



Non-metallic functionalized biochar obtained from cornstalk biowaste for rapid organics removal via peroxymonosulfate activation

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Abstract

The degradation of anionic organic dyes, specifically Eriochrome Black T (EBT) and Methyl Orange (MO), presents a significant challenge in wastewater treatment due to their persistence and potential environmental hazards. This research investigates the degradation of EBT and MO via peroxymonosulfate (PMS) activation using a biochar catalyst. Specifically, pristine, O-doped, and B-doped corn stalk biochars were investigated for their effectiveness in promoting dye degradation. Characterization techniques, including SEM, FTIR, and XRD, were employed. The effects of operational parameters, including catalyst dosage, initial pollutant concentration, PMS concentration, inorganic ions, and a binary dye mixture, on degradation efficiency were studied. The experimental results demonstrated that B-doped biochar exhibits superior catalytic performance, degrading 95% and 89% of MO and EBT, respectively, in 60 min, compared to O-doped biochar, which degrades 89% and 75% of MO and EBT, respectively, in 90 min. Meanwhile, the radical-scavenging experiment showed that the radical pathway was the dominant reaction pathway. Furthermore, the catalyst's reusability was assessed over three cycles, with effective regeneration demonstrated after each cycle. The findings from this research not only underscore the pivotal role of O- and B-doped biochar catalysts in advanced oxidation processes for water purification but also the imperative of sustainable and efficient approaches to environmental remediation.

Keywords: Heteroatom doping; Biochar; Peroxymonosulfate; Methyl orange; Eriochrome black T

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Introduction

The importance of water for the sustainability of life cannot be overstated, with applications ranging from domestic to industrial purposes. However, with rapid population growth, economic development, and urbanization, the frequent occurrences of organic pollutants from various sources including pharmaceuticals (Yang *et al.* 2025), pesticides (Xie *et al.* 2025) and personal care products (Liu *et al.* 2025) in surface water environment have raised an alarming attention over their potential impacts on the aquatic lives and even the human health (Abalasei *et al.* 2025). Among pollutants, dyes are the most widely used across industries such as textiles, plastics,

paper, printing, and pharmaceuticals (Prajapati *et al.* 2026). Similarly, Eriochrome black T (EBT) and methyl orange (MO) are among the most problematic azo dyes in wastewater (Aslam *et al.* 2025). Therefore, treating such water is of paramount importance, given the high demand for clean water driven by the rapid global population growth.

Various physical, chemical, and biological approaches have been employed to diminish environmental pollutants (Rajendran *et al.* 2025). Nevertheless, these techniques are generally limited either economically or technically. In this regard, advanced oxidation processes (AOPs) have proven to be an

effective and cost-effective alternative for the degradation of pollutants (Alrefaey *et al.* 2025).

Peroxymonosulfate (PMS) is an oxidant that has shown promising results in dye degradation due to its strong oxidation potential (Sun *et al.* 2025). Activation of PMS can be achieved by external energy (UV, ultrasound, heat, etc.) or by a catalyst, thereby generating reactive species for dye degradation (Song *et al.* 2022). Catalytic PMS activation is a more viable solution than external energy activation, which requires a constant energy supply to operate effectively. Different carbon allotropes, such as graphene, carbon nanotubes, ordered mesoporous carbon, and nano-diamonds, have attracted widespread interest for activating PMS (Wang *et al.* 2018). However, these carbon allotropes are expensive, and, without modification, they are generally unreactive due to their highly graphitic structure.

Biochar (BC) derived from metal-free carbon materials has been employed as a PMS activator to replace metal-based materials, owing to the undesirable metal leaching that occurs during application (Tan *et al.* 2025). Among several agro-wastes, cornstalks, with an advanced carbonaceous structure, are ideal materials for synthesising biochar. Furthermore, the incorporation of O/B dopants into biochar has shown potential for PMS activation, leading to the destruction of electron-rich hazardous pollutants via ROS generation and subsequently improving dye degradation efficiency (Liu *et al.* 2020; Yi *et al.* 2025).

In this study, O/B-doped BC was prepared via a facile one-pot synthesis for the degradation of anionic azo dyes (EBT and MO). The incorporation of the heteroatom is anticipated to create a more efficient catalyst for the degradation of these dyes.

Materials and Methods

Chemicals

All chemicals and reagents procured were of analytical grade and used as received. Oxone[®] as a source of peroxymonosulfate (HSO_5^-) was obtained from Merck. Methyl orange ($C_{14}H_{14}N_3NaO_3S$) was supplied by QReC. Eriochrome black T ($C_{20}H_{12}N_3O_7SNa$) was supplied by Merck. Oxalic acid dihydrate ($C_2H_2O_4 \cdot 2H_2O$), boric acid (H_3BO_3), methanol (CH_3OH), nitrobenzene ($C_6H_5NO_2$), and sodium perchlorate ($NaClO_4$) were purchased from Sigma-Aldrich. Sodium chloride ($NaCl$) was obtained from HMBG Chemicals. Sodium bicarbonate ($NaHCO_3$) was purchased from ACS Chemicals. Potassium dihydrogen phosphate (KH_2PO_4) was supplied by Loba Chemie.

Sample Collection and Pre-treatment

The cornstalk (CS) biomass (Figure 1) was obtained locally from Jogana (12.0233°N, 8.7011°E), Kano State. The collected sample was washed several times with deionized (DI) water and dried for a couple of days until it reached a constant weight. The dried sample was ground, sieved, and washed several times with deionized water. It was then dried in an oven at 60 °C for 2 days before use.



Figure 1. Cornstalk.

Synthesis of Pristine BC

The CS powder was transferred to a crucible sealed tightly with several layers of aluminum

foil to provide an oxygen-deficient atmosphere. The sample was placed in a Carbolite muffle furnace and pyrolyzed at 600

°C with a ramping rate of 10 °C/min for 30 min to produce pristine BC (Figure 2a). The synthesis follows the method reported by Hung *et al.* (2022).

Synthesis of Modified BC

The modified biochar was prepared via a facile one-pot synthesis, incorporating CS biomass with oxalic acid dihydrate and boric acid as precursors. During the process, the mass ratio of the biomass to oxalic acid dihydrate (oxygen precursor) and boric acid (boron precursor) was 1:1. Typically, 20 g of boric acid was mixed with 20 g of CS biomass for B-BC. Similarly, 20 g of oxalic acid was incorporated into 20 g of cornstalk biomass to form O-BC. The samples were sonicated with

a Branson Sonicator to ensure thorough mixing and homogenization. Afterwards, the resulting mixture was transferred to an enclosed crucible, sealed tightly with several layers of aluminium foil to provide an oxygen-deficient atmosphere, and then pyrolysed in a muffle furnace (Carbolite Gero Ltd.) at 600 °C with a ramp rate of 10 °C/min for 30 min. Subsequently, the crucible was removed from the furnace and allowed to cool to room temperature. The products obtained from pyrolysis were designated O-BC (Figure 2b) and B-BC (Figure 2c). The modified BC obtained was washed several times with deionized water and dried in an oven at 60 °C before use.



