

Article

Adsorptive Removal of Sunscreen-Derived Benzophenone-3 Using Iron-Impregnated Biochar Fabricated with *Chlorella pyrenoidosa* Biomass

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Abstract

Benzophenone-3 (BP-3), an organic UV filter extensively applied in sunscreens, cosmetics and daily plastic products, is classified as a typical emerging endocrine-disrupting micropollutant. This compound is highly susceptible to bioaccumulation in aquatic organisms, triggers coral bleaching, and incurs oxidative damage to algae, fish and invertebrates. Conventional wastewater treatment processes cannot efficiently eliminate BP-3 from aqueous media, thereby imposing severe ecological risks on freshwater and marine ecosystems. In this study, iron-impregnated biochar (Fe-BC) was synthesized via an impregnation–pyrolysis route using powder of cultivated *Chlorella pyrenoidosa* (green microalga) as raw feedstock. Batch adsorption experiments revealed that iron impregnation remarkably enhanced the removal efficiency of BP-3. The maximum Langmuir saturated adsorption capacity of Fe-BC reached 91.7 mg/g, considerably exceeding the value of 51.5 mg/g for pristine biochar. Kinetic data exhibited favorable fitting with the pseudo-first-order kinetic model, demonstrating that Fe-BC rapidly captures BP-3 and achieves adsorption equilibrium within 120 min. Solution pH exerted a prominent influence on adsorption performance: the material maintained high BP-3 adsorption capacity at pH 7–10, whereas adsorption capacity declined drastically under strongly acidic (pH < 5) and extreme alkaline conditions (pH > 10.5). Fourier-transform infrared spectroscopy (FTIR) validated the successful loading of iron species onto the biochar surface, as well as the binding of BP-3 onto Fe-BC. Combined with pH-controlled experimental results, hydrogen bonding, hydrophobic interactions, and pore-filling effects are inferred as the dominant adsorption mechanisms for BP-3 removal. Furthermore, Fe-BC retained favorable BP-3 removal performance in simulated seawater matrices, endowing it with preliminary potential for wastewater treatment in coastal zones and tourist scenic areas. This work offers basic laboratory insights into BP-3 adsorption, while further verification concerning environmental low-concentration conditions, authentic water matrices, material reusability and stability is essential for its practical application.

Keywords: sunscreen-derived Benzophenone-3; endocrine-disrupting micropollutant; adsorptive removal; iron-impregnated biochar; *Chlorella pyrenoidosa* biomass



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1. Introduction

Oxybenzone is a widely utilized organic ultraviolet (UV) filter incorporated into various personal care products, including sunscreens, cosmetics, and toothpaste, due to its efficacy in absorbing UV radiation. Beyond personal care, oxybenzone is also added to consumer goods such as textiles, glasses, plastics, and coatings, serving as a UV stabilizer. Its extensive application has led to concerns regarding its environmental persistence and potential adverse effects on aquatic ecosystems, as oxybenzone is known to be resistant to biodegradation and can accumulate in water bodies through various pathways, including direct application and wastewater discharge [1,2].

Oxybenzone pollution in aquatic environments, including rivers, wastewater treatment plant (WWTP) effluents, and oceans, has become a significant environmental concern. In rivers, oxybenzone concentrations have been reported to range from nanograms per liter (ng/L) to micrograms per liter ($\mu\text{g/L}$), with higher levels often detected near urban areas and recreational sites where sunscreen use is prevalent [3]. Similarly, WWTP effluents are recognized as major sources of oxybenzone pollution, as conventional wastewater treatment processes are inefficient in removing this compound, leading to its release into receiving waters at concentrations ranging from ng/L to mg/L levels [4]. In coastal and marine environments, oxybenzone concentrations can vary widely depending on proximity to human activities. Studies have shown that oxybenzone levels in coastal waters can reach up to several $\mu\text{g/L}$, particularly in areas with high tourist activity and coral reefs [5]. Furthermore, the compound's lipophilicity and persistence enable it to bioaccumulate in aquatic organisms, posing a threat to marine ecosystems. The presence of oxybenzone in the Weser estuary of the North Sea, for instance, has been linked to significant ecological risks, with calculated risk quotients indicating potential impacts on marine algae such as *Isochrysis galbana* [6]. The continuous release and accumulation of oxybenzone in aquatic environments underscore the urgent need for effective management strategies to mitigate its ecological impacts.

Oxybenzone exhibits a range of toxic effects on aquatic organisms, including phytoplankton, zooplankton, and higher trophic levels. For phytoplankton, such as microalgae, oxybenzone has been shown to inhibit growth, disrupt photosynthetic processes, and induce oxidative stress [7]. Studies on freshwater microalgae, including *Selenastrum capricornutum* and *Chlorella vulgaris*, have demonstrated that oxybenzone exposure leads to reduced cell proliferation, altered pigment content, and impaired photosynthetic efficiency, ultimately affecting energy conversion and substance metabolism [1,7]. In marine diatoms, another important group of phytoplankton, oxybenzone exposure has been linked to growth inhibition, membrane integrity disruption, and increased malondialdehyde (MDA) levels, indicating lipid peroxidation and cellular damage [2]. Furthermore, oxybenzone can induce the overproduction of reactive oxygen species (ROS), leading to oxidative stress and potential damage to cellular components, including DNA, proteins, and lipids [8]. Zooplankton, such as crustaceans and sea urchins, are also susceptible to oxybenzone toxicity, with observed effects including reduced survival, impaired reproduction, and developmental abnormalities [9]. These toxic effects can cascade through the food web, affecting higher trophic levels and overall ecosystem health. The widespread presence of oxybenzone in aquatic environments, combined with its bioaccumulative properties and toxic effects on aquatic organisms, highlights the need for comprehensive risk assessments and regulatory measures to protect aquatic ecosystems from this emerging pollutant.

Benzophenone-3 (2-hydroxy-4-methoxybenzophenone, BP-3) is among the most frequently utilized benzophenone derivatives [10–12]. The presence of BP-3 in the Weser estuary of the North Sea has been found to pose a significant risk to the plankton [6]. Water contaminated with organics such as PPCPs has been purified by various methods such as

electrochemical processes [13], oxidation (including advanced oxidation processes) [14,15], and biotransformation [16]. However, further work is required for developing effective ways to purify contaminated water, and adsorption might be one such strategy considering the low cost (especially cheap investment), mild operation conditions, and wide range of applications [17–19]. For efficient adsorptive removal, however, effective adsorbents (with high capacity, high selectivity, and ready reusability) are essential in commercial applications. To date, only a limited number of adsorbents have been developed for the adsorptive treatment of BP-3, including waste biomass of plant origin [20], polyaniline-derived porous carbons [21], and porous carbon derived from metal azolate framework-6 [22]. Previous studies have demonstrated that carbon-based materials or biomass with abundant pore structures and hydroxyl groups hold great potential for development as BP-3 adsorbents [23]. This suggests that using plant biomass as an adsorbent material represents a promising avenue; moreover, existing research indicates that hydrogen bonding is the primary mechanism underlying BP-3 removal [22]. However, whether the modification of algal biochar with metal ions can achieve satisfactory adsorption performance remains unclear.

