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SLUDGE-BASED CATALYSTS FOR THE REMEDIATION OF PAH-CONTAMINATED SEDIMENTS

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Highlights:

- biochar derived from sludge containing iron and manganese from domestic wastewater treatment was produced via pyrolysis and utilized to activate persulfate for the degradation of benzo(a)pyrene (BaP) in sediments;
- BaP removal efficiency of 99.85% under optimal conditions, which we believe can have important implications for environmental engineering practices, particularly in the remediation of contaminated sediments;
- the findings also contribute to the growing field of resource recovery and the development of cost-effective, eco-friendly catalysts from waste materials.

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Abstract. The advanced persulfate oxidation method is known for its efficiency, stability, and capacity to breakdown organic contaminants without secondary contamination. In this study, biochar derived from sludge containing iron and manganese from domestic wastewater treatment was produced via pyrolysis and utilized to activate persulfate for the degradation of benzo(a)pyrene (BaP) in sediments. Results demonstrated that biochar pyrolyzed at 800 °C for 2 hours exhibited the highest catalytic performance. Under optimal conditions: a catalyst concentration of 1 g/L, persulfate concentration of 0.4 mM, and a reaction pH of 3, BaP removal efficiency reached 99.85% after 180 min. Characterization of the biochar showed that it was rich in Fe and Mn oxides, along with functional groups such as C-O-C, C=C, and C=O, which facilitated the adsorption and catalytic degradation of BaP. Quenching experiments revealed that the degradation of BaP was driven by reactive species such as $\bullet SO_4^-$ and •OH radicals, as well as singlet oxygen ($^1O_2$). The integration of radical dand non-radical pathways was essential for effective BaP removal. This research presents an innovative method for managing polycyclic aromatic hydrocarbons (PAHs) contamination in sediments by utilizing sludge-derived biochar in conjunction with activated persulfate.

Keywords: sludge biochar, persulfate, benzo(a)pyrene, PAHs, sediments.

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1. Introduction

Sediment is a crucial natural foundation for the continued survival and growth of organisms (Ambade et al., 2022). Polycyclic aromatic hydrocarbons (PAHs), generated by exhaust from vehicles, fossil fuel burning, and anthropogenic organic matter, are deposited in sediments via air deposition, surface runoff, and wet deposition (Zhang et al., 2022). Since PAHs in enriched sediments are characterised by low water solubility and difficult biodegradation, they will eventually endanger organisms at all levels through the food chain and even human autochthonous health (Hong et al., 2022). Given the carcinogenic, teratogenic, and mutagenic consequences of PAHs on humans, U.S. EPA has designated this species as a priority control pollutant, which has practical implications for the cleanup of PAHs in sediments (Dai et al., 2022; Shahsavari et al., 2019; Kumari et al., 2023; Ma et al., 2023; Mallah et al., 2022). Currently, commonly used solid-phase PAHs remediation technologies include chemical oxidation, biological treatment, electrochemical remediation and drenching (Shih et al., 2016), of which biological treatment is the most widely used, but in the actual sediment remediation process is a long remediation cycle and high treatment costs. There is a pressing requirement to create- and cost-effective techniques for the cleanup of PAHs in sediments.

Advanced oxidative sediment remediation processes (AOPs) based on persulfate have attracted significant focus in lately. The primary benefits of the AOPs method include the enhanced oxidative capacity and stabilization of $\bullet SO_4^-$ in the sediment compared to •OH or O_3, along with a reduced cost (Shih et al., 2016). For example, Ma et al.