Figure 2. Image of (a) pristine BC, (b) O-BC, and (c) B-BC.

Characterization of Catalyst

The Pristine BC, O-BC, and B-BC were characterized using X-ray Diffraction (Rigaku Miniflex 600 X-ray), Scanning Electron Microscopy (Hitachi S-4800 SEM), and Fourier Transform Infrared Spectroscopy (FTIR spectrometer (Thermo Scientific Nicolet)).

Catalytic Performance Study

To evaluate the catalysts' performance (pristine BC, O-BC, B-BC) in degrading MO and EBT via PMS activation, a batch experiment was conducted. Before the catalytic reaction, 50 mg of the catalyst was added to a 100 mL solution containing the dye solution (20 mg/l) at the inherent solution pH. The mixture was stirred using a magnetic stirrer for 20 minutes to establish the adsorption-desorption equilibrium. Afterward, (100 mg) PMS was added to the solution to initiate the reaction. At selected time intervals (0, 5, 10, 15, 30, 45, 60, 75, 90 min), 2 mL of the samples were withdrawn using a 0.22 µm Teflon syringe and filtered using a cellulose acetate filter. The filtrate was

quenched with 0.1 mL of methanol, which effectively terminated the reaction by preventing further oxidation of MO and EBT. The dye concentration was subsequently determined using a UV-visible spectrophotometer at wavelengths of 465 and 560 nm for MO and EBT, respectively. The effects of experimental conditions, including catalyst dosage, initial pollutant concentration, PMS concentration, and inorganic ions, were also examined. The percentage removal was calculated using equation (1) below.

$$\% \text{Removal} = \frac{(C_o - C_t)}{C_o} \times 100\% \quad (1)$$

Where C_o is the initial concentration of the dye and C_t is the final concentration of the dye at time t (min), respectively.

Results and Discussion

Characterizations

Scanning Electron Microscope (SEM)

The catalyst's surface morphology was observed using SEM (Figure 3). The pristine BC surface was observed to be relatively smooth. Upon doping with O & B, a

hierarchical porous structure was significantly observed. The introduction of these heteroatoms disrupts the carbon matrix,

leading to increased surface roughness, as observed predominantly in B-BC.

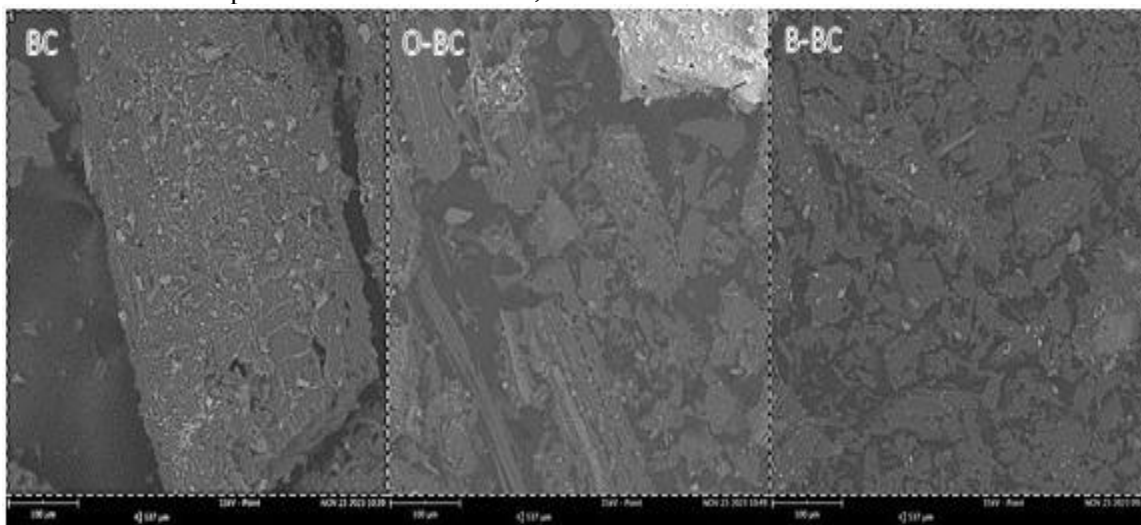


Figure 3. SEM images of pristine BC, O-BC, and B-BC.

Fourier Transform Infra-red (FTIR) Spectroscopy

The FTIR spectra (Figure 4) of the Pristine and modified biochar were analyzed in the 600-4000 cm^{-1} region to investigate the functional groups present. In the FTIR spectrum of the pristine BC, a small band at around 665 cm^{-1} is associated with the presence of the C-H aromatic group (Fang *et al.* 2014). The broad band in the region around 1033 & 1316 cm^{-1} was ascribed to the presence of aliphatic ethers (C-O-C) and alcohols (C-O) stretching or phenol groups (Thang *et al.* 2019). The band at 1596 cm^{-1} was indicative of a C=O stretching vibration associated with an oxygen-containing functional group present in Pristine BC. The bands at around 2113 and 2348 cm^{-1} are due to stretching of long-chain aliphatic symmetrical $-\text{CH}_2$ & asymmetrical $-\text{CH}_3$ groups. However, the peak is not obvious; a possible reason is that BC is prepared by pyrolysis, and some low-boiling-point components are removed during the process (Zhang *et al.*, 2022). In the FTIR spectrum of O-doped BC and B-doped BC, a more intense peak appeared around 1000-1600 cm^{-1} and

3100 cm^{-1} , and this could be because of the incorporation of the heteroatoms in the carbon framework (Gao *et al.* 2022). In addition, there is a presence of more intense peaks at around 1037 cm^{-1} , 1316 cm^{-1} , 1566 cm^{-1} , and 1897 cm^{-1} , which is a result of stretching vibrations of oxygen-containing functional groups (C-O and C=O) present on the surface of the biochar (Thang *et al.* 2019). The effect of incorporating O atoms into BC is evident in the shift of the O-H peak from 3400 cm^{-1} to 3300 cm^{-1} . Compared with pristine BC and O-BC, the FTIR spectrum of B-BC showed a new, visible, and intense peak at around 1194 cm^{-1} , indicating that the number of B-O, B-C & C-O bonds is significantly enhanced by B doping (Huang *et al.* 2018). This enhancement can be attributed to the formation of new chemical bonds between boron and oxygen (B-O bonds) and boron and carbon (B-C bonds), as well as to the interaction of boron with pre-existing carbon-oxygen (C-O) bonds (Gao *et al.* 2022). The FTIR spectra of B-BC show a shift in peaks for OH located at 3193 cm^{-1} , indicating that O-H stretching occurs due to the formation of B-OH species.

