a promising solution to overcome these limitations. For example, consumed biomass, like coffee grounds, can effectively adsorb microplastics with a size greater than 1 μm , while offering the advantages of sustainability and low-cost (14–15). Similarly, foams made from seafood waste, such as crayfish shells and squid bones, which are also low-cost and sustainable, showed strong adsorption performance for microplastics smaller than 1 μm , especially in seawater (16). Even though these two low-cost solutions had been studied relatively well, both of them have some shortcomings. While using coffee grounds resulted in good filtration of microplastics larger or equal to 1 μm , they only performed well in laboratory-grade water (e.g., deionized (DI) water) and did not perform well in environmental samples of seawater or river water (17). The pure chitin foam, which is made from crayfish shells and squid bones, performed best for microplastics smaller than 1 μm , but it has not been tested for the 1–5 μm microplastic size range. Moreover, the adsorption efficiency of the chitin foam was poor in DI water, and was best in seawater.

By integrating the advantages of coffee grounds and chitin foam, we developed a hybrid chitin-coffee ground biochar foam (hybrid foam) as an effective microplastic adsorbent material for the adsorption of microplastics with a size range of 0.5–5 μm in DI water, river water, and seawater. We especially intended to find a better hybrid foam that could provide better adsorption efficiency for $\geq 1 \mu\text{m}$ microplastics across most water sources.

METHODS AND MATERIALS

Coffee ground biochar preparation

The coffee ground was acquired from Goodme, a Chinese coffeeshop (No. 60 Xingzhuang Rd, Zhenhai District, Ningbo, Zhejiang, China). The collected coffee grounds were dried in an electric thermostatic drying oven (DHG-9070, Shanghai Yiheng Scientific Instruments Co., Ltd.) at 60 $^{\circ}\text{C}$ for 24 hours. The dried coffee ground was then placed in a quartz boat and then inserted into a nitrogen-purged tubular furnace (ZHK-B03123K-200, Tianjin Zhonghuan Electric Furnace Co., Ltd., China). The tubular furnace program was configured to increase the temperature at a heating rate of 5 $^{\circ}\text{C}/\text{min}$ for 55 min until reaching 300 $^{\circ}\text{C}$, after which the temperature was maintained at 300 $^{\circ}\text{C}$ for 1h, followed by natural cooling to room temperature under continuous nitrogen flow. The resulting coffee ground biochar was stored in an airtight container for later use.

Production of the hybrid foam

To prepare the hybrid foam, 100 mg of alpha chitin ($\alpha\text{-CT}$) from shrimp shell was mixed in a 200 mL beaker with 98 mL of 2% acetic acid (Macklin Biochemical Co., Ltd.) and 2 mL of water. A high-speed homogenizer (FJ200-PSH, Shanghai Specimen & Model Factory) was used to homogenize the mixture at 15,000 rpm for 40 minutes. After obtaining a nanofiber dispersion, 1,000 mg of β -chitin ($\beta\text{-CT}$), which is extracted from squid bones, was added into a beaker to yield a final concentration of 10 mg mL^{-1} . A high-speed homogenizer was used to homogenize the mixture at 8,000 rpm for 5 min. The β/α -chitin gel was obtained (Figure 1). The weight of the obtained mixture was measured, and coffee ground biochar was mixed with the prepared β/α -chitin gel to achieve coffee ground biochar to β/α -chitin gel (coffee ground biochar-to-chitin-gel) mass ratios of 1:60, 1:80, and 1:100. Using a mechanical mixer (BGD 740/1, Biuged Precise Instruments (Guangzhou) Co., Ltd.), the chitin-biochar mixture was stirred at 1,000 rpm for 24 hours. After 24 hours, the mixture was split into equal volumes of 8 mL and placed inside 35 mm diameter petri dishes. The β/α -chitin gel and coffee ground biochar mixture was then frozen at -80 $^{\circ}\text{C}$ for 6 hours in the freeze chamber of a freeze dryer (YW-10A-80, Nanjing Yanwo Biotechnology Co., Ltd.). The frozen foams were then removed from the petri dish and placed onto a piece of tin foil paper. The foams were then moved back into the vacuum chamber of the freeze dryer to be lyophilized for 30 hours under vacuum to obtain the hybrid foams. (Figure 2).



Figure 1. The homogeneous β/α -chitin gel obtained after high-speed homogenization of α -chitin nanofibers and β -chitin in dilute acetic acid.



Figure 2. Hybrid foams with coffee ground biochar-to-chitin-gel mass ratio from left to right 1:60, 1:80, and 1:100. Pure chitin foam with no coffee ground biochar is shown on the far right.

Preparation of the water samples

Three types of water samples were used in the experiments. River water was collected from Ningbo University of Technology (No. 201 Fenghua Road, Jiangbei District, Ningbo, Zhejiang Province, China); seawater was collected from Zhoushan fishing ground (East China Sea, 29°00'–31°00' N, 121°00'–123°00' E, off the coast of Zhejiang Province, China); DI water gained from Ningbo University of Technology. Each water sample was aliquoted into several 50 mL centrifuge tubes, each tube containing a 40 mL water sample. Fluorescent polystyrene (PS) microspheres of different sizes (0.5, 1, and 5 μm , TianJinDae, Tian Jin, China) were added to the tubes to obtain a fixed concentration of 0.01 mg mL^{-1} .