Chlorella pyrenoidosa was selected as the precursor material for several reasons. First, this microalga is a large-scale-cultivable microalga with favorable material accessibility. Moreover, this microalga can proliferate rapidly in nutrient-rich water, generating substantial algal residues after harvesting for lipid production or wastewater-treatment operations. Such residues represent an abundant, low-cost, and underutilized waste-biomass resource suitable for circular-economy applications. Second, microalgal biomass naturally contains abundant nitrogen-containing functional groups, which may provide more binding sites after pyrolysis compared with many lignocellulosic terrestrial biomasses [24]. Nevertheless, pristine microalgae-derived biochar still suffers from moderate adsorption capacity and unsatisfactory anti-interference performance under high-salinity coastal seawater conditions, limiting its practical application for marine-relevant BP-3 removal. Iron impregnation-pyrolysis modification is expected to overcome these drawbacks. Compared with unmodified raw biomass and pristine biochar adsorbents reported in previous studies, iron loading can (i) create abundant Fe-O-containing active sites and enrich surface hydroxyl groups for hydrogen-bonding interactions with BP-3; (ii) etch carbon skeletons during pyrolysis to optimize pore architecture and facilitate pore-filling of BP-3 molecules; and (iii) maintain dominant non-electrostatic adsorption mechanisms (hydrogen-bonding and hydrophobic interactions). In this study, we fabricated iron-impregnated derived biochar (Fe-BC) and applied it to the adsorptive purification of water contaminated with BP-3. The potential interaction mechanisms between Fe-BC and BP-3 were explored *via* Fourier-transform infrared spectroscopy and pH-controlled experiments. Additionally, we investigated the adsorption equilibrium and kinetic processes, with a specific focus on evaluating the applicability of the adsorbent under seawater conditions. We anticipate that these findings will provide valuable guidance for the adsorptive treatment of BP-3.

2. Materials and Methods

2.1. Chemical Reagents

BP-3 (CAS: 131-57-7, 99%) was purchased from Macklin (Shanghai, China). The adsorbate was used directly without further purification. Biological activated carbon was derived from *Chlorella pyrenoidosa* pre-cultured using the method described by Li et al. [24]. After the algal cells reached the late exponential growth phase, the biomass was harvested via centrifugation at $10,000 \times g$, followed by vacuum freeze-drying. Prior to biochar preparation, the biomass was sieved through a 20-mesh sieve to ensure uniform particle size (0.85 mm).

2.2. Preparation of Carbon-Based Adsorbents

Fifty grams of plant biomass powder (PBP) was weighed accurately and placed into a 2 L beaker. Eight hundred milliliters of FeCl_3 solution (Fe^{3+} concentration: 2 mol/L) was added, and the mixture was stirred with a magnetic stirrer at 300 rpm for 24 h to ensure sufficient adsorption of FeCl_3 by the biomass, resulting in a biomass- FeCl_3 suspension. The suspension was placed in a forced-air oven at 70 °C for 12 h to gradually reduce the water content, with manual stirring every 2 h during the process to prevent agglomeration. When the suspension turned into a thick paste (water content: 68–78%, determined by oven-drying method), it was centrifuged at $8000 \times g$ for 15 min to obtain a solid product. The solid was transferred to a quartz boat and pyrolyzed in a vacuum tube furnace under a continuous N_2 flow (purity: 99.99%) at a flow rate of 50 mL/min, heating from room temperature to 400 °C at a rate of 8 °C/min and maintaining the temperature for 2 h. The selection of this condition was determined through preliminary optimization experiments conducted in our laboratory (unpublished data). A pyrolysis temperature of 400 °C and holding time of 2 h were adopted in this work. Lower temperatures (<350 °C) cannot sufficiently decompose algal organic components and form well-developed porous carbon frameworks, leading to low adsorption capacity. Excessively high temperatures (>500 °C) would cause severe sintering and agglomeration of loaded iron oxide nanoparticles, reduce surface hydroxyl-rich active sites, and further consume carbon matrix. A 2 h holding duration guarantees thorough carbonization and sufficient formation of Fe-O active sites, while longer holding time brings negligible performance improvement but increases energy consumption [25–27]. Detailed orthogonal optimization results regarding pyrolysis temperature and holding time will be presented in our follow-up dedicated study. After the biochar cooled to room temperature naturally under N_2 atmosphere, it was ground using an agate mortar and sieved through a 100-mesh sieve to obtain Fe-modified biochar (Fe-BC). For preparation of pristine biochar (BC, control group): equal mass of raw PBP was immersed in deionized water following identical stirring, drying and centrifugation procedures without FeCl_3 addition and then pyrolyzed under exactly the same heating and N_2 atmosphere conditions as Fe-BC to guarantee single variable (only iron impregnation differs between BC and Fe-BC).

2.3. Determination of the Oxybenzone

The quantification of BP-3 was performed via high-performance liquid chromatography (HPLC) following a previously reported protocol [28]; briefly, 100 mg of BP-3 reference standard was accurately weighed into an amber volumetric flask, dissolved in methanol and diluted to a final volume of 10 mL to prepare a 10 mg/mL stock standard solution stored under refrigeration, after which a series of mixed working standard solutions at concentrations of 100, 50, 10, 5 and 1 µg/mL were prepared for calibration curve construction by HPLC and preserved under refrigeration. The HPLC operating conditions were set as follows: Poreshell 120 EC-C18 chromatographic column (4.6 mm $\times$ 150 mm, particle size of 4 µm, Agilent Technologies, Inc., Santa Clara, CA, USA), column temperature of 35 °C, mobile phase consisting of aqueous solution containing 0.1% formic acid (Phase A) and ethanol-methanol mixture at a volume ratio of 1:3 (Phase B), flow rate of 1.0 mL/min, detection wavelength of 311 nm, and injection volume of 20 µL. For sample testing, 20 µL of sample extract was precisely injected and determined under the above chromatographic conditions; upon completion of the experiment, the corresponding peak areas of samples were recorded, and the concentrations of analytes were calculated according to the linear relationship between peak area and concentration.