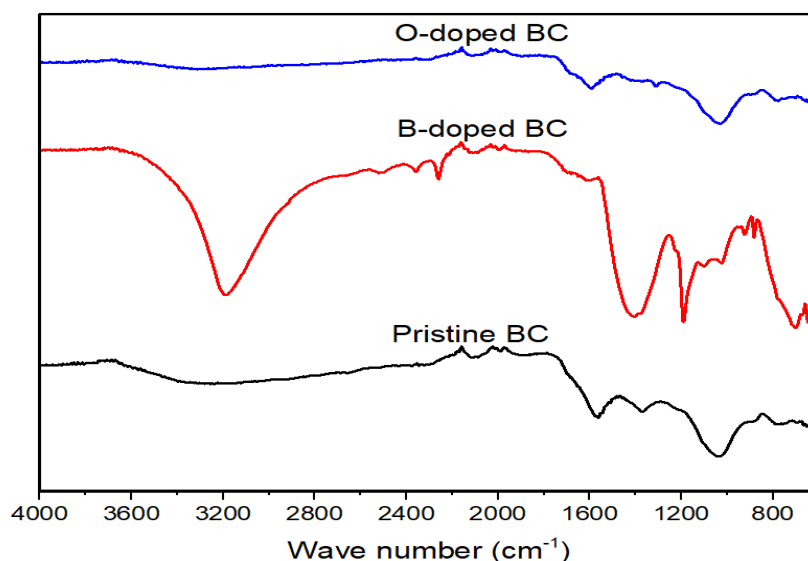


Figure 4. FTIR spectra of pristine BC, O-BC, and B-BC.

X-ray Diffraction Pattern (XRD)

Figure 5 below shows the XRD patterns of pristine BC, boron-doped (B-BC), and oxygen-doped (O-BC). In the case of pristine BC, no peaks were detected within the 2θ range. The absence of the (100) plane, which is characteristic of the crystalline carbon polymorph, indicates that BC consists solely of amorphous carbon. This is consistent with the typical amorphous nature of biochar, characterised by a disordered arrangement of carbon atoms resulting from the pyrolysis of biomass feedstock (Z. Hu and Wei 2023). Conversely, in the case of oxygen- and boron-doped biochar, the XRD pattern showed a

sharp diffraction peak at $2\theta = 14.56^\circ$, corresponding to the (001) plane. The presence of peaks at 27.92° and 27.90° is due to the (002) plane of graphitic materials. The B-BC and O-BC catalysts also exhibit peaks at 40.12° and 39.91° , respectively, which are associated with (100) planes of crystalline carbon (Y. Zhang *et al.* 2018). This indicates that the introduction of boron and oxygen dopants marks a departure from the amorphous nature of pristine biochar, suggesting that incorporating crystalline phases or an ordered structure into the biochar changes its crystallinity, which could improve its catalytic performance.

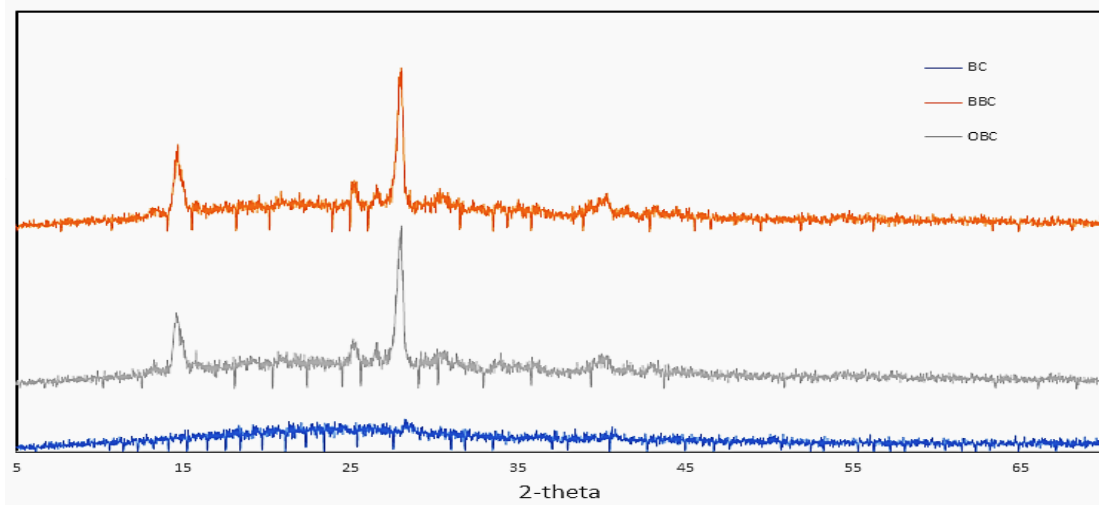


Figure 5. X-ray diffraction pattern of pristine BC, O-BC, and B-BC.

Catalytic Performance Study

Control Study

A control study on the effect of the PMS-only system, pristine biochar (BC), oxygen-doped biochar (O-BC), and boron-doped biochar (B-BC) on MO (Figure 6(a)) and EBT (Figure 6(b)) degradation was performed using a 0.05 g/l catalyst dosage, 100 mg PMS concentration, 20 mg/L MO and EBT concentration under ambient temperature. From the graphs, during MO degradation, the PMS-only system could degrade approximately 9% of MO and EBT within 90 min. indicating that the oxidation capacity of PMS alone was negligible for both pollutant degradation. However, In the control study where PMS was used, a tremendous increase in the removal efficiency of the Pristine BC,

O-doped BC, B-doped BC (49%, 77%, 89%) during MO degradation and (43%, 65% and 75%) during EBT degradation was observed within 45 min after which there was no significant change, indicating that the biochar effectively activates PMS facilitating more efficient pollutant degradation until the most available active sites on the biochar have been occupied. The maximum catalytic efficiency has been achieved. The results also showed that the percentage removal of EBT was lower than that of MO degradation. This decrease could be attributed to their complex nature, as studies have revealed that low-molecular-weight, simple-structure azo dyes tend to degrade faster than high-molecular-weight azo dyes (Chaieb, 2016).