Adsorption Experiment Procedure

Lyophilized hybrid foams and pure chitin foams were placed in 50 mL centrifuge tubes that contained 40 mL of the prepared water sample spiked with fluorescent PS microplastics. One hybrid filter was added to each tube. The tubes were then shaken using a standard testing sieve shaker (200, Xinxiang Yucheng Vibration Machinery Co., Ltd.) for 20 min to make sure all microplastics in the water sample fully contacted the foam. After shaking, the foam was removed, and the remaining solutions were collected and placed in a 48-well plate. Fluorescence intensity in each tube was measured using a microplate reader (Synergy H1 Hybrid Multimode Microplate Reader, BioTek Instruments, Inc., a part of Agilent Technologies, Inc.) with an excitation wavelength of 488 nm. The specific fluorescence intensity was recorded at the emission wavelength of 518 nm. The fluorescence intensity was converted to concentrations based on a standard calibration curve for each microplastic size (Figure 3). We considered the fact that fluorescence signals can be influenced by other factors in the background water samples. Therefore, for

each type of water, a blank well containing the same water sample without PS microspheres was measured and subtracted from all corresponding sample readings prior to concentration conversion.

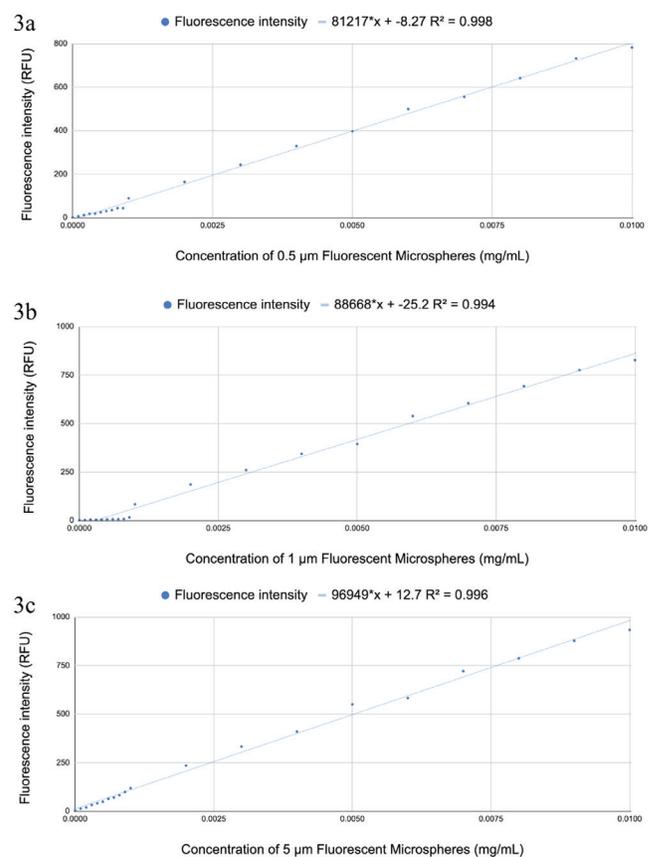


Figure 3. Standard curve showing fluorescence intensity plotted against different sizes' microplastics concentration. a) Standard curve showing fluorescence intensity plotted against 0.5 μm microplastics concentration. b) Standard curve showing fluorescence intensity plotted against 1 μm microplastics concentration. c) Standard curve showing fluorescence intensity plotted against 5 μm microplastics concentration.

Standard Curve

A separate standard curve was determined for each microplastic size: 0.5 μm (Figure 3a), 1 μm (Figure 3b), and 5 μm (Figure 3c). Twenty different concentrations, including 0.001, 0.002, 0.003, 0.004, 0.005, 0.006, 0.007, 0.008, 0.009, 0.01, 0.02, 0.03, 0.04, 0.05, 0.06, 0.07, 0.08, 0.09, and 0.1 mg mL^{-1} , were used to create the standard curve. Linear regression was applied to the fluorescence intensity versus concentration data to establish an equation describing microplastic concentration as a function of measured fluorescence intensity for each microplastic particle size.

Adsorption percentage calculation

The concentration of microplastic that was absorbed was calculated using Equation 1, where C_0 is the initial concentration of microplastics in the solution and C_t is the residual concentration of microplastics after adsorption.

$$C_{\text{adsorption}} = C_0 - C_t \quad (\text{Equation 1})$$

The adsorption percentage (A) was then determined using Equation 2:

$$A (\%) = \frac{(C_0 - C_t)}{C_0} \times 100 \quad (\text{Equation 2})$$

Porosity Test

Weigh fully dried foams (m_1), then soaked in ethanol. When the foam was filled with ethanol, the weight was measured again (m_2).