(2018) employed the activated persulfate method for the remediation of PAH-contaminated soils, achieving complete degradation of 57.3% of the PAHs present. Activation of persulfate is crucial for the efficient production of $\bullet SO_4^-$. Common techniques for activating persulfate include thermal, transition metal, ultraviolet, alkali, and electrochemical methods (Tian et al., 2021, 2023; Chen et al., 2019; Zhang et al., 2023). The methods in question are characterized by significant energy consumption during the activation process, as well as the issue of secondary environmental pollution resulting from the leaching of metal ions. Nevertheless, these challenges may be addressed through the utilization of non-metallic catalysts, specifically carbon-based catalysts, for the activation of persulfates (Juni et al., 2023; Gao et al., 2022). Low cost and resource recycling are two benefits of preparing catalysts from water treatment sludge as opposed to carbon-based catalysts such carbon nanotubes, reduced graphene oxide, activated carbon, and graphene (Yu et al., 2019, 2022). In the field of sludge-based activated carbon resource reuse, significant development has also been achieved in recent years. Applications of heavy metal adsorption: Chen et al. (2024) prepared sludge biochar pellets by adiabatic pyrolysis at 350 °C and 500 °C, and analysed the differences between the two by a variety of characterization means. Dynamic adsorption studies were performed to examine the purifying efficacy of SBP produced by pyrolysis at several temperatures on simulated acidic mine wastewater, revealing that sludge biochar subjected to high-temperature conditions exhibited effective heavy metal adsorption. Lin et al. (2024a) prepared iron-rich sludge-based biochar FBC900 for the adsorption of tetracycline (TC) in water by using iron-rich sludge as the raw material at 900 °C. The adsorption rate of FBC900 on TC was 81.4% after five regeneration-adsorption cycles, indicating a high degree of reusability. It introduces a novel approach to the utilization of iron-rich sludge-based biochar in the remediation of wastewater antibiotics. Xie et al. (2024b) explored the adsorption performance of magnetic-containing sludge biochar on TC in wastewater, prepared magnetic-containing sludge biochar (FBC800) and common sludge biochar (BC800) as adsorbent at carbonisation temperature of 800 °C, and the adsorption mechanism of TC by F-BC800 was analyzed through a comparison with the adsorption mechanism of magnetic-containing sludge. The use of biochar for the extraction of tetracyclines from water exhibits enhanced application potential. Lin et al. (2024b) used a sludge-based biochar-loaded magnesium-iron bimetallic oxide composite material as an adsorbent to study the competitive adsorption of Cd(II) and Pb(II). The results showed that Pb and Cd have an antagonistic effect in the binary system, with Pb having a stronger inhibitory effect on Cd than Cd on Pb. The presence of Na^+, Mg^{2+} and Ca^{2+} has a certain inhibitory effect on the adsorption of Pb(II) and Cd(II) by (Mg/Fe)LDO-ASB , with Mg^{2+} and Ca^{2+} being the most effective. Ren et al. (2024) prepared sodic sludge biochar composite Aubergine material using waste sludge and Aubergine and used it for the treatment of Cu^{2+} containing wastewater, and concluded that sodic sludge biochar composite Aubergine is a promising adsorbent in the actual treatment of Cu-containing wastewater. The application of advanced oxidation for treating recalcitrant organic matter: Huang et al. (2024) reviewed the current status of sludge char modification methods, compared and analysed the advantages and disadvantages of different modification methods, as well as its activation of persulfate and antibiotic degradation mechanism, and in the future research can be used to adopt a variety of activation methods jointly and synergistically, as well as innovate the modification conditions for the sludge biochar, in order to achieve the sludge biochar on a variety of complex. The efficient degradation of complex antibiotics by sludge biochar can be achieved in the future. Li et al. (2024) prepared sludge biochar (SBC) from sewage landfill sludge at different pyrolysis temperatures, and found that SBC could efficiently activate peroxynitrite (PMS) and promote rhodamine B (RhB) degradation, revealing the potential of SBC activation of PMS in the treatment of difficult-to-degrade organic pollutants, and elucidating the main catalytic mechanism of SBC in the advanced oxidation process. Additionally, Nie et al. (2022) produced biochar by pyrolyzing wastewater treatment plant secondary sedimentation tank sludge. In order to prepare iron-containing biochar (ISBC), Liu et al. (2021) modified printing and dyeing biochemical sludge with sodium hydroxide. They then loaded Mn/Cu bimetallic on this biochar using an impregnation and calcination method. The degradation of organic pollutants using sludge-derived biochar-activated persulfate has primarily focused on pollutants in the aqueous phase, with limited research on sediment pollutants. The impact of activated persulfate on the degradation of PAHs in sediment from water-treated sludge remains ambiguous.

This study consists of the following:

- Water treatment sludge from a wastewater plant was collected and prepared into biochar using pyrolysis and used to activate persulfate for degradation of BaP in sediments.
- Experiments were conducted to analyse the efficiency of pyrolysis conditions, biochar dosing, persulfate dosing and pH on the degradation of BaP by biochar-activated persulfate.
- To analyse the mechanism of BaP degradation in sediments by biochar-activated persulfate through material characterisation and quenching tests.