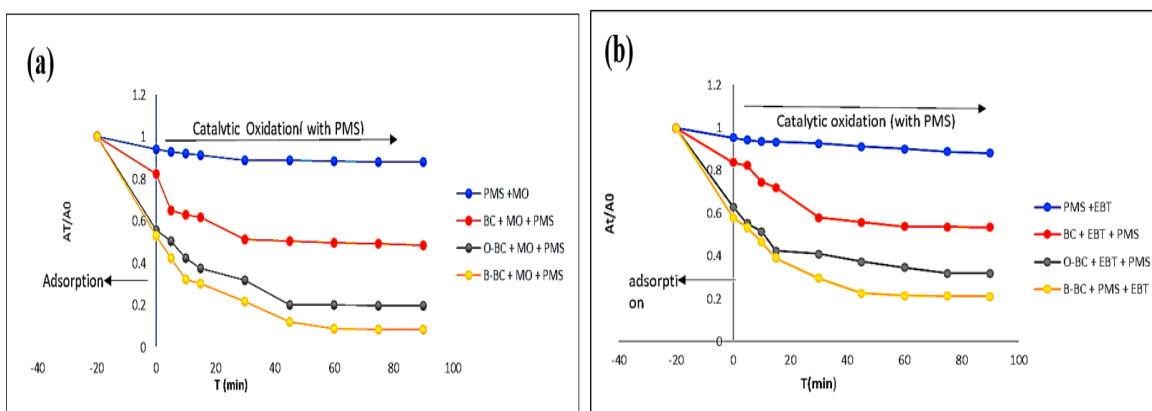


Figure 6. Performance evaluation of PMS on degradation of (a) MO and (b) EBT via activation using BC, O-BC, and B-BC.

Moreover, the trend in catalytic performance follows the order: B-doped BC > O-doped BC > Pristine BC, because O- and B-doped bio-based carbon materials effectively improve the electronic structure of the carbon framework by creating unique vacancies that activate PMS (S. Wang and Wang 2022). Moreover, the increase in the catalytic performance of B-doped biochar is due to its greater ability to disrupt the conjugated sp²-bonded carbon network, thereby increasing its surface affinity for the environmental sulfate radical advanced oxidation process (Liu *et al.* 2020).

Effect of Catalyst Dosage

The effect of catalyst dosage (0.1, 0.2, 0.3, and 0.4 g) was studied using O-BC and B-BC, and the results for MO are shown in Figure 7, and

for EBT are shown in Figure 8. According to the results obtained, slow degradation rate from (0-15) min was observed for both pollutant with catalyst dosage of (0.1 & 0.2) g, subsequently the degradation rate increased slowly over time, which is an indication that at lower catalyst dosage the rate of PMS activation and radical generation might be insufficient to effectively degrade the target pollutants (MO and EBT), thereby leading to a low degradation rate. However, upon increasing the catalyst dosage to 0.3g a significant increase in removal efficiency initially from (0-15) min was observed, and the degradation efficiency continued to increase rapidly between (30-45) min, with B-doped BC having the greatest removal efficiency of about 95% & 89% within

(90min) during MO and EBT degradation, respectively. This increase in catalytic efficiency, as observed, indicates that a higher catalyst dosage can enhance catalyst dispersion in solution, thereby improving mass transfer. Subsequently (30-45 min), some intermediates formed during the slow degradation stage may be more stable or more recalcitrant to oxidation. However, during this stage, the intermediates may undergo further transformation (breakdown), facilitating faster

degradation and reducing their levels. After an hour, the degradation rate becomes static. The stabilization stage occurs when most readily degradable dye molecules have been consumed, leaving only a few recalcitrant molecules. These residual dye molecules may be strongly adsorbed onto the biochar surface, thereby reducing their accessibility to the remaining active sites (Zhang *et al.* 2018).

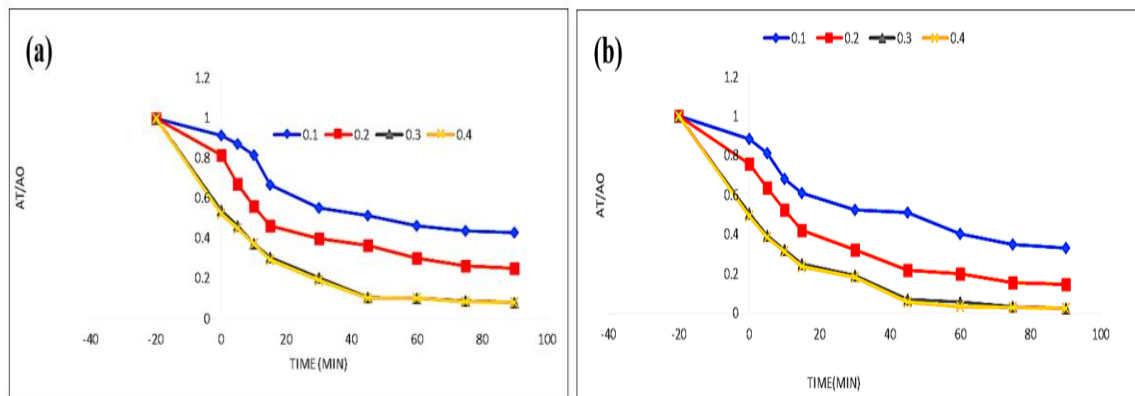


Figure 7. Effect of (a) O-doped and (b) B-doped biochar dosage on MO degradation.

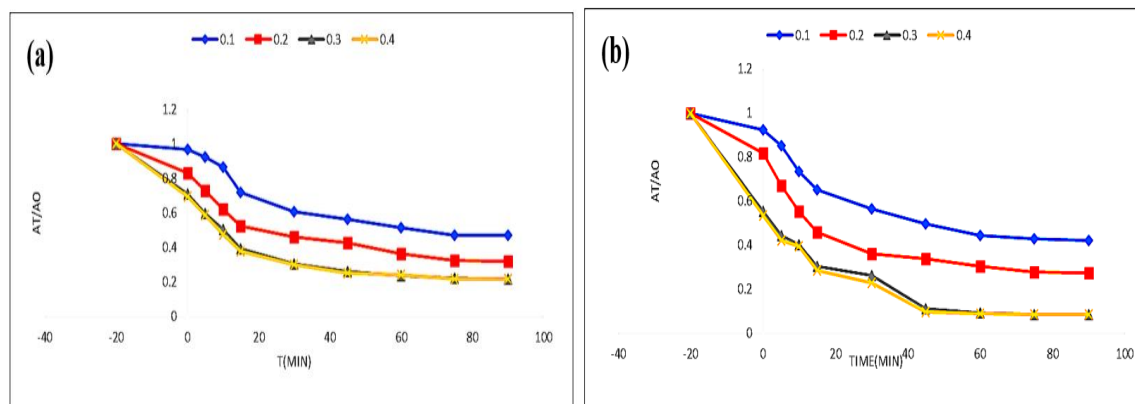


Figure 8. Effect of (a) O-doped and (b) B-doped biochar dosage on EBT degradation.