The porosity of the foam was calculated using Equation 3:

$$P (\%) = \frac{(m_2 - m_1)}{\rho_{\text{ethanol}} V} \times 100 \quad (\text{Equation 3})$$

Where ρ_{ethanol} is the density of ethanol in current room temperature, V is the volume of the foam.

RESULTS

To determine whether the hybrid is effective in adsorbing microplastics, we evaluated the adsorption percentage of fluorescence PS microspheres (0.5, 1, and 5 μm) for different mass ratios of coffee ground biochar to β/α -chitin gel in DI water, river water, and seawater samples. We produced coffee ground biochar by heating coffee grounds in a nitrogen-purged tubular furnace. We bought α -CT made from shrimp shells and β -CT made from squid bones. The α -CT and β -CT were mixed to

obtain a chitin gel mixture (Figure 1). Biochar was added to the mixture with coffee ground biochar-to-chitin-gel mass ratios of 1:60, 1:80, and 1:100 in addition to a pure chitin control (Figure 2). Foams were placed into DI, river, and seawater samples, which were spiked with 0.5 μm , 1 μm , and 5 μm fluorescent PS microspheres with a fixed concentration of 0.01 mg mL^{-1} . After shaking for 20 minutes, each water sample was collected and processed using a microplate reader to determine the residual concentration of PS microspheres using fluorescence calibration curves. The adsorption percentage (A) was then calculated for different coffee ground biochar-to-chitin-gel ratios, microplastic sizes, and water types from three independent replicates ($n = 3$) and reported as mean \pm standard deviation in Table 1.

Effect of coffee ground biochar-to-chitin-gel mass ratio on adsorption

The presence of coffee ground biochar was associated

Table 1. The adsorption percentage (A) for different coffee ground biochar-to-chitin-gel ratios, microplastic sizes, and water types. Values are presented as mean \pm standard deviation (SD), $n=3$.

Size\ Water Type	DI Water	Seawater	River Water
1:60 (coffee ground biochar-to-chitin-gel mass ratio)			
0.5 μm	36.6 \pm 14.4%	82.6 \pm 8.6%	42 \pm 21.2%
1 μm	58 \pm 25.8%	94.4 \pm 5.9%	78.3 \pm 8.1%
5 μm	92.4 \pm 7.0%	99.3 \pm 0.6%	91.9 \pm 10.0%
1:80 (coffee ground biochar-to-chitin-gel mass ratio)			
0.5 μm	42.9 \pm 8.4%	79.3 \pm 9.1%	47.3 \pm 13.5%
1 μm	78.5 \pm 4.6%	92.4 \pm 8.9%	70.4 \pm 18.2%
5 μm	90.4 \pm 5.8%	99.1 \pm 0.8%	87.7 \pm 12.1%
1:100 (coffee ground biochar-to-chitin-gel mass ratio)			
0.5 μm	35.1 \pm 5.4%	85.3 \pm 13.8%	38.9 \pm 21.1%
1 μm	73.9 \pm 14.7%	92.7 \pm 7.5%	63.8 \pm 14.9%
5 μm	96.3 \pm 2.4%	99.7 \pm 0.3%	94.4 \pm 6.0%
No coffee ground biochar			
0.5 μm	54.4 \pm 12.9%	77.5 \pm 29.2%	70.6 \pm 17.2%
1 μm	72.7 \pm 18.7%	91.5 \pm 10.5%	74.4 \pm 16.9%
5 μm	80.0 \pm 15.3%	98.9 \pm 1.0%	90.0 \pm 9.8%

with higher adsorption efficiency in several conditions. For example, in seawater, for 5 μm particles, hybrid foams achieved adsorption efficiencies ranging from $99.1 \pm 0.8\%$ – $99.7 \pm 0.3\%$ (1:60–1:100), compared with $98.9 \pm 1.0\%$ for the pure chitin foam. Similarly, for 1 μm particles, hybrid foams containing coffee ground biochar showed adsorption of 92.4 ± 8.9 – $94.4 \pm 5.9\%$, exceeding the $91.5 \pm 10.5\%$ observed for the pure chitin without coffee ground biochar control (Table 1). However, the higher adsorption efficiency was not universal. In some cases, the benefit for coffee ground biochar addition was unshown. For example, in DI water, for 0.5 μm PS microspheres, the 1:80 hybrid foam achieved $42.9 \pm 8.4\%$ adsorption efficiency, which was lower than the $54.4 \pm 12.9\%$ observed for the pure chitin foam without coffee ground biochar. Similarly, in river water, for 0.5 μm PS microspheres, the adsorption efficiency was $47.3 \pm 13.5\%$ for 1:80 hybrid foam, whereas the pure chitin foam's adsorption efficiency was $70.6 \pm 17.2\%$; for 5 μm PS microspheres, the adsorption efficiency was $87.7 \pm 12.1\%$ for 1:80 foam, whereas the pure chitin foam's adsorption efficiency was $90.0 \pm 9.8\%$ (Table 1). For 1 μm PS microspheres, the adsorption efficiency was $78.3 \pm 8.1\%$ for 1:60 hybrid foam, $70.4 \pm 18.2\%$ for 1:80 hybrid foam, and $63.8 \pm 14.9\%$ for 1:100 hybrid foam; whereas the pure chitin foam's adsorption efficiency was $74.4 \pm 16.9\%$ (Table 1).