2. Materials and methods

2.1. Test materials

BaP (No. GBW08701, 99% purity, purchased from the Research and Management Centre for Standard Substances, China Academy of Metrology and Science); sodium persulfate (KHSO5-0.5KHSO4 -0.5K2SO4), sodium hydroxide, hydrochloric acid, p-benzoquinone (BQ), ethanol (EtOH), furfuryl alcohol (FFA), acetone, and tertiary butyl alcohol (TBA) were purchased from the China National Pharmaceutical Group Co. Ltd. and were of superior purity.

The catalyst preparation sludge was sourced from the sludge storage facility of a domestic wastewater treatment plant in Beijing. The sludge samples were air-dried in a fume hood (Purair Basic, Shanghai Shengya International Trade Co., Ltd.) for 7 d. The sludge samples were homogenised, pulverised, passed through a 100-mesh mesh sieve, and freeze-dried for 72 h. The samples were stored for later use.

The PAH-contaminated soil used in the experiment was prepared by artificial cultivation method. Before the experiment, BaP at a concentration of 20 mg/L was dissolved in acetone and then mixed with the soil. The soil was incubated under dark conditions for 14 d. Once the acetone had fully evaporated, the soil was available for additional research.

2.2. Material preparation

Dewatered sludge from a domestic wastewater treatment facility was used as a precursor for the synthesis of biochar material. The freeze-dried sludge was subjected to heating in a tube furnace at temperatures of 400, 600, and 800 °C, at a regulated rate of 10 °C/min, and this condition was sustained for 1–3 h. The biochar samples obtained were designated as SBC-X/T, with X representing the pyrolysis temperature and T indicating the pyrolysis time. The preparation process is shown in Figure 1.

Figure 1. Material preparation process

2.3. PAH degradation

As illustrated in Figure 2, a 50 mL conical flask was filled with 5 g of soil polluted with PAHs. The pH was subsequently adjusted using 0.1 M NaOH or HCL, followed by the addition of a specified quantity of biochar catalyst and 20 mL of a predetermined concentration of sodium persulfate solution. The reaction was conducted in a water-bath shaking apparatus in a light-avoiding environment for 3 h at a constant temperature of 25 °C. Another 5 g of PAH polluted soil was placed in a water bath as a control group. Upon completion of the reaction, the combined solution was subjected to centrifugation, and the supernatant was removed, followed by drying of the centrifuged soil using anhydrous sodium sulfate (w/w=1:1). Subsequently, Ultrasonic extraction was performed by introducing a dichloromethane/hexane combination to the desiccated soil samples. The extract was concentrated by nitrogen blowing, purified by magnesium silicate, purified by column chromatography and filtered through 0.22 μm PTEE, and then the target compounds were determined by gas chromatography-mass spectrometry.

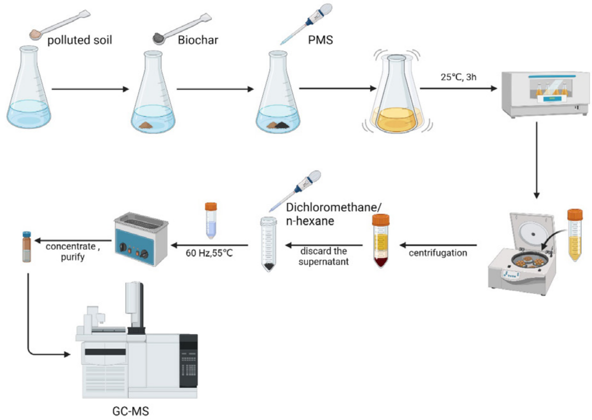

Figure 2. Main process of degradation experiment

2.4. Test analyses

XRD analysis was tested using an X-ray diffractometer (FRINGE model, Suzhou Wave Sound Scientific Instrument Co., Ltd.), with Cu-Ka as the radiation source; FTIR analysis was tested using a Fourier transform near-infrared spectrometer (FT 9700 model, Perten); and UV spectra were tested using an integrating sphere (diffuse reflectance mode) spectrophotometer (U-3900 model, Hitachi, Japan). The test was carried out; the PAHs content in the liquid phase was determined using a gas chromatography-mass spectrometer (MS3100 model, Beijing East-West Analytical Instruments Co., Ltd.), the temperatures of ion source, transmission line, and the injector were set at 230, 280 and 300 °C, respectively. A capillary column (HP-5MS model, 30×0.25×0.25 mm, Agilent Technologies Co., Ltd.) was used to separate the PAHs for the analysis. The PAHs were separated on a capillary column (HP-5MS model, 30×0.25×0.25 mm, Agilent Technologies Ltd.) with a carrier gas of helium at a flow rate of 1 mL/min. The column temperature was initially set at 40 °C for a duration of 1 minute. Subsequently, it was raised to 120 °C at a ramp rate of 25 °C/min, then to 160 °C at a ramp rate of 10 °C/min, and ultimately to 300 °C at a ramp rate of 5 °C/min.