Effect of Pollutant Concentration

To explore the effect of pollutant(s) concentration on degradation performance, experiments were conducted at 30, 40, 50 & 70 mg/L. The results for MO are shown in Figure 9, and for EBT in Figure 10. As shown in the figures, pollutant degradation efficiencies varied with pollutant concentration. During MO degradation, when the initial concentration of MO was 30 mg/l, about 80% and 85% (using O-BC and B-BC, respectively) were degraded within 90 minutes. Furthermore, it could be observed

that the removal efficiency of MO with concentrations of 40, 50 & 70 mg/l reduced to 73%, 65%, and 55%, respectively, using O-BC, and 80%, 74%, and 65% using B-BC. A similar trend was observed during EBT degradation, with the initial concentration at 30 mg/l: 70% and 79% (using O-BC and B-BC, respectively) were degraded within 90 minutes. While removal efficiency for O-BC reduced to 65%, 59%, and 50%, and B-BC reduced to 70%, 64%, 58%, with an increase in the concentration of EBT to (40, 50 & 70) mg/l, respectively. These results show that at

low concentrations of pollutants, more $SO_4^{\bullet-}$ radicals were generated due to less competition between PMS and the pollutant at the biochar surface, resulting in high pollutant removal efficiencies (Hung *et al.* 2022). The decrease in degradation efficiency observed in

the results should be due to the high concentration of pollutants, which may occupy the catalytically active sites of the biochar, thereby inhibiting the activation of PMS to produce sulfate radicals by BC (Huong *et al.* 2020).

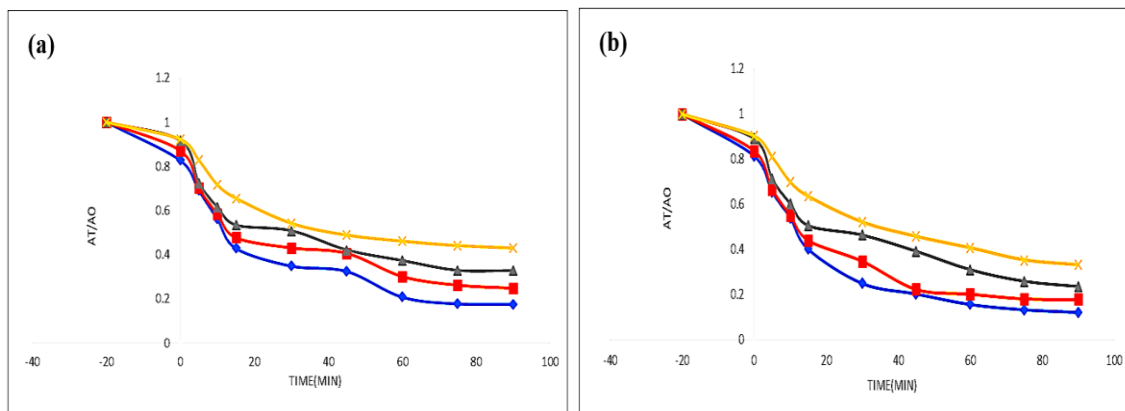


Figure 9. Effect of MO concentration on degradation performance in (a) O-BC/PMS and (b) B-BC/PMS systems.

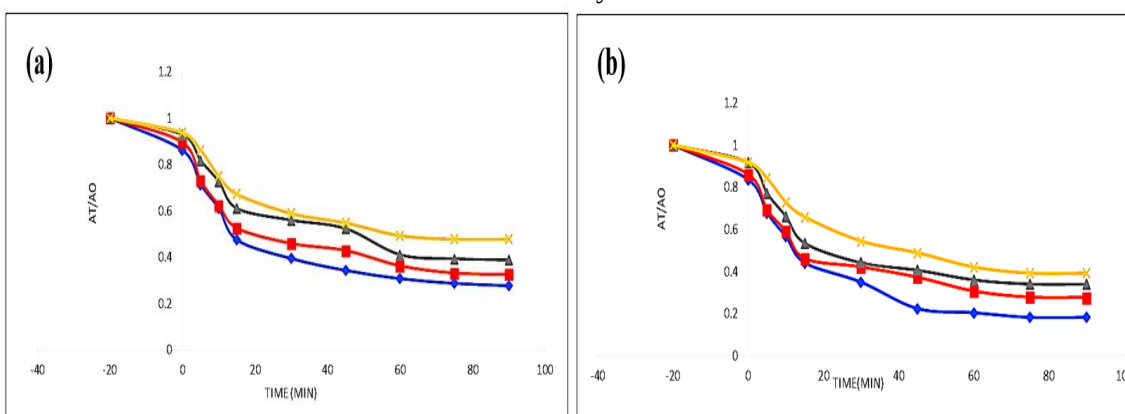
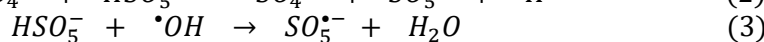
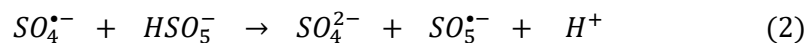


Figure 10. Effect of EBT concentration on degradation performance in (a) O-BC/PMS and (b) B-BC/PMS systems.

Effect of PMS Concentration

The effect of PMS concentration (25 mg, 50 mg, 100 mg, 200 mg) on the catalytic performance of O-BC and B-BC was also investigated, and the results are shown in Figures 11 and 12. The findings revealed that an excessive dose of PMS may reduce the catalytic activity by scavenging the already produced radicals. This occurs as a result of

the self-quenching reaction of PMS, which leads to the conversion of the sulfate radical ($SO_4^{\bullet-}$) and hydroxyl radical ($\bullet OH$) to form the anion of PMS ($SO_5^{\bullet-}$) with the weaker oxidation capacity, thus reducing the rate of degradation. Equations 2 and 3 show the reaction mechanism.