Across the tested conditions, even though the 1:80 coffee ground biochar-to-chitin-gel mass ratio was not consistently the highest in every combination, it showed generally comparable and often higher adsorption efficiencies. For example, for 1 μm PS microspheres in DI water, compared with the pure chitin control adsorption efficiency of $72.7 \pm 18.7\%$, the 1:80 hybrid foam increased adsorption efficiency to $78.5 \pm 4.6\%$; and in seawater, compared with the pure chitin foam control adsorption efficiency of $91.5 \pm 10.5\%$, the 1:80 hybrid foam slightly increased adsorption efficiency to $92.4 \pm 8.9\%$ (Table 1). However, the benefit was not universal to all water types. In river water sample, the 1:80 coffee ground biochar-to-chitin-gel mass ratio adsorption efficiency was $70.4 \pm 18.2\%$, which did not outperform the pure chitin foam with adsorption efficiency $74.4 \pm 16.9\%$ (Table 1).

To isolate the effect of coffee ground biochar-to-chitin-gel mass ratio, PS microsphere size and water matrix were fixed at 1 μm and DI water, respectively (Figure 4a). 1:80 hybrid foam adsorption efficiency was $78.5 \pm 4.6\%$, which was higher than 1:60 hybrid foam adsorption efficiency of $58 \pm 25.8\%$, 1:100 foam adsorption efficiency of $73.9\% \pm 14.7\%$ and no coffee

ground biochar foam efficiency of $72.7\% \pm 18.7\%$ (Table 1). However, under these controlled conditions, even though 1:80 foam had better adsorption efficiency than other ratios in numerical data, no statistically significant differences were observed among the ratios (one-way ANOVA, $p = 0.6945$).

Effect of PS microsphere size on adsorption

Across all water types and coffee ground biochar-to-chitin-gel mass ratios, the adsorption efficiency increases with particle size. For example, at a coffee ground biochar-to-chitin-gel mass ratio of 1:60 in DI water, adsorption increased from $36.6 \pm 14.4\%$ (0.5 μm) to $58.0 \pm 25.8\%$ (1 μm) and $92.4 \pm 7.0\%$ (5 μm) (Table 1). A similar size-dependent trend was observed in river water at a ratio of 1:100, where adsorption increased from $38.9 \pm 21.1\%$ (0.5 μm) to $63.8 \pm 14.9\%$ (1 μm) and $94.4 \pm 6.0\%$ (5 μm) (Table 1).

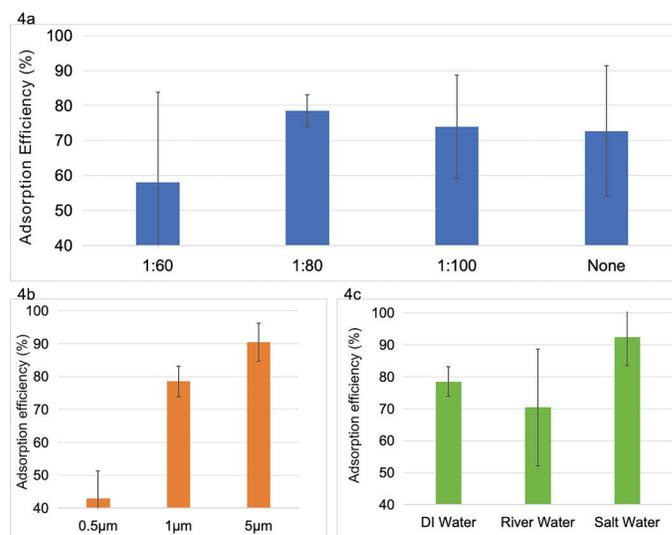


Figure 4. Effect of key parameters on PS microsphere adsorption efficiency under controlled conditions. (4a) Effect of coffee ground biochar-to-chitin-gel mass ratio on adsorption efficiency at a fixed PS microsphere size of 1 μm in DI water. (4b) Effect of PS microsphere size on adsorption efficiency at a fixed coffee ground biochar-to-chitin-gel mass ratio of 1:80 in DI water. (4c) Effect of water type on adsorption efficiency at a fixed coffee ground biochar-to-chitin-gel mass ratio of 1:80 and PS microsphere size of 1 μm . Data are presented as mean \pm standard deviation from three independent replicates ($n = 3$). Error bars represent standard deviation. Statistical analysis was performed using one-way ANOVA ($\alpha = 0.05$) with Tukey's post hoc test.

To isolate the effect of particle size, the coffee ground biochar-to-chitin-gel mass ratio and water matrix were fixed at 1:80 and DI water, respectively (Figure 4b). Under these controlled conditions, adsorption efficiency increased from $42.9 \pm 8.4\%$ ($0.5 \mu\text{m}$) to $78.5 \pm 4.6\%$ ($1 \mu\text{m}$) and $90.4 \pm 5.8\%$ ($5 \mu\text{m}$) (Table 1). This size effect was statistically significant (one-way ANOVA, $p = 0.000859$). Tukey's post hoc test indicated that $0.5 \mu\text{m}$ differed significantly from $1 \mu\text{m}$ ($p = 0.0038$) and $5 \mu\text{m}$ ($p = 0.0008$), while the difference between $1 \mu\text{m}$ and $5 \mu\text{m}$ was not significant ($p = 0.243$).