3. Results and discussion

3.1. Effect of pyrolysis conditions on the efficiency of biochar-catalysed persulfate degradation of BaP

The synthesis of biochar and the extraction of organic pollutants from sediments are primarily influenced by the temperature and length of the pyrolysis process. This section maintains constant circumstances of PMS dosing at

0.2 mM, BC dosing at 0.5 g/L, and a reaction solution pH of 7, while analyzing and discussing the impact of pyrolysis temperature on the breakdown of BaP in sediments using the BC/PMS system. From Figure 3a, it can be found that with the increase of pyrolysis temperature, the BC/PMS system's BaP degradation rate gradually increased. When the pyrolysis temperatures were 400, 600 and 800 °C, the removal rates of BaP after 180 min of reaction could reach 14.20%, 86.58% and 90.97%, respectively. The increased release of volatiles may be attributed to the higher pyrolysis temperature of the sludge. The sludge acquired a more porous configuration, resulting in a substantial increase in the specific surface area of the activated carbon, which consequently improved the stability of C- and O- functional groups. The ideal pyrolysis temperature was determined to be 800 °C.

Figure 3b illustrates that the rate of BaP deterioration by the BC/PMS system exhibited an initial increase followed by a decline as pyrolysis time extended, with BC-800/2 h achieving the maximum BaP removal rate. Prolonged exposure to excessively high pyrolysis temperatures compromises the interior porous topology of biochar, causing a decrease in the specific surface area. O- and N-functional groups decrease as a consequence of the breakdown of the structure and surface functional groups, hence impacting the activation of PMS. Through the above analysis, we chose the pyrolysis time of 2 h, and all the subsequent ones used BC-800/2 h as the system catalyst.

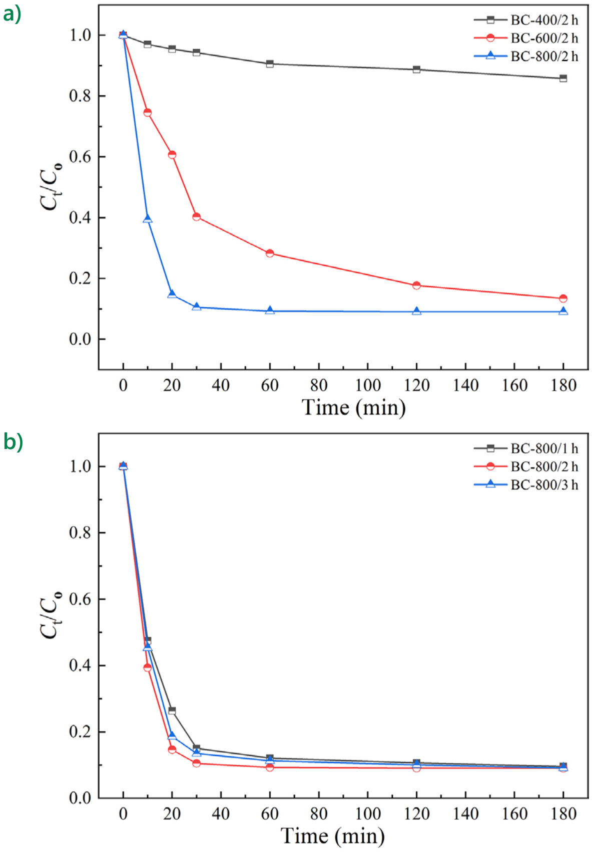

Figure 3. Pyrolysis condition: a) temperature; b) time

3.2. Factors affecting the efficiency of biochar-catalysed persulfate degradation of BaP

3.2.1. Catalyst dosage

To find out how varying catalyst doses affect the BC-800/2 h activated PMS system's ability to efficiently degrade BPA, the conditions of PMS dosage of 0.2 mM and pH value of reaction solution of 7 were controlled unchanged, and the changes of BaP removal rate were observed when the dosage of BC-800/2 h was 0.25, 0.50, 0.75, and 1.00 g/L. From Figure 4, with the increase of BC-800/2 h catalyst dosage, the BaP removal rate showed a gradual increase, and when the catalyst dosage reached 1.00 g/L, the BaP removal rate could reach 99.81% after 180 min of reaction. The increased dosage of the catalyst likely results in increasing PMS activation and adsorption sites, thereby facilitating the formation of free radicals that enhance the degradation of BaP (Liu et al., 2021). The optimum catalyst dosage for this system was 1.00 g/L.

