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Coupling magnetic biochar assisted Fenton-like and activated sludge processes for the elimination of phenol and carbamazepine to promote wastewater reuse in agricultural systems

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ABSTRACT

Contamination of soil and water by pharmaceutically active compounds (PhACs), such as phenol and carbamazepine (CBZ), poses significant environmental risks, particularly in the reuse of treated wastewater for agriculture. This study explored the integration of activated sludge (AS) treatment with a magnetic biochar-assisted Fenton-like process to eliminate CBZ and co-pollutants such as phenol. The AS process achieved complete phenol degradation (100 mg/L), whereas iron-modified biochar (SBC-Fe) targeted CBZ. SBC-Fe exhibited superior adsorption efficiency (90 %) for CBZ due to hydrogen bonding and enhanced electrostatic interactions. When combined with H_2O_2 (1 g/L, 1 mM), SBC-Fe facilitated complete CBZ removal via catalytic oxidation. The integrated system effectively treated wastewater containing phenol and CBZ, with AS and SBC-Fe/H_2O_2 addressing each pollutant sequentially. Additionally, irrigation with treated wastewater resulted in an 80 % increase in *Lepidium sativum* growth, demonstrating the potential for sustainable agricultural reuse. This approach enhances pollutant removal and supports circular water management practices.

1. Introduction

As global freshwater scarcity intensifies, the need for innovative, sustainable wastewater management strategies has become increasingly urgent. The application of treated wastewater in agriculture has emerged as a promising solution to address water scarcity [1]. As conventional water resources become increasingly limited, utilizing treated wastewater for irrigation provides a viable alternative that not only conserves freshwater but also recycles valuable nutrients back into the soil.

Treated wastewater, which typically contains essential nutrients such as nitrogen, phosphorus, and potassium, can contribute to plant nutrition while significantly reducing the reliance on synthetic fertilizers. This approach reduces agricultural costs while supporting the sustainable reuse of water and nutrients, contributing to closing the nutrient loop in agricultural systems [2]. Unlike inorganic fertilizers, which are typically produced through energy-intensive processes and rely on non-renewable resources, treated wastewater provides a sustainable alternative by recycling existing nutrients, thereby lowering the environmental impact associated with fertilizer production [2]. However, the use of organic-based nutrients, such as those in treated wastewater, may increase the risk of nutrient runoff and eutrophication compared with the use of mineral fertilizers, depending on management practices [3]. Therefore, proper management is essential to mitigate these risks, ensuring that the environmental benefits of nutrient recycling are maximized while minimizing potential water pollution.

However, the presence of pharmaceutically active compounds (PhACs) in wastewater effluents is a concern because of their possible ecological and health impacts, such as possible toxic effects on aquatic microorganisms and the generation of antibiotic-resistant bacteria (ARBs) and antibiotic resistance genes (ARGs) [4,5]. The literature indicates that most conventional and widely used biological treatment

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systems for the treatment of industrial and municipal effluents are not able to efficiently eliminate PhACs [6,7]. Carbamazepine (CBZ, $C_{15}H_{12}N_O$) is an antiepileptic and mood-stabilizing drug commonly used to treat bipolar disorder, trigeminal neuralgia, and epilepsy [8]. It generally represents a low to negligible biodegradation rate (K_{biol}) and very low adsorption coefficient (K_d) in activated sludge (AS) processes [9,10]. Thus, it is crucial to implement degradation strategies to prevent the release of CBZ into the environment. In this context, various techniques, such as adsorption [11], membrane filtration [12,13], and advanced oxidation processes, e.g., ozonation [14], the Fenton reaction [15], electrochemical treatments [16], and photocatalysis [17], have been developed. However, the practical application of such technologies faces challenges, including their high operating costs and the relatively low degradation rates of some AOPs, such as those used for photocatalysis [17], for the removal of recalcitrant PhACs such as CBZ. Hence, it is essential to develop cost-effective, efficient, and environmentally friendly methods to integrate with and upgrade the current conventional AS processes [18].

Recent advances in carbon-based composites highlight their potential for synergistic pollutant degradation and resource recovery. For example, biomass-derived catalysts have been shown to enhance oxidation processes while enabling practical applications such as desalination [19] or targeted single oxygen generation [20]. Furthermore, biomass waste valorization (e.g., conversion of sludge to biochar) is increasingly recognized as a scalable strategy for sustainable water treatment [21].

Biochar (BC), a carbonaceous material derived from waste materials, has gained increasing attention in recent years for its application in wastewater treatment [22 –24]. The production of BC from various biomass wastes, including agricultural residues, woody materials, and waste activated sludge from wastewater treatment plants (WWTPs), has progressed significantly. This process not only converts and stabilizes carbon-rich compounds in these wastes but also produces BC, which can be used in a wide range of applications, such as soil amendment, carbon sequestration and wastewater treatment [25 –27].