2.4. Adsorption Tests

BP-3 was first dissolved in deionized water containing 5% methanol to prepare an aqueous solution with a fixed concentration of 50 mg/L. It should be noted that methanol may act as a competing adsorbate and potentially interfere with BP-3 adsorption behavior. Therefore, all final working adsorption solutions were prepared by diluting the stock solution so that the residual methanol fraction in the adsorption system was far below 5%. Owing to the intrinsic poor water solubility of BP-3, it is technically difficult to perform complete parallel adsorption tests in 100% pure water at the investigated concentration range. The adsorption results reported in this work are obtained under the condition of trace residual methanol, which should be taken into account when comparing with data from purely aqueous systems in other studies in the literature [29]. Considering the typical pH range of river water and rainwater, the pH of the solution was adjusted to 7 by adding an appropriate amount of 0.1 mol/L sodium hydroxide. Stock BP-3 solution was serially diluted with deionized water to obtain BP-3 solutions with concentrations ranging from 5 to 50 mg/L. Prior to adsorption experiments, all adsorbents were dried at 100 °C for 12 h in an oven equipped with a vacuum pump. A predetermined mass of adsorbent was introduced into 200 mL of each prepared BP-3 solution at a fixed adsorbent dosage of 0.75 g/L. The adsorption reactions were performed at 25 °C for 12 h in a thermostatted shaker operating at a constant agitation speed of 250 rpm. The adsorption capacity (q_t , mg/g) and removal efficiency (R , %) were calculated by the following formulas:

$$q_t = \frac{(C_0 - C_t)V}{W}$$

$$R = \frac{(C_0 - C_t)}{C_0} \times 100\%$$

where C_0 and C_t are the initial concentration and the concentration at time t (h) of BP-3 (mg/L), respectively. At adsorption equilibrium, C_t is substituted by equilibrium concentration C_e , and the corresponding adsorption capacity was expressed as q_e . V stands for the volume of solution (L), and W denotes the mass of adsorbent (g).

Langmuir adsorption isotherm, Freundlich adsorption isotherm and Sips adsorption isotherm were adopted to describe the adsorption equilibrium behavior of BP-3 onto adsorbents, while the pseudo-first-order kinetic model and pseudo-second-order kinetic model were used to characterize the dynamic adsorption process. The mathematical formulas of each model are presented as follows. All these models were first processed via linear regression using transformed linear forms of the respective equations.

$$\text{Langmuir isotherm: } \frac{1}{q_e} = \frac{1}{Q_{\max}K_L C_e} + \frac{1}{Q_{\max}}$$

$$\text{Freundlich isotherm: } \lg q_e = \frac{1}{n} \lg C_e + \lg K_F$$

$$\text{Sips isotherm: } q_e = \frac{Q_{\max-s} K_S C_e^{N_S}}{1 + K_S C_e^{N_S}}$$

$$\text{Pseudo-first-order kinetic model: } \lg(q_e - q_t) = \lg q_e - \frac{k_1 t}{2.303}$$

$$\text{Pseudo-second-order kinetic model: } \frac{t}{q_t} = \frac{1}{K_2 q_e^2} + \frac{t}{q_e}$$

where q_e , C_0 and C_t denote the same parameters as defined above; $Q_{\max}$ represents the maximum adsorption capacity obtained from Langmuir adsorption isotherm (mg/g); $Q_{\max-s}$ represents the maximum adsorption capacity obtained from Sips adsorption isotherm

(mg/g); K_L denotes the Langmuir adsorption constant (L/mg); K_S is the KS is the Sips constant, while n (reflecting adsorption intensity) and K_F are the Freundlich constants; K_1 is the pseudo-first-order adsorption rate constant (/min), and K_2 is the pseudo-second-order adsorption rate constant (g/mg/min). N_S represents the heterogeneity of the adsorbent.

2.5. Analysis of Adsorption Mechanisms

The adsorption mechanism of modified activated carbon was investigated via two approaches, namely adsorption equilibrium experiments in liquid-phase systems with varying pH values and FTIR characterization. First, BP-3 solutions with pH ranging from 3.0 to 12.0 were prepared by adding 0.1 mol/L HCl or NaOH aqueous solutions. A fixed dosage of 100 mg adsorbent was introduced into each solution, and the corresponding q_t values under different pH conditions were compared. Second, FTIR spectra of adsorbents before and after adsorption were contrasted to identify variations in surface functional groups. Vacuum freeze-dried adsorbent samples were placed on a sample stage for spectral analysis using a Thermo Scientific Nicolet iN10 Fourier-transform infrared micro-FTIR spectrometer (Thermo Electron Scientific Instruments LLC, Madison, WI, USA). All measurements were conducted in transmission mode with scanning wavenumbers ranging from 400 to 4000/cm. The spectral resolution was set to 4/cm, and the data acquisition time for a single scan was 3 s.

Simulated seawater was prepared by dissolving commercial sea salt (Qingdao Haizhiyan Aquarium Technology Co., Ltd., Qingdao, China) in Milli-Q water. No natural organic matter or trace organic pollutants were supplemented in this salt-only simulated seawater.

2.6. Statistical Tests

All data presented in the figures are expressed as mean values of replicates $\pm$ standard error (SE) calculated from these three independent replicates. One-way analysis of variance (one-way ANOVA) was performed for statistical comparisons using SPSS Statistics 26.0. Normality test and homogeneity of variance test were conducted on all datasets. Differences with $p < 0.05$ were considered statistically significant. All figures were plotted using Origin software 2025.

3. Results

3.1. Effect of Aqueous Phase pH on the Adsorption Capacity of Adsorbents

The solution pH exerts a remarkable influence on the equilibrium adsorption capacity of the adsorbent (q_e) and the pollutant removal efficiency (R), as shown in Figure 1. Within the pH range of 3–10, both the parameters increase continuously with rising pH value. The adsorption performance is the poorest under strongly acidic conditions (pH = 3), where the q_e value is merely 13.32 mg/g with a removal efficiency below 20%. As the competitive effect of hydrogen ions weakens, the adsorption efficiency rises sharply at pH 7, with q_e reaching 59.37 mg/g. A stable and high-efficiency adsorption plateau is observed at pH 7–10, and the optimal adsorption performance is achieved at pH 10, with a q_e of 61.08 mg/g and a removal efficiency of 91.6%. When the system shifts to strongly alkaline environments at pH > 10, excessive hydroxide ions interfere with the interfacial interactions during adsorption, leading to a slight decline in adsorption capacity; the q_e decreases to 57.80 mg/g at pH 12. The optimal applicable pH range for this adsorbent is determined to be 8–10.