Effect of water type on adsorption

Based on the results shown in Table 1, in all tested conditions, seawater had the highest PS microsphere adsorption efficiency numerically. For example, at a 1:100 and $5 \mu\text{m}$ condition, adsorption efficiency in seawater is $99.7 \pm 0.3\%$, which is higher than $96.3 \pm 2.4\%$ in DI water and $94.4 \pm 6.0\%$ in river water (Table 1). Similarly, at 1:60 and $1 \mu\text{m}$ condition, adsorption efficiency in seawater is $94.4 \pm 5.9\%$, which is higher than $58 \pm 25.8\%$ in DI water, $78.3 \pm 8.1\%$ in river water. To isolate the effect of water types, the coffee ground biochar-to-chitin-gel mass ratio and PS microsphere size were fixed at 1:80 and $1 \mu\text{m}$ (Table 1). Under this controlled condition, adsorption efficiency in seawater is $92.4 \pm 8.9\%$, which is higher than $78.5 \pm 4.6\%$ in DI water and $70.4 \pm 18.2\%$ in river water (Table 1). But the differences among water types were not statistically significant in this condition (one-way ANOVA, $p = 0.2639$) (Figure 4c).

DISCUSSION

The adsorption efficiency improved in some conditions due to the addition of coffee ground biochar. Especially including conditions of PS microspheres larger than $1 \mu\text{m}$ or in seawater, the hybrid foams often exhibited numerically higher adsorption efficiencies. According to existing studies, coffee ground biochar has a porous structure and a large number of non-polar aromatic carbon skeletons. This structure enables adsorbing PS microspheres through pore filling, π - π interactions, and hydrophobic interaction (18-20). The β/α -chitin with coffee ground biochar forms a nanofiber network through hydrogen-bond rearrangement, exposing abundant functional groups ($-\text{OH}$, $-\text{NH}_2$, $-\text{NHCO}-$) that enhance its ability to bind PS microspheres, since these groups interact with PS microspheres mainly via $\text{O}-\text{H}-\pi$ and $\text{C}-\text{H}-\pi$ interactions, with the $\text{O}-\text{H}-\pi$ being dominant (15-16). The coexistence of these two materials' adsorption

mechanisms is supported by Fourier-transform infrared (FTIR) spectroscopy analysis. According to FTIR, the $\approx 3200-3600 \text{ cm}^{-1}$ regions confirmed the $-\text{OH}/-\text{NH}$ bond; $\approx 1650 \text{ cm}^{-1}$ regions confirm the amide I bond; and the $\approx 1550 \text{ cm}^{-1}$ regions confirm the amide II bond (Figure 5e). The FTIR showed intensified bonds associated with aromatic carbon around 1600 cm^{-1} , which confirmed that introduction of coffee ground biochar also increased the π - π interactions, providing non-electrostatic affinity (Figure 5e). These FTIR features demonstrate that both β/α -chitin and coffee ground biochar-related interaction domains coexist within the hybrid foam, enabling complementary adsorption mechanisms.

Nitrogen adsorption analysis indicates that enhanced adsorption efficiency of the hybrid foams is not caused by increased nano-scale surface area. According to Brunauer–Emmett–Teller (BET) surface area analysis, the 1:80 hybrid foam has a BET surface area of $12.65 \text{ m}^2/\text{g}$, lower than BET surface area of $15.35 \text{ m}^2/\text{g}$ for pure chitin foam (Figure 5a). In addition, Barrett-Joyner-Halenda (BJH) desorption analysis shows that the nitrogen-accessible mesoporous surface area of the 1:80 hybrid foam is $46.31 \text{ m}^2/\text{g}$, and the volume is 0.077 cc/g , which are lower than the pure chitin foam with nitrogen-accessible mesoporous surface area of $63.59 \text{ m}^2/\text{g}$ and volume 0.107 cc/g (Figure 5b-5c). However, through micropore analysis, it is shown that there is a micropore diameter increase after adding coffee ground biochar. The 1:80 hybrid foam's most probable micropore diameter is 3.72 nm , whereas the pure chitin foam's most probable micropore diameter is 1.76 nm , indicating that coffee ground biochar addition might widen the pores (Figure 5d).

Among the tested different coffee ground biochar-to-chitin-gel mass ratios hybrid foams, the 1:80 hybrid foam exhibited the relatively better overall adsorption performance across different PS microsphere sizes and water types. From looking at the SEM image for foam with 1:60, 1:80, 1:100 and no coffee ground biochar, the foam with 1:80 coffee ground biochar-to-chitin-gel mass ratio perform the most porous structure and interconnected macroporous structure, which increase the accessible surface area to volume ratio (Figure 6). This is quantitatively supported by the porosity test. The test shows that 1:80 hybrid foam has the highest bulk porosity of 95.4% , higher than 1:60 hybrid foam bulk porosity of 91.3% , 1:100 hybrid foam bulk porosity of 93.6% , and pure chitin foam bulk porosity of 93.1% . This higher porosity suggests that the increase in liquid accessibility to adsorption sites, which results in higher PS microsphere adsorption efficiency.