The aim of this study is to evaluate the integration of aerobic biological systems with advanced oxidation processes (AOPs) to increase the removal of phenol and CBZ from wastewater. In this regard, this study specifically investigates the role of modified sludge biochar (SBC-Fe) in the degradation of CBZ, exploring how its modification enhances CBZ removal through adsorption and other potential mechanisms. The novelty of this research lies in the integration of SBC-Fe with the conventional activated sludge (CAS) process and the evaluation of their combined effectiveness. This study fills a knowledge gap by assessing how BC modification, particularly with iron, enhances CBZ removal through adsorption and additional mechanisms such as electrostatic interactions. Furthermore, research explores the integration of a Fenton-like reaction utilizing a heterogeneous catalytic system into the AS process. This combined treatment approach aims to increase the degradation of phenol and persistent pollutants such as CBZ, improve effluent quality for agricultural reuse, and minimize environmental impacts. The purpose of these treatments is to assess their individual and combined efficiencies in terms of pollutant removal and to evaluate their potential for improving existing wastewater treatment processes.

2. Materials and methods

2.1. Chemicals

Carbamazepine ($C_{15}H_{12}N_2O$, analytical grade, ≥98 %) was purchased from Sigma–Aldrich (Germany). Iron chloride ($FeCl_3$) was purchased from Fisher (UK) for the synthesis of magnetic BC. Other chemicals used are ammonium chloride (NH_4Cl) from Sigma–Aldrich (USA), sodium nitrate ($NaNO_3$, 99 % ACS reagent) from Acros Organics (India), sodium sulfite (Na_2SO_3) from Merck (Germany), sodium sulfide (Na_2S) from Acros Organics (Germany), hydrogen peroxide (H_2O_2) from VWR Chemicals (Belgium), sodium thiosulfate ($Na_2S_2O_3$) from Merck (Germany), magnesium sulfate ($MgSO_4$) from Honeywell (Germany), sodium bicarbonate ($NaHCO_3$) from Janssen Chimica (Belgium), dipotassium hydrogen phosphate (K_2HPO_4) from Chem-Lab (Belgium), and phenol (C_6H_6O) from Sigma–Aldrich (Germany).

2.2. Synthesis of biochar

Waste activated sludge from the municipal WWTP of Mechelen-Noord (Aquafin, Belgium) was used as the precursor for the synthesis of the BCs. The sludge was first oven-dried at 70 °C. The dried sludge was then ground and passed through an 80-nm mesh sieve. 4 g of ground sludge were then placed in a crucible and wrapped in aluminum foil. A tube furnace (Nabertherm) was used for the synthesis of the BC under a N_2 atmosphere, and BC was heated at a rate of 10 °C/min until 550 °C was reached. This temperature was then further maintained for 2 h. For the synthesis of the magnetic BC, 3 g of ground sludge was mixed with 10 mL of deionized water and 1.96 g of $FeCl_3$ and shaken for 30 min. The slurry was then dried at 95 °C. The resulting powder was used for the synthesis of the magnetic BC via a tube furnace under identical pyrolysis conditions as those described above.

2.3. Characterization of the materials

X-ray powder diffraction (XRPD) (Panalytical X'Pert PRO 3, Netherlands) analysis was performed to examine the crystalline structure of the powders. A continuous scan over a 2θ range from 10° to 100° was conducted at a scan rate of 2° 2θ/min. The elemental composition of the materials prepared was also determined using a Philips PW-1400 sequential X-ray fluorescence spectrometer (XRF) for elemental analysis. Scanning electron microscopy (SEM, TESCAN, VEGA, USA) was utilized to study the surface morphology of the prepared materials. The Brunauer–Emmett–Teller (BET) method was also employed to acquire N_2 gas adsorption/desorption isotherms and determine the specific surface area and porosity of the materials utilizing a Micromeritics Gemini V2 (USA). Particle size distribution (PSD) analysis was performed using a CILAS 1064 (France). In addition, Fourier transform infrared (FTIR) analysis was carried out using a Bruker Tensor 27 system to identify the vibration bonds of the surface functional groups of the BC and CBZ. The spectrum was obtained from 256 scans with a resolution of 4 cm^{-1} under transmittance mode in the range of 4000–400 cm^{-1}. The zeta potential of the prepared SBC-Fe particles was measured using a particle analyzer (Litesizer 500, Anton Paar). The pH of each sample was adjusted from 3 to 10 with NaOH (1 mol/L) and HCl (1 mol/L). Inductively coupled plasma–mass spectrometry (ICP–MS) was performed to determine the concentrations of specific elements in the effluents using a Spectro Acros FHS12. The total organic carbon (TOC) of the effluents was tested using a TOC-L analyzer (Shimadzu). Total carbon (TC) was measured using a Primacs SNC100 (Skalar, Netherlands).