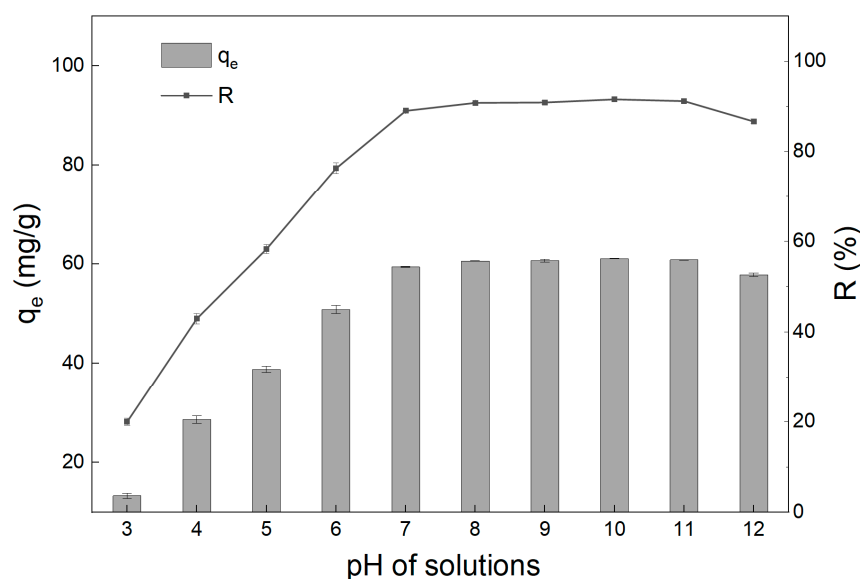


Figure 1. Effects of pH on adsorbed amounts of BP-3 and removal efficiency over Fe-BC. The adsorption temperature, initial BP-3 concentration and contact time were 25 °C, 50 mg/L and 12 h, respectively.

3.2. FTIR Characterization of Biochar and Its Adsorption Mechanism

The band at 3335 cm^{-1} corresponds to free (non-hydrogen-bonded) hydroxyl groups. The absorption peak at 1720 cm^{-1} is assigned to C=O stretching vibration [1], which is mainly derived from ketone, aldehyde or carboxylic acid groups. The peak at 1647 cm^{-1} is identified as the N-H stretching vibration, while it should be noted that this region overlaps with the bending vibration of adsorbed water. The absorption signal at 1585 cm^{-1} is attributed to vibrational modes of secondary amines. The band located at 3610 cm^{-1} is ascribed to stretching vibrations of aromatic hydroxyl groups [30], originating from aromatic functional groups formed during pyrolysis. Meanwhile, the disappearance of characteristic peaks assigned to ketones, aldehydes and carboxylic acids indicates the dehydrogenation and aromatization of organic components in biomass. Two prominent spectral variations are observed after iron impregnation (Figure 2). A distinct peak emerges at 1035 cm^{-1} , corresponding to C-OH stretching vibration. The newly formed band at 512 cm^{-1} originates from Fe-O bond vibration within iron-containing particles. The newly appeared absorption band near 512 cm^{-1} may be assigned to Fe-O-related vibrational modes. This spectral feature is consistent with the existence of iron-oxide-related species on Fe-BC. Nevertheless, FTIR data alone are insufficient to confirm the exact crystalline phase, chemical state, or spatial distribution of these iron-containing species. Further complementary characterizations such as XRD, XPS or SEM-EDS would be required for such definitive identification. In the FTIR spectrum of Fe-BC-BP-3, the stretching vibration peak of hydroxyl groups shifts to a lower wavenumber at 3380 cm^{-1} . This spectral change is consistent with possible interactions between BP-3 molecules and surface hydroxyl groups on Fe-BC. However, such an FTIR peak shift alone cannot constitute definitive proof that hydrogen bonding acts as the dominant adsorption mechanism. Other concurrent surface-level changes may also contribute to this band shift. New peaks around 1687 cm^{-1} and 1369 cm^{-1} are assigned to the carbonyl stretching vibration and conjugated aromatic C=C vibration of BP-3, respectively [23]. The band at 1214 cm^{-1} corresponds to the C-O stretching vibration introduced by adsorbed BP-3. These results confirm that BP-3 is adsorbed onto the surface of the adsorbent and reveal that surface hydroxyl groups and iron-related sites participate in the adsorption of BP-3.

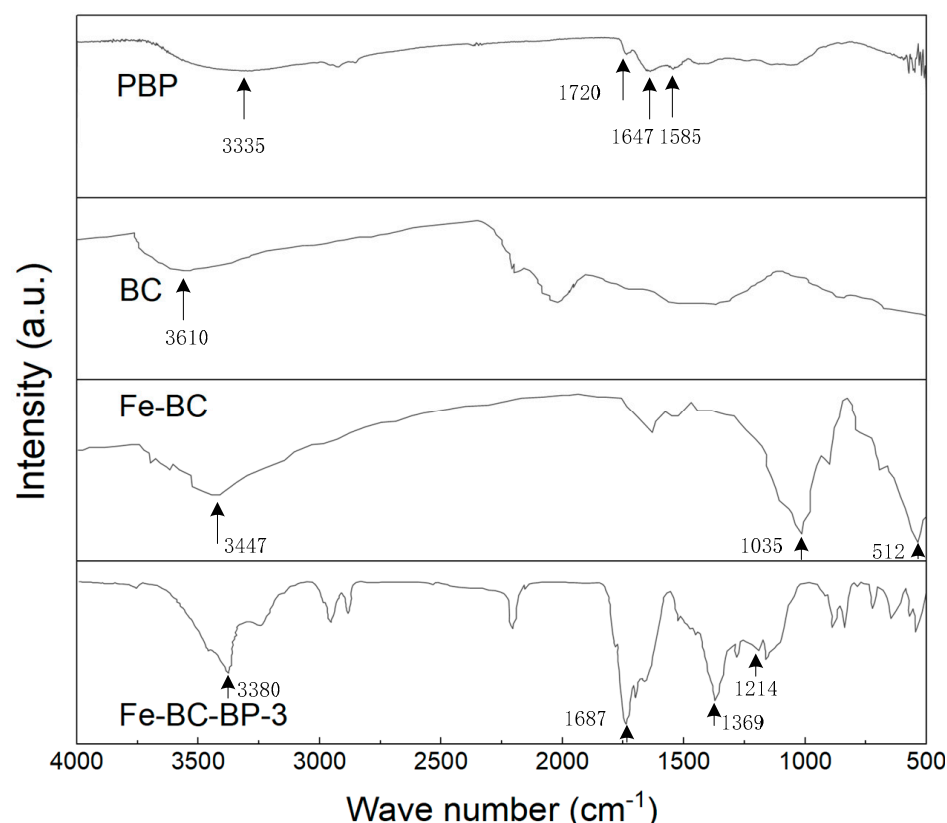


Figure 2. FTIR spectra of PBP, raw BC, Fe-BC, and Fe-BC after BP-3 adsorption (Fe-BC-BP-3) over the wavenumber range of 4000–500 cm^{-1} .