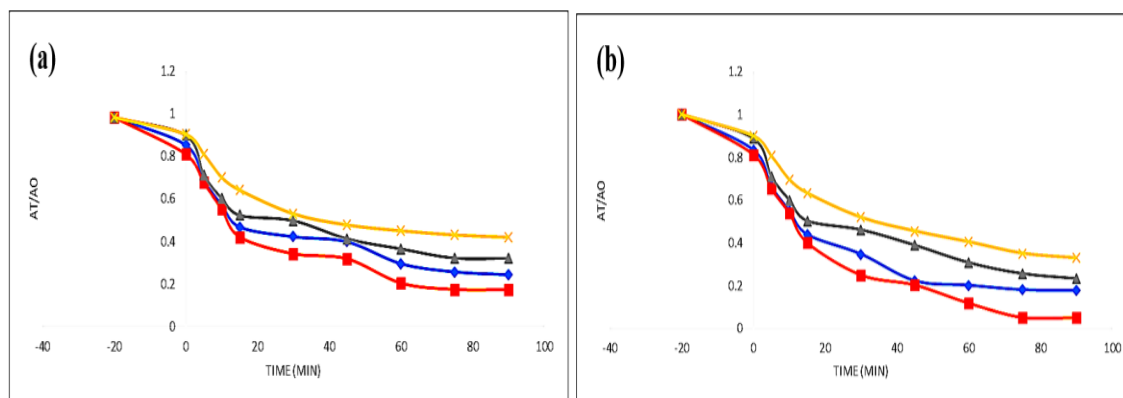


Figure 11. Effect of PMS concentration using (a) O-BC and (b) B-BC for MO degradation.

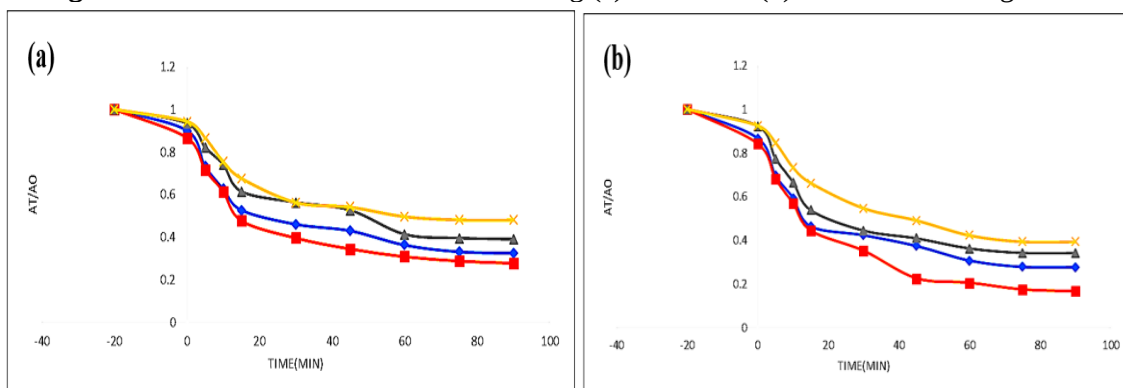
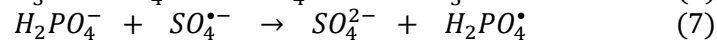
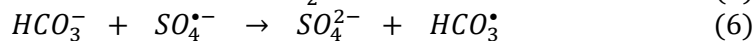
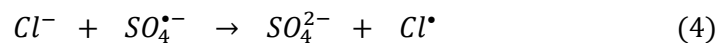


Figure 12. Effect of PMS concentration using (a) O-BC and (b) B-BC for EBT degradation.

Effect of Inorganic Ions

The effect of inorganic ions on the pollutant degradation was also investigated. The optimal conditions (0.3 g catalyst dosage, 20 mg/l pollutant concentration, and 50 mg PMS) were used for this analysis. The results (Figures 13 and 14) showed that the inorganic ions exhibited varying degrees of inhibition of dye degradation. Cl^- showed relatively slight inhibition. The inhibition of Cl^- could be a result of the reaction with $SO_4^{\bullet-}$ to generate

secondary chlorine radicals with lower redox potential (Equations (4) and (5)). HCO_3^- and $H_2PO_4^-$ have been reported to be common $SO_4^{\bullet-}$ and $\bullet OH$ scavengers (Equations (6) and (7)) possessing a greater inhibitory effect on the degradation of pollutants (Hu et al. 2018). However, the inhibitory effect of $H_2PO_4^-$ was stronger due to electrostatic interactions and hydrogen bonds with the surface functional groups of the biochar.



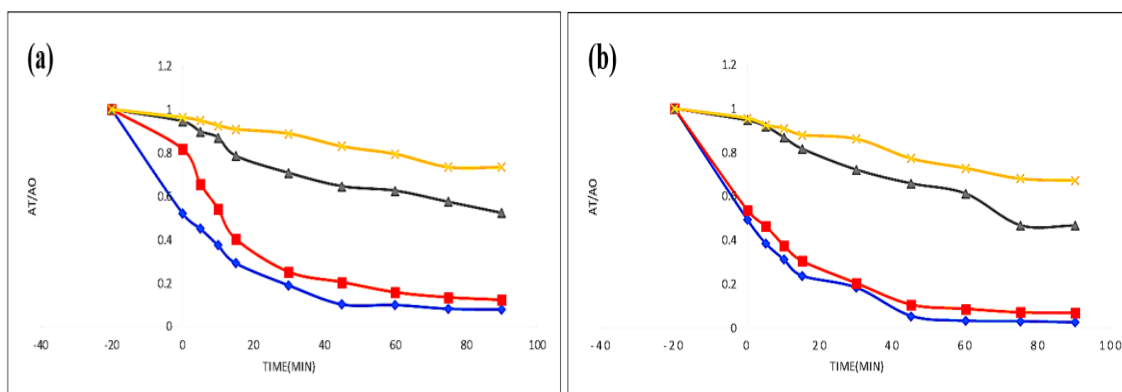


Figure 13. Effect of inorganic ions on the degradation of MO using (a) O-BC and (b) B-BC.

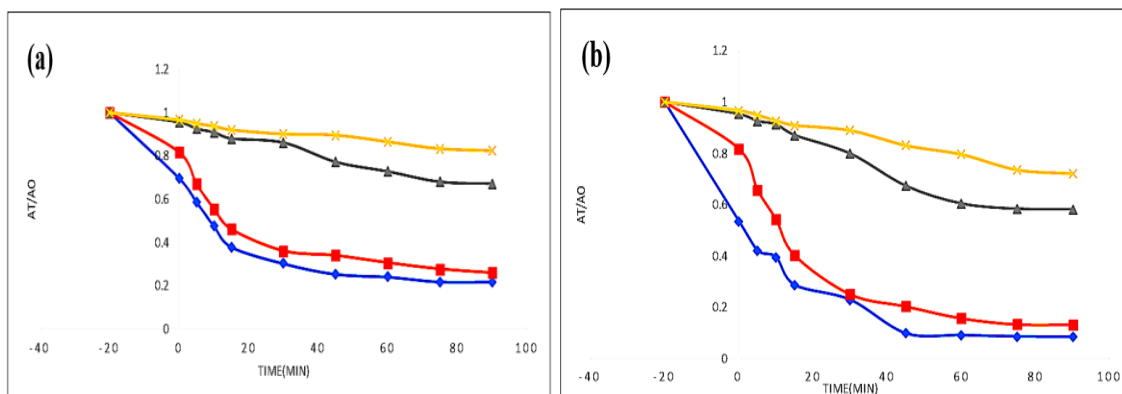


Figure 14. Effect of inorganic ions on the degradation of EBT using (a) O-BC and (b) B-BC.