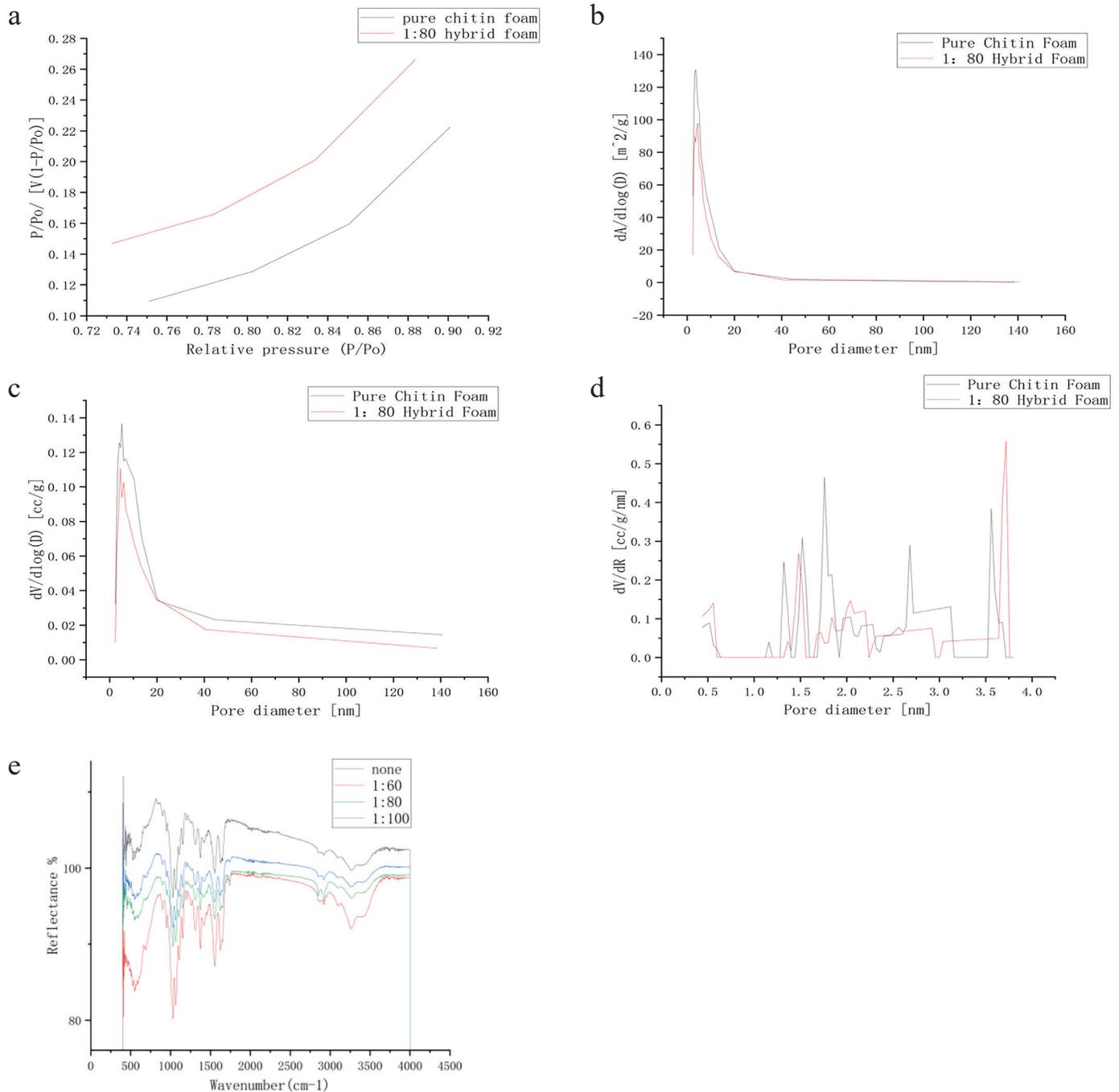


Figure 5. Structural and surface characterization of pure chitin foam and hybrid foam (1:80 coffee ground biochar-to-chitin-gel mass ratio). 5a) BET linearized plots obtained from nitrogen adsorption measurements; black is pure chitin foam and red is a 1:80 coffee ground biochar-to-chitin-gel mass ratio hybrid foam. 5b) BJH pore surface area distributions; black is pure chitin foam; red is the 1:80 coffee ground biochar-to-chitin-gel mass ratio hybrid foam. 5c) BJH pore volume distributions; black is pure chitin foam; red is the 1:80 coffee ground biochar-to-chitin-gel mass ratio hybrid foam. 5d) Micropore size distributions; black is pure chitin foam; red is the 1:80 coffee ground biochar-to-chitin-gel mass ratio hybrid foam. 5e) FTIR spectra of foam with different coffee ground biochar to chitin gel ratios. Black is pure chitin foam with no coffee ground biochar; red is 1:60 coffee ground biochar-to-chitin-gel mass ratio; blue is 1:80 coffee ground biochar-to-chitin-gel mass ratio; green is 1:100 coffee ground biochar-to-chitin-gel mass ratio.