3.3. Adsorption Equilibrium

Figure 3 presents the linear fittings of the Langmuir and the Freundlich adsorption isotherms. Table 1 summarizes the isotherm parameters of the Langmuir and Freundlich models for BP-3 adsorption onto PBP, BC and Fe-BC. The Sips isotherm model was further fitted to the equilibrium data to probe the energetic heterogeneity of adsorption sites, and the corresponding fitted parameters are also compiled in Table 1. For PBP and Fe-BC, the Langmuir model yielded higher R^2 values (0.9794 and 0.9976) compared with the Freundlich model, indicating monolayer-dominated adsorption. By contrast, BC fitted better to the Freundlich model ($R^2 = 0.9936$). The Sips model-derived heterogeneity parameter (N_S) provides supplementary clues regarding site energy distribution. For raw BC, the deviation of N_S away from unity suggests substantial surface heterogeneity. In comparison, PBP and Fe-BC exhibited N_S values closer to 1, which tentatively implies relatively more homogeneous adsorption site energetics after iron impregnation modification. Nevertheless, these model-based outputs are treated only as qualitative hints, rather than definitive proof of surface homogeneity or heterogeneity, since isotherm fitting alone cannot fully disentangle complex surface-site behaviors. It should be noted that high R^2 values only reflect the goodness-of-fit of regression and cannot independently serve as definitive mechanistic evidence for monolayer or heterogeneous multilayer adsorption. Combined with FTIR characterization and pH-dependent adsorption results discussed in subsequent sections, Fe-BC tends toward relatively homogeneous-site-governed adsorption behavior after iron impregnation modification, whereas pristine BC exhibits more heterogeneous surface characteristics. The Langmuir-predicted maximum adsorption capacity q_{max} followed the order of Fe-BC (91.7 mg/g) > BC (51.5 mg/g) > PBP (3.76 mg/g), confirming that iron-impregnated modification significantly boosted the BP-3 adsorption

performance of biochar. All adsorbents exhibited Freundlich n values > 1 , indicative of favorable adsorption toward BP-3.

Table 1. Adsorption constants and correlation coefficients for two adsorption isotherms of BP-3.

Absorbents	Langmuir Isotherm			Freundlich Isotherm			Sips Isotherm		
	$q_{\max}$ (mg/g)	K_L (mg/L)	R^2	K_F	n	R^2	$Q_{\max-S}$	N_S	R^2
PBP	3.76	0.03	0.9794	5.15	1.65	0.9401	8.13	0.65	0.8400
BC	51.5	0.14	0.9551	6.85	1.78	0.9936	101.40	0.70	0.9766
Fe-BC	91.7	0.27	0.9976	15.0	2.22	0.9355	132.74	0.97	0.9571

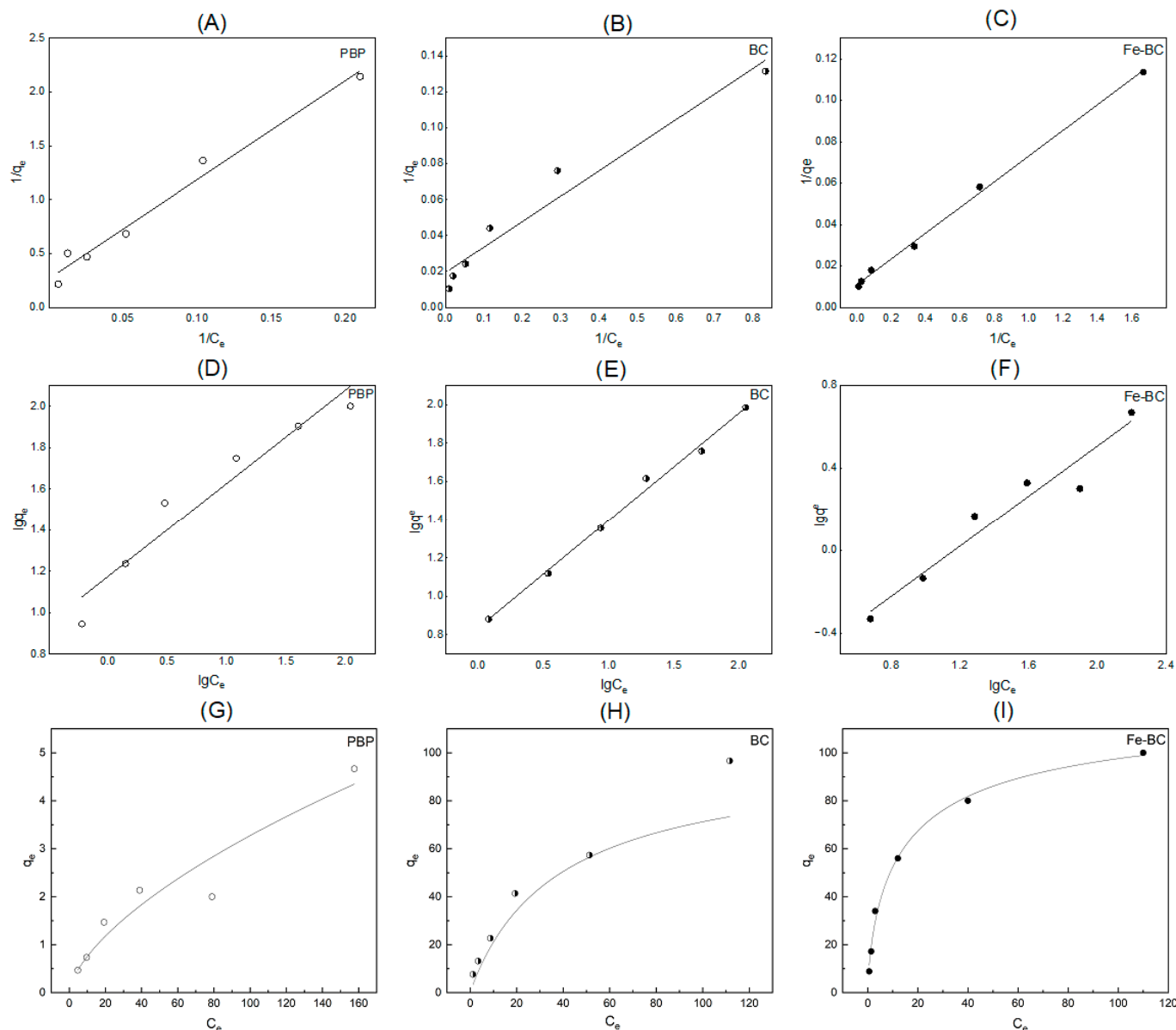


Figure 3. Linear fittings of the Langmuir (A–C), the Freundlich (D–F), and the Sips (G–I) adsorption isotherms of BP-3 onto PBP, BC and Fe-BC. Conditions: adsorbent dosage = 0.75 g/L, 25 °C, pH = 7.