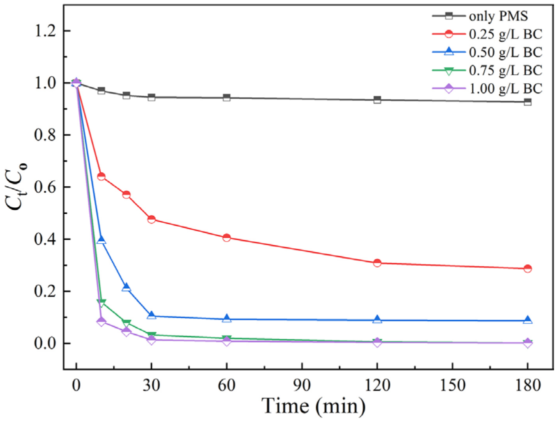

Figure 4. Catalyst dosage

3.2.2. PMS dosing rate

The dose of PMS has a significant impact on the catalytic reaction rate. Using a reaction solution pH of 7 and 0.5 g/L catalyst dosage, the researchers examined the effects of various persulfate dosages on the effectiveness of

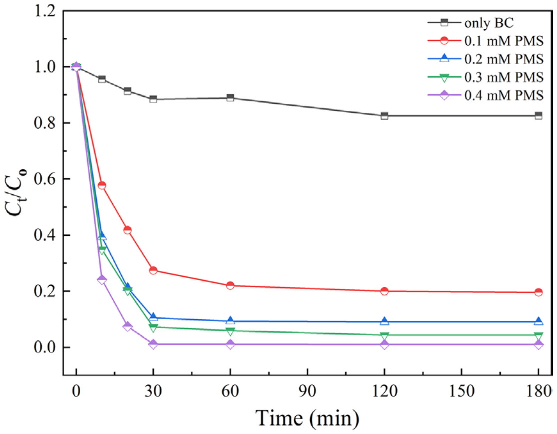

Figure 5. Persulfate dosage

the BC-800/2 h activated PMS system for BPA degradation. The results are displayed in Figure 5 and indicate the changes in the BaP removal rate that occurred at PMS dosages of 0.1, 0.2, 0.3, and 0.4 mM, respectively. In Figure 5, the BaP removal rate showed a gradual increase with higher PMS concentration in the concentration range of 0.1 to 0.4 mM. The increased concentration of PMS likely activates a greater amount of PMS, resulting in the generation of additional free radicals, including •OH, $\bullet SO_4^-$, and $\bullet O_2^-$, which contribute to the oxidative degradation of BPA (Zhou et al., 2018). Therefore the optimum PMS dosage for the system was 0.4 mM.

3.2.3. pH of the system

Prior research demonstrates that the pH significantly affects PMS activation. The results of the BaP removal rate changes at pH values of 3, 5, 7, 9, and 11 are depicted in Figure 6, which were obtained by controlling the catalyst dosing to 0.5 g/L and PMS dosing to 0.2 mM. At an initial pH of 3, the reaction showed the maximum rate of BaP degradation, as evidenced by Figure 6a. The rate of BaP degradation decreased (pH = 5). Also, the degradation rate of BaP was more comparable when the pH was 7–11; however, it was still greater than 89% after 180 minutes of reaction. As shown in Figure 6b, the surface of BC-800/2 h carried a negative charge when the solution pH exceeded 4.35, which corresponds to its zero-point charge at pH 4.35. The acidity coefficients (pKa) of BaP were in the range of 9.6~10.2. When the solution pH < 9.6, BaP exists in the form of molecules or monoanions, and when the solution pH > 10.2, BaP in the solution mainly exists in the form of double anions. At pH 3, the van der Waals forces enabled BC-800/2 h to exhibit stronger adsorption of BaP. However, at pH 11, the surface of BC-800/2 h became negatively charged, while BaP existed in the form of a dianion. This resulted in Coulombic repulsion between the catalyst and BaP, hindering their interaction and ultimately leading to a reduced BaP removal efficiency.