Effect of Radical Scavengers

The effect of radical scavengers was studied with all the operational parameters kept constant, and the results are shown in Figures 15 and 16. Nitrobenzene (NB) was used to scavenge $\cdot OH$ (without significantly affecting $SO_4^{\cdot -}$) while sodium perchlorate was employed to inhibit the initiation of the non-radical pathway on the BC surface. Meanwhile, methanol was used to scavenge both $\cdot OH$ and $SO_4^{\cdot -}$.

It was observed that B-BC and O-BC exhibited high degradation efficiency in the absence of a scavenger, with B-BC showing the highest degradation efficiency of about 95% and 89% during MO and EBT degradation, respectively. This implies that, in the absence of a scavenger, the radicals produced by PMS activation are free to react with the pollutant, resulting in the highest degradation efficiency and the least inhibitory effect. Thus, proving that the radicals generated during PMS activation are the primary agents responsible for pollutant degradation. However, the addition of NB

resulted in a 5% decrease in degradation efficiency for both pollutants. This result shows that NB only induced a minor inhibitory effect on the dye degradation, indicating that $\cdot OH$ contribution was minimal (Sharma et al., 2021). Meanwhile, the addition of perchlorate and methanol results in a rapid decrease in degradation efficiency. Therefore, perchlorate could be predicted to inhibit PMS activation by competing with PMS in the reaction. Such competition reduces the production of radicals and also scavenges the already produced radicals ($\cdot OH$ and $SO_4^{\cdot -}$), leading to a moderate inhibitory effect. This moderate inhibitory effect suggests that non-radical mechanisms also contribute to the degradation, but to a lesser extent than sulfate radicals. This implies that both radical and non-radical pathways (as shown in Figure 17) contribute to dye degradation in the BC/PMS system, with the radical pathway playing a dominant role. Such pathways often lead to the generation of $SO_4^{\cdot -}$, 1O_2 and surface-activated complex for dye removal

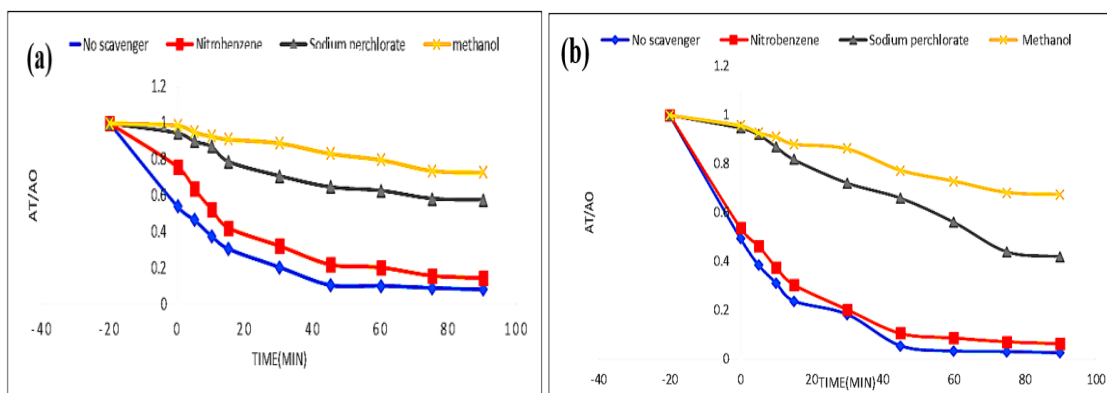


Figure 15. Effect of radical scavengers on the degradation of MO using (a) O-BC and (b) B-BC.

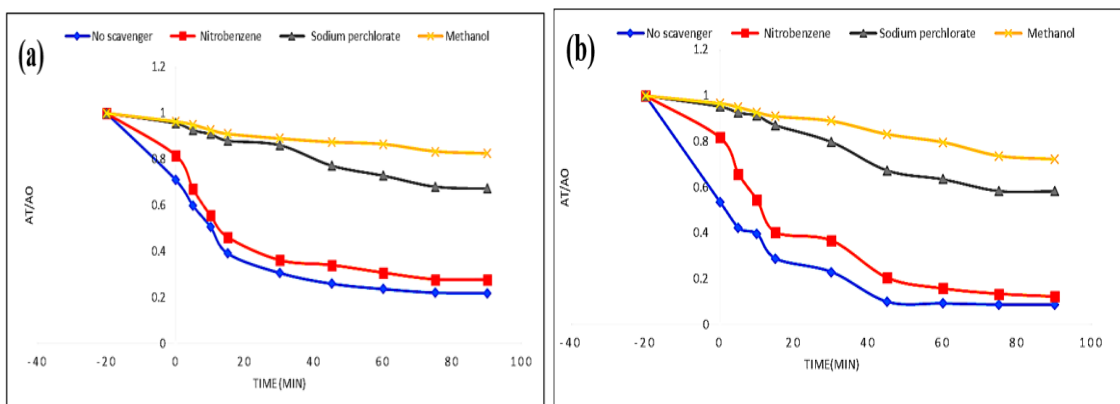


Figure 16. Effect of radical scavengers on the degradation of EBT using (a) O-BC and (b) B-BC.