3.4. Adsorption Kinetics

Adsorption kinetic experiments were performed to evaluate the adsorption rate and equilibrium time of BP-3 onto PBP, BC and Fe-BC, and the fitted parameters of pseudo-first-order and pseudo-second-order kinetic models are summarized in Table 2 (Figure 4). The

whole adsorption process can be divided into two phases: a fast adsorption stage within the initial 120 min, followed by a slow increasing stage approaching adsorption equilibrium. At the initial stage, abundant unoccupied surface sites on biochar were available, and BP-3 molecules rapidly diffused toward adsorbent surfaces. For Fe-BC, adsorption equilibrium was achieved within 120 min, demonstrating favorable mass-transfer performance.

As shown in Table 2, the correlation coefficients of the pseudo-first-order model for PBP, BC and Fe-BC were 0.9871, 0.9826 and 0.9915, respectively, which were obviously higher than those of the pseudo-second-order model. Moreover, the values calculated from the pseudo-first-order model were much closer to the experimental values. These results indicated that the BP-3 adsorption process over the three adsorbents complied better with the pseudo-first-order kinetic model, implying that physical-controlled processes dominated the overall adsorption kinetics. The equilibrium adsorption capacity followed the sequence Fe-BC > BC > PBP, which was consistent with the isotherm results. Iron-impregnated biochar supplied abundant surface active sites and optimized pore structure, accelerating BP-3 mass transfer and shortening the equilibrium time. It should be emphasized that the kinetic-fitting results obtained herein can only provide suggestive clues for possible adsorption behavior rather than conclusive mechanistic proof. Further dedicated diffusion analysis experiments would be required to identify whether mass transfer or intra-particle diffusion acts as the rate-limiting step.

Table 2. Kinetic parameters for BP-3 adsorption onto PBP, BC and Fe-BC.

Adsorbent	$q_{e, exp}$ (mg/g)	Pseudo-First-Order Model			Pseudo-Second-Order Model		
		$q_{e, cal}$ (mg/g)	K_1 ($\times 10^{-2}/\text{min}$)	R^2	$q_{e, cal}$ (mg/g)	K_2 ($\times 10^{-4}$ g/mg/min)	R^2
PBP	3.61	3.54	2.14	0.9871	3.28	9.26	0.9425
BC	48.23	47.68	1.67	0.9826	44.12	2.81	0.9374
Fe-BC	88.46	87.91	1.23	0.9915	82.35	1.45	0.9482

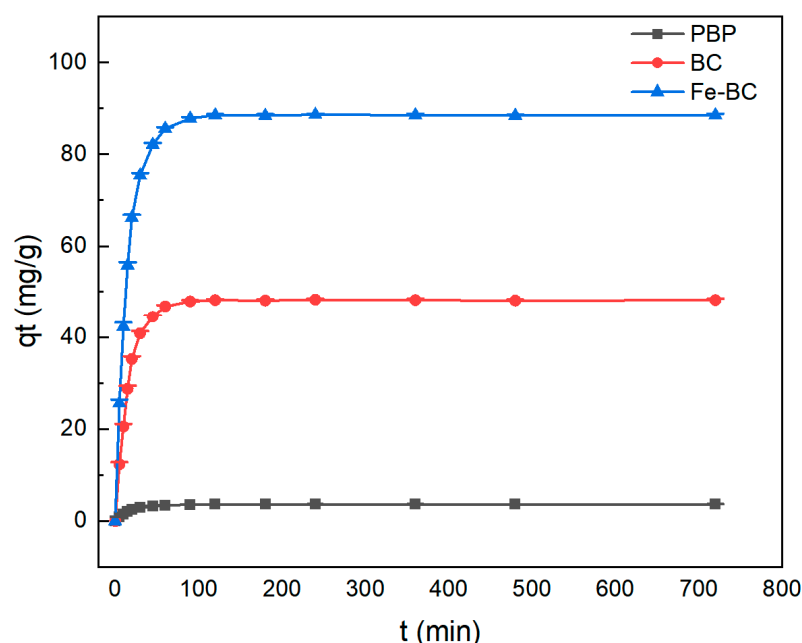


Figure 4. Adsorption kinetics of BP-3 onto PBP, BC and Fe-BC; fitting curves of pseudo-first-order and pseudo-second-order models. Conditions: adsorbent dosage = 0.75 g L^{-1} , 25°C , $\text{pH} = 7$.

3.5. Simulated Seawater Experiment

To test the practicability of Fe-BC in complex coastal water, adsorption experiments were conducted in simulated seawater (35 PSU; main ion composition: Na^+ , Ca^{2+} , Mg^{2+} ,

Cl^- and SO_4^{2-} matching natural seawater ratio) at an initial BP-3 concentration of 50 mg/L. Fe-BC achieved a BP-3 removal efficiency of 91.4% in deionized water and 90.5% in simulated seawater. Although abundant inorganic ions in seawater slightly competed for adsorption sites, Fe-BC maintained stable removal performance with weak matrix interference. This adsorbent is thus suitable for remediating BP-3 pollution in coastal and tourist marine wastewater.

4. Discussion

4.1. Promotion Mechanism of Iron Impregnation on Microalgal Biochar Adsorption Capacity

Raw *C. pyrenoidosa* powder displays weak BP-3 adsorption ability, with merely 3.76 mg/g, which can be attributed to undeveloped pore structures and insufficient exposed functional groups of uncarbonized algal biomass. Deyris et al. directly utilized waste plant biomass, including *Eichhornia crassipes* and *Fallopia japonica*, to adsorb aqueous BP-3, achieving nearly 90% removal efficiency under low-concentration water systems. Their study verified that natural lignocellulosic biomass possesses intrinsic adsorption affinity toward UV filters, yet they did not adopt metal modification strategies to further boost saturated adsorption capacities [20]. Direct pyrolysis converts microalgae into porous carbon material BC, which significantly elevates adsorption capacity to 51.5 mg/g, benefiting from carbonization-induced pore generation and surface aromatic functional group enrichment. Further ferric chloride impregnation–pyrolysis modification dramatically improves BP-3 uptake performance of biochar, with Langmuir monolayer saturated adsorption capacity reaching 91.7 mg/g for Fe-BC. Based on FTIR (Figure 2) results and the relevant published literature [31,32], it can be inferred that iron–salt pyrolysis is likely to form iron-oxide-related species anchored on biochar skeletons. Additional iron-based active sites and enriched hydroxyl groups jointly provide more binding sites for BP-3 molecules, thus substantially raising the saturated adsorption capacity of microalgal biochar. Moreover, iron ion etching during pyrolysis expands the internal pore volume of biochar, facilitating BP-3 molecule diffusion into inner pores and strengthening the pore-filling adsorption effect.