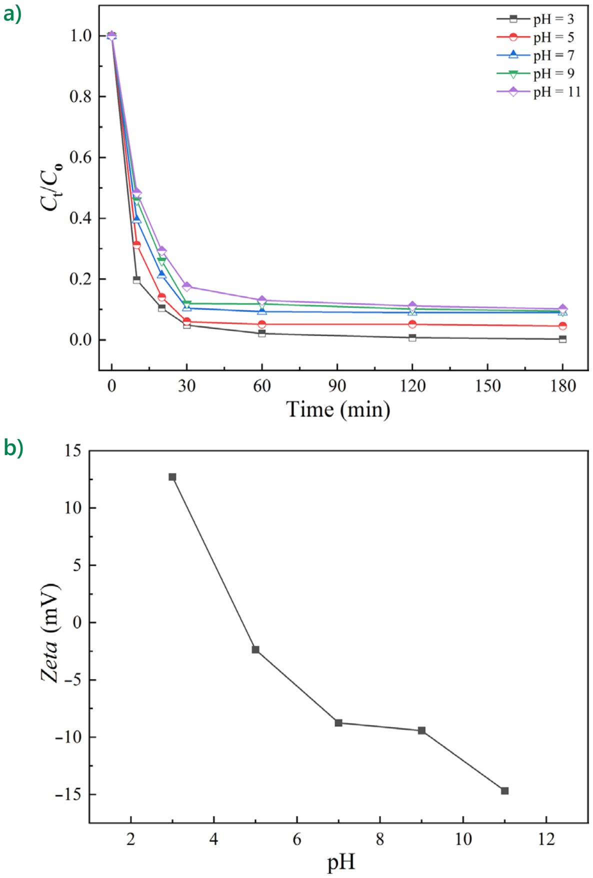

Figure 6. Effect of pH: a) pH; b) zero-point charge test of BC-800/2 h

3.3. Analysis of degradation mechanisms

3.3.1. Characterisation of sludge activated carbon materials

Figure 7a exhibits the outcomes of the FTIR analysis of sediment activated carbon that was prepared at various pyrolysis temperatures. Through FTIR analysis, we identified that the absorption peaks at 481 cm^{-1} and 578 cm^{-1} corresponded to Fe-O stretching vibrations, while the peaks at 1535 cm^{-1} were attributed to the interaction between carboxylic acid groups and Fe_3O_4 particles. Additionally, the peaks at 643 cm^{-1} and 522 cm^{-1} originated from the stretching vibration of the Mn-O bond. The Mn-O-Mn bond stretching was responsible for the absorption peaks at 1402 cm^{-1} and 797 cm^{-1}, indicating the presence of iron and manganese oxides on the BC surface. Secondly, the absorption peaks at 1036, 1619, and 1558 cm^{-1} are attributed to the stretching vibrations of the C-O-C, C=C, and C=O bonds in the aromatic ring, respectively. The strong absorption peak at 1640 cm^{-1} is derived from the bending vibration of the hydroxyl group. These O-containing functional groups on the surface are regarded as the main electron donors and active sites responsible for the adsorption and degradation of organic pollutants within the material. The sludge was analyzed by XRD at various pyrolysis temperatures. The results of XRD analysis of activated carbon are shown in Figure 7b. From the XRD analysis results, it can be seen that the diffraction peaks with peaks located at 30.1, 35.5, 43.2, 57.0 and 62.7 in the XRD characterization diagram belong to the (220), (311), (400), (511) and (440) crystal planes of Fe_3O_4, while the diffraction peaks of 28.9, 32.3, 36.1, 50.7 and 59.8 diffraction peaks belong to (112), (103), (211), (105) and (224) crystal planes of MnO_2, respectively. In contrast, the (311) and (112) crystal surfaces of Fe_3O_4 and MnO_2, respectively, exhibited a substantial increase in response to an increase in pyrolysis temperature. This phenomenon may be attributed to the material's enhanced structural stability and crystallinity. Additionally, the derived peak at 43.9 is attributed to the (100) crystal face of amorphous graphite, suggesting the presence of aryl rings with increased polarity on the surface of BC. This results in enhanced electron donor and acceptor interactions between BC and aromatic compounds. Additionally, as illustrated in Figure 7c, the UV spectral analysis of the sludge activated carbon prepared at various pyrolysis temperatures was conducted.