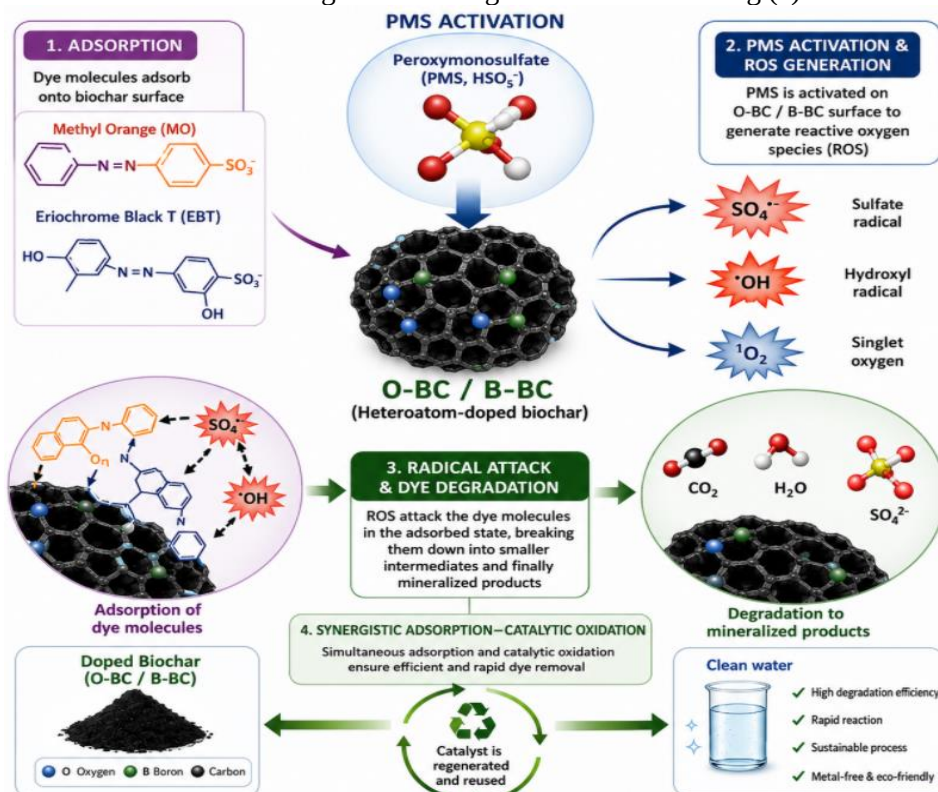


Figure 17. Proposed degradation mechanism of MO and EBT via PMS Activation using O-BC and B-BC.

Reusability Studies

The reusability of the catalysts was studied over four cycles. From the results (Figures 18 and 19), it was observed that after the second cycle, the catalytic performance decreased

during both MO and EBT degradation. The continuous decrease in performance is attributed to the difficulty of catalyst recovery after successive cycles and to the adsorption of dyes on the catalyst surface.

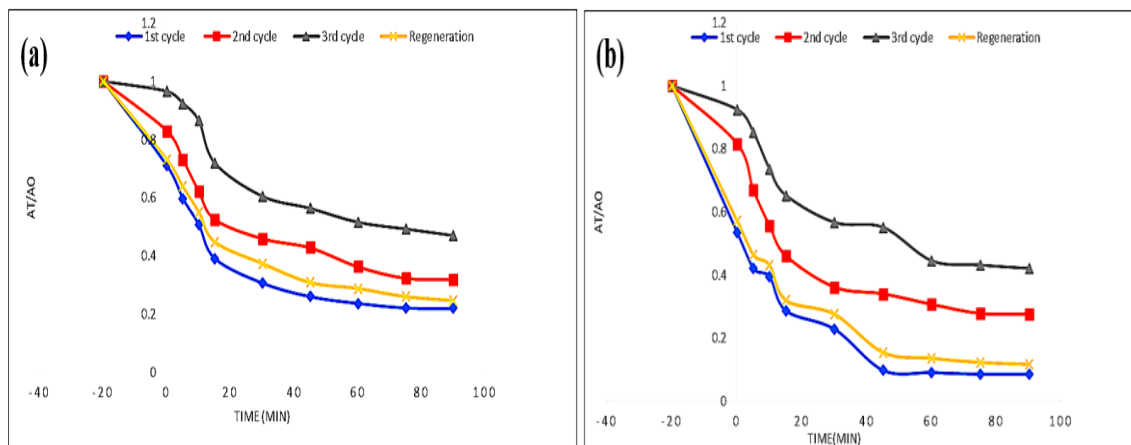


Figure 18. Reusability studies using (a) O-BC and (b) B-BC for MO degradation.

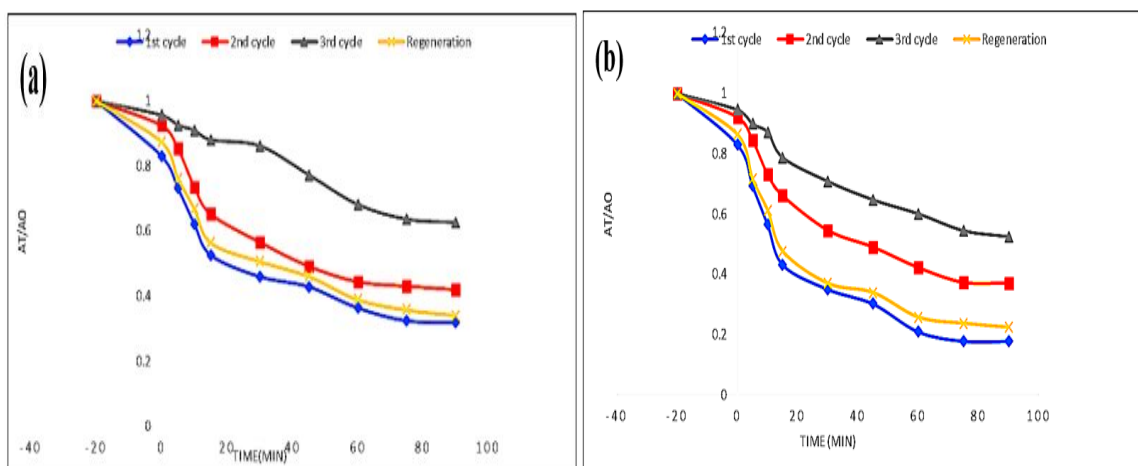


Figure 19. Reusability studies using (a) O-BC and (b) B-BC for EBT degradation.

Conclusion

This study developed oxygen- and boron-doped biochar catalysts from cornstalk using oxalic acid dihydrate and boric acid as precursors via pyrolysis, effectively converting agricultural waste into efficient, eco-friendly catalysts. The FTIR analysis revealed the presence of an oxygen-containing functional group on the biochar surface, which facilitated the activation of PMS and the generation of reactive oxygen species (ROS), leading to the oxidative degradation of the dye molecules. Structural analysis revealed significant changes in the crystallinity and phase composition of the doped biochar, indicative of the formation of crystalline

phases that contributed to its catalytic activity. SEM images provided visual confirmation of the morphological alterations induced by the doping process. The experimental results demonstrated that B-doped biochar exhibits superior catalytic performance, degrading 95% and 89% of MO and EBT in 60 min, respectively, compared to O-BC, which degraded 89% and 75% of MO and EBT in 90 min, respectively. The radical-scavenging experiment showed that the radical pathway was the dominant reaction. The catalyst's reusability was assessed over three cycles, with effective regeneration demonstrated after the third cycle.

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