A variety of remediation technologies have been developed to eliminate BP-3 from aqueous matrices, each with inherent limitations. Advanced oxidation processes (AOPs), including ozonation, $\text{UV}/\text{H}_2\text{O}_2$, persulfate activation and sonochemical oxidation, can achieve high BP-3 degradation efficiency (91–98%), yet they suffer from high energy consumption, expensive reagent costs, and the risk of generating toxic transformation byproducts that retain aquatic toxicity [10,16]. Bioremediation approaches relying on microalgae, fungi or aquatic plants require long treatment cycles and are vulnerable to fluctuations in water temperature, salinity and coexisting pollutants; conventional activated sludge systems also fail to fully intercept BP-3 during municipal wastewater treatment [17,19]. Membrane separation technologies such as ultrafiltration can remove up to 87% BP-3 but are limited by membrane fouling and high operating expenses [23]. Among adsorption materials previously reported for BP-3 removal, commercial activated carbon exhibits excellent uptake capacity but suffers high raw material costs; terrestrial plant biomass carbons display moderate adsorption performance while lacking anti-interference capacity in high-salinity seawater; MOF-derived porous carbon materials deliver outstanding adsorption efficiency but rely on costly synthetic precursors and complex preparation procedures [21,22].

In the present study, iron-modified biochar (Fe-BC) was fabricated from *C. pyrenoidosa* microalgal biomass via a facile impregnation–pyrolysis route. Fe-BC integrates multiple merits including low raw material cost, straightforward synthesis procedure, high monolayer adsorption capacity, and strong resistance to interference by inorganic ions in seawater. This material expands the repository of low-cost biomass-based adsorbents for

BP-3 remediation, delivers a novel microalgae biomass valorization technical strategy for eliminating sunscreen-derived organic UV filters in complex coastal water matrices, and fills the research gap regarding the application of iron-modified microalgal biochar for BP-3 adsorption.

4.2. Adsorption Equilibrium and Kinetic Mechanism Analysis

Isotherm fitting results demonstrated that Fe-BC follows Langmuir monolayer uniform adsorption behavior, which implies that iron impregnation homogenizes the surface chemical environment of algal biochar. The slight deviation between experimental equilibrium capacity (88.46 mg/g at pH = 7) and the Langmuir theoretical model (q_{max}) (91.7 mg/g) is reasonable: q_{max} is a theoretical parameter fitted by multi-concentration gradient isotherm tests, while kinetic and pH experiments adopt a single initial BP-3 concentration of 50 mg/L without reaching complete saturated monolayer coverage. Pseudo-first-order kinetic models better describe the whole adsorption process of BP-3 on three adsorbents, confirming that physical mass transfer, including external surface diffusion and internal pore diffusion, acts as the rate-limiting step of adsorption. The fast equilibrium within 120 min of Fe-BC reveals that iron impregnation optimizes pore channel structure and reduces mass transfer resistance of organic pollutant molecules, which is favorable for rapid on-site water treatment engineering applications. Notably, iron impregnation uniformly disperses proposed iron-oxide-related species across the carbon skeleton and homogenizes surface active sites, which explains the monolayer Langmuir fitting behavior despite heterogeneous iron species.

4.3. pH-Dependent Adsorption Regulation Mechanism

BP-3 remains predominantly neutral below its pK_a ; hence, hydrophobic interactions and hydrogen bonding serve as major driving forces for adsorption across acidic-to-near-neutral pH ranges. Nevertheless, at strongly acidic pH 3, abundant aqueous H^+ competes for oxygen-rich surface functional groups of Fe-BC, interfering with hydrogen-bond formation between Fe-BC and neutral BP-3 molecules and lowering adsorption efficiency. With increasing pH from 3 to 7, H^+ -site competition diminishes. Since BP-3 is still largely molecular under these conditions, hydrophobic partitioning and hydrogen-bonding interactions are restored, which accounts for the marked enhancement in adsorption. At pH values above the pK_a , partial deprotonation of BP-3 generates anionic species; additional electrostatic effects may also contribute to the overall adsorption behavior (Figure 1). Within the neutral to weakly alkaline range (pH 7–10), H^+ competitive interference fades; abundant neutral BP-3 molecules form stable hydrogen bonds with surface -OH groups of Fe-BC, and hydrophobic interaction between the aromatic BP-3 skeleton and carbonized biochar matrix is strengthened, generating the optimal adsorption plateau. At pH > 10, excessive OH^- ions bind with surface iron sites and hydroxyl hydrogen protons, breaking hydrogen bond networks between Fe-BC and BP-3 and weakening hydrophobic affinity, resulting in mild adsorption efficiency decline. The optimal applicable pH window (8–10) matches the natural pH range of most coastal seawater, which lays a foundation for Fe-BC application in offshore pollution remediation. Combined with the FTIR peak shift of hydroxyl groups, hydrogen bonding, hydrophobic interaction and the pore-filling effect are synergistic core mechanisms controlling BP-3 adsorption.

The pH-dependent adsorption trend of BP-3 on Fe-BC can be further explained by the acid dissociation constant (pK_a) of BP-3. The previous literature confirmed that the neutral molecular form of BP-3 possesses pK_a values of 7.6, 7.8 and 8.1 [33,34], indicating that BP-3 mainly exists as electrically neutral molecules under pH < 7.6–8.1, while it gradually deprotonates to form anionic species when pH exceeds this pK_a threshold. According to

acid–base speciation theory, around pH 8, neutral and anionic BP-3 co-exist at comparable fractions; at pH 9–10, the anionic deprotonated form becomes the dominant species in solution. At pH 2–7, BP-3 predominantly exists as neutral molecules. Neutral BP-3 carries no electrostatic repulsion with the slightly negatively charged carbon skeleton of Fe-BC, and abundant aromatic structures enable strong hydrophobic affinity between adsorbate and adsorbent. Meanwhile, neutral BP-3 contains free phenolic -OH groups that readily form hydrogen bonds with the rich hydroxyl groups anchored on iron oxide sites of Fe-BC, jointly maintaining high adsorption capacity in this pH interval. Within pH 8–10, anionic BP-3 becomes progressively dominant, especially at pH 9–10. Even so, Fe-BC still maintains favorable BP-3 removal. This observation implies that hydrophobic interactions and hydrogen-bonding contributions remain substantial, even for deprotonated BP-3 anions. Although electrostatic repulsion emerges between negatively charged anionic BP-3 and the deprotonated, negatively charged surface of Fe-BC, this repulsive effect is not sufficient to offset the non-electrostatic adsorption driving forces, forming the stable high-efficiency adsorption plateau observed in the pH gradient experiment. At pH > 10.5 (far higher than pK_a), massive BP-3 molecules lose phenolic hydrogen and transform into negatively charged anions. Meanwhile, high-concentration OH^- in the solution deprotonates most surface -OH groups on Fe-BC, turning the adsorbent surface highly electronegative. Electrostatic repulsion arises between anionic BP-3 and negatively charged Fe-BC surfaces; additionally, excess hydroxide ions compete for hydrogen bond binding sites, jointly reducing the equilibrium adsorption capacity of Fe-BC. This pK_a characteristic of BP-3 perfectly interprets the pH response curve in Figure 1, further verifying that hydrogen bonding and hydrophobic interactions rather than electrostatic attraction act as the dominant adsorption driving forces between Fe-BC and BP-3.