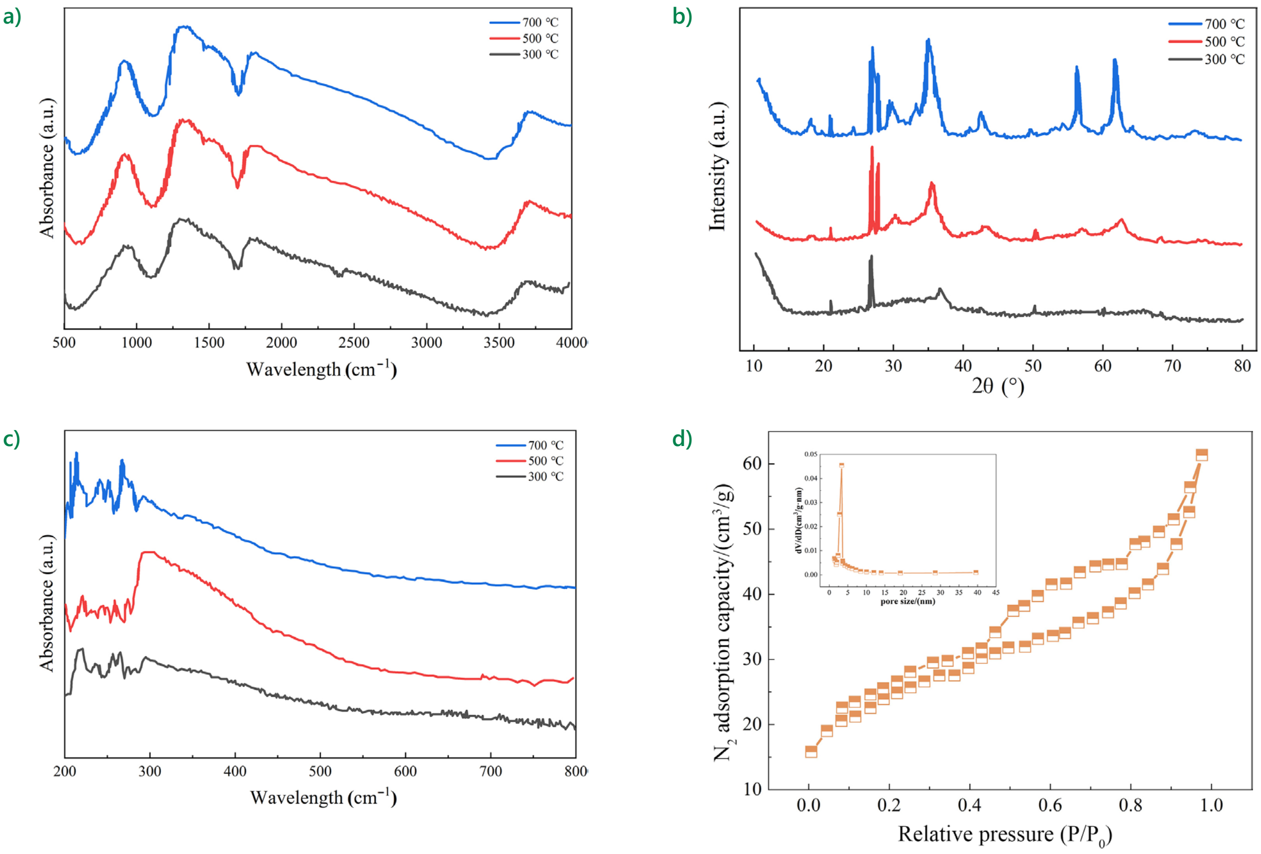

Figure 7. Characterisation of sludge activated carbon materials: a) FTIR; b) XRD; c) UV; d) N_2 adsorption-desorption isotherm and pore size distribution of SBC-800 °C/2 h

The results showed the presence of the electron transfer effect of manganese oxychloride, as indicated by the very high absorption peaks at 260 and 310 nm. In addition, considering that the specific surface area and pore size of the material are critical for carrying active sites, the N_2 adsorption–desorption isotherm and pore size distribution of SBC-800 °C/2 h were studied, and the results are shown in Figure 7d. The material shows an N_2 adsorption–desorption isotherm of type IV, indicating the presence of a mesoporous structure, which helps to reduce mass transfer resistance and accelerate the transport of pollutants to the surface of the material. The pore size distribution diagram further shows that the sample is rich in porous structures. Data analysis shows that the specific surface area, total void volume and average pore size of this material are 84.22 m^2/g, 0.095 cm^3/g and 4.53 mm, respectively.