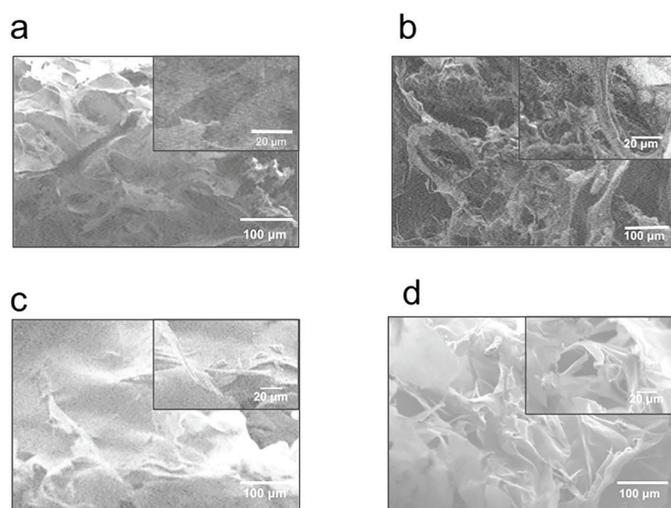


Figure 6. The scanning electron microscopy for different coffee ground biochar-to-chitin-gel mass ratios. a) 1:60 coffee ground biochar-to-chitin-gel mass ratio b) 1:80 coffee ground biochar-to-chitin-gel mass ratio c) 1:100 coffee ground biochar-to-chitin-gel mass ratio d) No coffee ground biochar.

There are some conditions where the addition of coffee ground biochar to foams did not provide better adsorption efficiency compared to no coffee ground biochar foam. For the 0.5 µm PS microspheres conditions, across different water types except seawater and coffee ground biochar-to-chitin-gel mass ratios, no coffee ground biochar foam had a numerically higher adsorption efficiency than any other foams with coffee ground biochar addition. According to research papers, we consider it to be because smaller PS microspheres have a higher surface-to-volume ratio, thus more available binding sites per volume, so β/α -chitin alone is especially effective in removing smaller particles. According to existing papers, coffee ground biochar may block chitin's active sites or cause partial charge neutralization between the slightly negative coffee ground biochar and the slightly positive chitin, thereby reducing overall adsorption efficiency (21–23). For river water condition, foam without coffee ground biochar addition performed numerically higher adsorption efficiency than foam with coffee ground biochar-to-chitin-gel mass ratio 1:80 foam across all tested sizes of PS microspheres and higher adsorption efficiencies than all coffee ground biochar added foams for 1 µm PS microspheres. This trend might be attributed to a relatively higher dissolved organic matter (DOM) concentration in river water. Existing studies have reported that the adsorption efficiency of

coffee ground biochar will be decreased due to DOM occupying active sites or alternating coffee ground biochar surface properties (24). In contrast, according to existing research, electric charges is one of the most important mechanism for chitin foam PS microspheres adsorption, and DOM has been shown to enhance negative charge of PS microspheres. Thus, DOM might strengthen interaction between PS microspheres and chitin foam, therefore positively increasing adsorption efficiency. As a result, when coffee ground biochar is added into the foam, the relative proportion of chitin, whose PS microsphere adsorption performance is positively affected by DOM, is reduced, and relative proportion of coffee ground biochar, whose adsorption performance is negatively effected by DOM, increased. Thus, this combined effect might be the reason why coffee ground biochar addition lowered adsorption efficiency under river water conditions.

As for the effect of PS microsphere sizes on adsorption, among all water types and coffee ground biochar-to-chitin-gel mass ratios, the adsorption efficiency increases as particle size increases. There are several underlying factors that might have led to this result. According to existing researches, the van der Waals adhesion increases as the radius of the particle increases; therefore, larger PS microsphere particles may experience stronger van der Waals forces, allowing them to adhere more firmly to the hybrid foam (25). Moreover, larger PS microsphere particles are more readily captured through physical mechanisms such as interception and impaction, facilitated by the porous structure of the foam. In contrast, nano-sized PS microspheres exhibit enhanced Brownian motion, which may reduce attachment to the foam surface (26).

Water type had an important impact on foam performance with seawater leading to the highest adsorption efficiency, while DI water and river water led to lower adsorption efficiencies. According to existing researches, the high adsorption efficiency in seawater might be because seawater contains more ions including Na^+ and Cl^- . The high ionic strength compresses the electrical double layer and reduces electrostatic repulsion, allowing PS microspheres to approach and adhere better to the foam surface (16).