4.4. Anti-Interference Performance in Simulated Seawater and Practical Application Prospects

High-salinity seawater contains massive coexisting inorganic ions, such as Na^+ , Ca^{2+} , Mg^{2+} , Cl^- and SO_4^{2-} , which generally compete for adsorption sites and suppress organic pollutant removal on conventional carbon adsorbents. However, Fe-BC only exhibits a 0.9% drop in BP-3 removal efficiency in simulated seawater relative to pure deionized water, showing moderate anti-interference performance. This stable performance originates from multiple synergistic adsorption mechanisms of Fe-BC: in addition to weak electrostatic attraction, BP-3 capture relies on hydrogen bonding and hydrophobic interaction, which are barely interfered with by background inorganic ions in seawater.

As shown in Table 3, various carbon-based and MOF-derived adsorbents have been reported for BP-3 removal. Although MOF-derived carbons (CDM-6 and PDC-800) possess ultra-high adsorption capacities, their expensive precursors and complicated fabrication procedures may restrict practical large-scale applications. Natural plant-based biosorbents exhibit ultra-low cost yet suffer from poor adsorption performance. In comparison, the as-prepared Fe-BC is fabricated from readily available microalgal biomass via a facile one-pot synthetic route and delivers competitive adsorption capacity. More importantly, Fe-BC exhibits excellent salt tolerance, rendering it a promising candidate for remediating high-salinity coastal water contaminated with BP-3.

This study investigates static BP-3 adsorption by Fe-BC under lab conditions, with several limitations hindering its large-scale application. Batch-only tests cannot reflect continuous-flow performance; fixed-bed column trials are required to evaluate breakthrough curves, treatment volume and long-term stability. Adsorbent recyclability and regeneration strategies (ethanol elution, thermal treatment) remain unoptimized. Moreover, competitive adsorption with mixed pollutants in authentic coastal seawater requires verification to confirm Fe-BC's universal applicability. The applied BP-3 concentration

(50 mg/L) is orders of magnitude higher than environmental trace levels (ng–µg/L, <34.3 µg/L in typical seawater), even in heavily polluted tourist coral reef regions [5,35]. This concentration gap leaves Fe-BC's performance at trace pollution unvalidated, necessitating low-concentration tests for in situ coastal remediation evaluation. Nonetheless, high-concentration saturation tests help determine theoretical maximum adsorption capacity and core kinetic mechanisms, providing fundamental data for emergency disposal of sudden high BP-3 contamination at coastal bathing sites. Furthermore, this study lacks complementary characterizations, including X ray diffraction, scanning electron microscopy–energy-dispersive X ray spectroscopy, and Brunauer–Emmett–Teller pore structure analysis, which prevents direct observation of iron oxide nanoparticle formation and quantitative assessment of pore modification effects. These measurements will be conducted in follow-up work. Due to the lack of regeneration and cyclic reusability experiments, the long-term practical feasibility of Fe-BC cannot be verified based on the current results. Further systematic regeneration investigations are required before practical application.

Table 3. Comparison of representative adsorbents for aqueous benzophenone-3 (BP-3) removal. * This value represents adsorbed amount after 12 h contact rather than the Langmuir-fitted maximum adsorption capacity (q_{max}).

No.	Adsorbent	Precursor	Modification Method	q_{max} (mg/g)	Main Advantages	Main Limitations	Ref.
1	Spent coffee grounds Powder of <i>Eichhornia crassipes</i> , <i>Pistia stratiotes</i> , and <i>Fallopia japonica</i>	Dried plant biomass	Raw dried biomass	0.31–0.89	Ultra-low cost; waste biomass utilization	Very low capacity	[20]
2	PDC-800	Polyaniline (pANI)	Two-step pyrolysis + KOH activation at 800 °C	906 *	Ultra-high adsorption capacity; good reusability via solvent washing	Costly pANI precursor; complex two-step synthesis	[21]
3	CDM-6	MAF-6 (Zn-based azolate frameworks)	Pyrolysis at 800 °C followed by acid washing	440	Ultra-high adsorption capacity; good salt tolerance; easy solvent regeneration	Costly MOF precursor; complicated synthesis	[22]
4	MAF-6	Zn-based metal azolate framework-6	As synthesized, no pyrolysis	71	Intrinsic high surface area; porous MOF structure	Relatively low BP-3 adsorption capacity; potential aqueous-phase stability risk in complex water	[22]
5	Fe-BC	<i>Chlorella pyrenoidosa</i> microalgal waste biomass	FeCl ₃ impregnation + pyrolysis (400 °C, 2 h)	91.7	Waste microalgae source; facile one-pot synthesis; high salt tolerance; competitive capacity	-	This study

5. Conclusions

C. pyrenoidosa biomass was converted to iron-impregnated biochar (Fe-BC) via simple impregnation–pyrolysis for aqueous BP-3 elimination. Iron doping enriched surface hydroxyl and Fe–O sites and greatly boosted BP-3 uptake; Fe-BC complied with the Langmuir monolayer model ($q_{max} = 91.7$ mg/g) and pseudo-first-order kinetics, reaching equilibrium

within 120 min. The maximum BP-3 adsorption took place at pH 8–10, consistent with coastal seawater pH. FTIR and pH-dependent experiments suggest that hydrogen bonding, hydrophobic interactions, and pore-filling effects collectively control BP-3 adsorption. Fe-BC retained 90.5% BP-3 removal in simulated seawater, with slight ion interference. Compared with oxidation, membrane and other adsorbents, this microalgae-derived material boasts cost and ecological merits, supplementing green adsorbents and providing a novel strategy for coastal UV micropollutant treatment. However, all batch tests adopted 50 mg/L BP-3, far above natural trace levels; further column, regeneration and low-concentration experiments are required to validate its engineering practicability.

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