3.3.2. Quenching test

In an activated persulfate (PS) system, organic pollutants are generally degraded via two main pathways: the free radical pathway, primarily driven by $\bullet SO_4^-$, •OH, and $\bullet O_2$, and the non-free radical pathway, which is mainly governed by $^1O_2$. Figure 8 displays the outcomes of this subsection's usage of EtOH, TBA, BQ, and FFA as quenchers for the identification of reactive species. The addition of quenchers significantly inhibited the degradation efficiency of BaP, and the removal of BaP decreased to 32.99%, 41.67%, 70.01%, and 58.91% after EtOH, TBA, BQ, and FFA treatments. EtOH showed the most significant inhibitory results for the reaction system, indicating that •OH dominated the reaction system. Whereas, the low effect of BQ on BaP indicated that $\bullet O_2$ was not the main radical involved in the reaction. And after FFA treatment, the BaP removal decreased from 99.03% to 58.91%, signifying the non-radical pathway of $^1O_2$ was also involved in the degradation of BaP. The results indicated that three active species such as $\bullet SO_4^-$, •OH and $^1O_2$ mediated both the radical and non-radical degradation pathways of BaP.

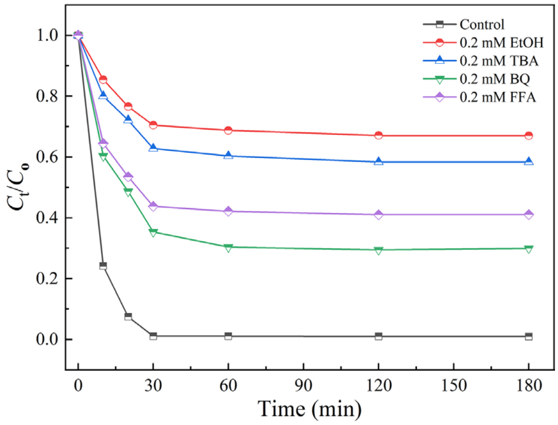

Figure 8. Analysis quenching test

4. Conclusions and outlook

In order to develop a green and environmentally friendly catalyst for the persulfate system, this study used sewage treatment sludge containing iron and manganese as a raw material to prepare activated carbon containing Fe and Mn by pyrolysis, and used it to activate the persulfate system to degrade BaP in sediments.

- Biochar with catalytic properties was prepared by pyrolysis of iron-manganese sludge from a sewage treatment plant. Different pyrolysis temperatures and times were used in the experiment, and the optimal preparation conditions were determined by comparison: 800 °C for 2 h.
- The processed sludge biochar was used to activate persulfate for the degradation of benzo[a]pyrene (BaP) in sediments. The experiment examined the impact of variables like catalyst quantity, persulfate concentration, and reaction pH on degradation efficiency. The findings indicated that, under ideal circumstances, the BaP removal rate was 99.85%.
- The research, using material characterization and quenching tests, identified active species including $\bullet SO_4^-$, •OH, and $^1O_2$ in this environment, which together facilitated the degradation of BaP via both radical and non-radical mechanisms. In addition, the presence of iron and manganese oxides and functional groups on the material surface significantly enhanced the catalytic activity.

This study successfully prepared a biochar catalyst that can efficiently activate PMS to degrade BaP using sewage treatment sludge containing iron and manganese as raw materials. This provides a new method for controlling polycyclic aromatic hydrocarbon pollution in sediments and developing catalysts for the persulfate system.

At the same time, there are also shortcomings in our experiment: First, the experiment was carried out under laboratory conditions, and environmental variables such as temperature, humidity, and actual pollutant concentrations may be different in the real environment, which leads to the need for further verification of the practical applicability of the experimental results. Second, there is insufficient research on the long-term application effects. Although the system has shown high degradation efficiency in a short period of time, the stability and regeneration ability of the catalyst and its potential impact on the environment have not yet been explored. Finally, there is a lack of research on complex pollutants. This study focused on the degradation of BaP, but pollutants in the real environment are often a mixture of various organic compounds. In the future, research should be conducted on the performance of this catalytic system when multiple organic pollutants are present at the same time. Based on the above three considerations, we believe that future research can be further extended to experiments in real environments to verify the performance of the catalyst under complex environmental conditions and optimize the operating conditions for practical applications. In order to improve economic and environmental sustainability, future research could investigate the regeneration and reuse of the catalyst to reduce costs and minimise waste. Future research could explore the synergistic removal of different PAHs and other organic pollutants by the activated sludge biochar persulfate system, thereby expanding its scope of application and improving its practical treatment capabilities.

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