There are a wide range of published materials for PS microsphere adsorption. For example, we tested microsphere adsorption efficiency of published research's filter made of only coffee ground biochar (17). In DI water, it achieved near-complete adsorption for 2–5 µm particles but approximately 45 ± 1.4 % adsorption for 1

μm PS microspheres. Our 1:80 hybrid foam could achieve $78.5 \pm 4.6\%$ adsorption efficiency, which is higher than published coffee ground biochar only filters.

The cost estimation for hybrid foam is approximately USD 0.26 per gram. Take 1:80 hybrid foam, for example, to obtain one gram of 1:80 hybrid foam, the acetic acid, squid bone β -CT, and shrimp shell α -CT total cost is around USD 0.096, coffee ground biochar production cost is USD 0.00548 and other energy consumption is USD 0.16 (calculated under Ningbo local electricity price 0.538 RMB kWh^{-1} , approximately USD 0.077 kWh^{-1}). As we can see, the energy cost is one of the dominant cost for this hybrid foam production. Under the current laboratory-scale condition, the mass of foam being processed is far under the maximum amount being produced under same energy input, resulting in a relatively high energy cost per gram. Thus, the price of hybrid foam production will be lower if the hybrid foam could be produced on a large scale.

To compare the cost of adsorption materials, the costs of other materials per gram were calculated. The calculation is based on preparation methods described in the research and the materials and equipment prices provided by the manufacturers explicitly specified in this research paper. The total cost for oxidized corncob biochar is around USD 3.82 per gram, with the cost for corncob biochar production being USD 0.006 per gram and cost for the oxidized treatment is USD 3.814 per gram (27); the total cost of CTAB modified magnetic biochar is around USD 3.297 per gram, with the cost for biochar production from rape straw being USD 0.15 per gram and the modification chemical cost is 3.147 per gram (28); the total cost of hardwood vessel-inspired chitosan-based sponge is around USD 1.46 per gram, with the chemical cost being around USD 0.57 per gram and the energy cost is USD 0.89 per gram (29). The cost of our hybrid foam per gram is estimated to be only 6.8%–17.8% of other materials.

This research has several limitations that should be acknowledged. First, adsorption efficiency was only evaluated by a single 20 min shaking period, which limits insight to examine the maximum adsorption capacity of PS microspheres per gram of foam and adsorption kinetics beyond the first 20-minute shaking period. Second, only fluorescent PS microspheres were tested, which limits the generalization to other microplastic types, shapes, and surface qualities that could be found in natural environmental scenarios. Third, regeneration or reuse of the foams was not investigated, thus limiting insight to long-term material stability and reusability.

Fourth, the adsorption experiments were conducted in DI water, river water, and seawater, which are simplified water types. The experiment did not use real wastewater or flow-through condition, so hybrid foam's adsorption efficiency in practical water treatment systems remains unknown. Fifth, even though our experiment was conducted under same batch with a temperature of 25 °C and humidity of 80%, there is no experiment testing how temperature and humidity influence the hybrid foam adsorption efficiency. Sixth, although BET, BJH surface area analysis, SEM imaging, FTIR spectroscopy, and bulk porosity measurements were conducted, additional characterization were not tested, which includes zeta potential, elemental composition, thermal stability, and mechanical properties, limiting a deeper experimental analysis to support the experimental data analysis. Seventh, there was no sterile control conducted in the study, nor did not do a microbiological analysis, which limits experimental data support on the discussion of microorganisms' effect on foam adsorption efficiency.

Future work should quantify the maximum capacity of PS microspheres that the one gram of hybrid foam can adsorb by conducting adsorption saturation tests. Future studies should also examine how to separate adsorbed PS microspheres from the hybrid foams so they can be regenerated for future use. Future studies should also explore the performance of the foam with different shapes (e.g., fibers, pellets, etc.), different microplastics materials (e.g., Polyethylene (PE), Polypropylene (PP), Polyethylene Terephthalate (PET), etc.) and different wastewater conditions. Flow-through experiments should also be incorporated to better assess practical applicability. Furthermore, more physicochemical characterizations should be included to support the study.

CONCLUSION

In this study, we tested a hybrid chitin-coffee ground biochar foam, which was shown to be an environmentally friendly and low-cost adsorbent to remove PS microspheres from water. We tested the adsorption efficiency across a wide PS microsphere size range (from 0.5 μm to 5 μm) and for a variety of water types including DI water, river water, and seawater.

Compared with pure chitin foam, the hybrid foam exhibited higher adsorption efficiency, especially for 1 μm PS microspheres, and for PS microspheres across all sizes in seawater. It is also noted that the highest adsorption efficiency was observed for the largest PS microsphere size of 5 μm for all experimental conditions.

The combined mechanisms from both coffee ground biochar and chitin might have led to the enhanced adsorption efficiency of the hybrid foam. The pore filling, π - π interactions and hydrophobic interaction were provided by coffee ground biochar; and O-H- π , C-H- π and moderate electrostatic interaction provided by β/α -chitin hydrogen bond rearrangement (16, 30-31). These findings show that this hybrid chitin-coffee ground biochar foam offers an affordable and sustainable solution for managing microplastic pollution in water.

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CONFLICT OF INTEREST

The author declares that there are no conflict of interests related to this work.

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