2.4. CBZ degradation assays

To test the degradation efficiency of CBZ using the prepared materials, a glass beaker with a working volume of 100 mL under continuous stirring at 100 rpm was used. Different dosages of BC (0.5, 1, and 2 g/L) were used in combination with various concentrations of H_2O_2 (0.5, 1, and 2 mM) for the degradation of CBZ. At each sampling interval, 1.5 mL of the effluent was collected and filtered through a 0.45 μm filter and then immediately quenched by adding 0.1 M $Na_2S_2O_3$ solution to terminate the oxidation process. The concentration of CBZ was measured via high-performance liquid chromatography (HPLC). Eqs. (1) and (2) were used to describe the degradation kinetics of CBZ, which were fitted to a pseudo-first-order model.

$$dC/dt = -k_{obs}\, C \quad (1)$$

$$\ln (C_t/C_o) = -k_{obs}\, t \quad (2)$$

where C_0 and C_t are the initial CBZ concentrations and those after the reaction time (t), respectively.

2.5. Activated sludge combined with BC and advanced oxidation for phenol and CBZ removal

Waste activated sludge was obtained from the secondary clarifier of the municipal WWTP of Mechelen-Noord (Aquafin, Belgium). A synthetic effluent containing phenol, CBZ and nutrients with the composition listed in Table 1 was prepared for the relevant experiments. The experiments were performed using 5 L reactors under continuous aeration with an air stone diffuser to maintain the DO level above 2.5 mg/L, with an initial concentration of 1 g VSS/L in the reactors. The sludge was first fed phenol (100 mg/L) for 10 days to estimate its performance in treating phenolic wastewater ($T = 15–18$ °C, pH ≈ 6). The performance of the reactors was assessed by monitoring key parameters such as phenol removal efficiency, biomass activity (VSS concentration), and changes in COD levels over time. Phenol was initially chosen as a model compound because it is a well-established pollutant in biodegradation studies, allowing us to validate the system's capacity to treat phenolic compounds before moving to more complex contaminants such as CBZ. This approach ensures that the reactors are stable and capable of degrading organic pollutants before CBZ is introduced. The biodegradation of synthetic wastewater containing phenol alone (100 mg/L) without nutrients (Ph_{100}), with nutrients (Ph_{100}-N), with phenol (100 mg/L) and CBZ (5 mg/L) without nutrients (Ph_{100}-CBZ_5), with nutrients (Ph_{100}-CBZ_5-N), and with Ph_{100}-CBZ_5-N in the presence of SBC-Fe was studied using an initial sludge dosage of 1 gVSS/L.

The ability of the AS process was analyzed as the sole treatment method for the biodegradation of both phenol and CBZ. Additionally, a combination of adsorption and biodegradation was studied by adding BC to the AS system.

Finally, the applicability of the advanced oxidation process (AOP), as a post-treatment for removing residual pollutants from wastewater treated by the AS process, was assessed. In this step, 100 mL of the biologically treated wastewater was subjected to further treatment with H_2O_2.

2.6. Plant growth and phytotoxicity assay

In the phytotoxicity assay, plant growth was evaluated via quadruplicate pots for each condition, resulting in a total of eight sets. The first two sets (in Fig. 7 from right to left) were irrigated with wastewater containing 5 mg/L CBZ at pH values of 3.5 and 7. The subsequent two sets were irrigated with effluents from the AS process, with and without the addition of nutrients. Sets 5 and 6 were irrigated with effluents from the advanced oxidation process, without nutrients, at pH 3.5 and 7. Finally, sets 7 and 8 were irrigated with wastewater treated by the advanced oxidation process (AOP), where the nutrients were derived from a biological process preceding oxidation at pH values of 3.5 and 7.

Soil samples were taken from the topsoil (0–20 cm) of the garden lawns (silty loam soil), dried at ambient temperature, crushed in a porcelain mortar, passed through a 2 mm sieve, and stored separately for further analysis (Table 2). The pH and electrical conductivity (EC) of the soil were measured via a suspension sample with soil-to-distilled water ratios of 1:1 and 1:2, respectively [28]. Phosphorus was determined via inductively coupled plasma optical emission spectroscopy (ICP–OES, Spectro Arcos FHX 76004553), which was equipped with a charge-coupled device (CCD) detector to measure the emitted intensities.

Table 1
Composition of the synthetic effluents used in the present study.

Composition	Chemical formula	Concentration (mg/L)
Ammonium chloride	NH_4Cl	172
Magnesium sulfate	$MgSO_4$	49
Dipotassium hydrogen phosphate	K_2HPO_4	250
Sodium bicarbonate	$NaHCO_3$	14.7
Phenol	C_6H_5OH	100
CBZ	$C_{15}H_{12}N_2O$	5

Table 2
Characteristics of the soil.

Parameter	Soil
TS (%)	98.37 ± 0.03
VS/TS (%)	5.43 ± 0.19
pH	6.67 ± 0.02
Electrical conductivity (EC) (μS/cm)	94.55 ± 7.14
Water holding capacity (WHC) (mL/g)	0.55 ± 0.02
Total Carbon (TC) (%)	3.20 ± 0.26
Total nitrogen (TN) (%)	0.20 ± 0.02

Lepidium sativum (*L. sativum*) seeds were chosen for these tests because of their rapid growth rate and short lifespan, making them suitable candidates for assessing the applicability of wastewater effluent in agricultural practices.

15 seeds of *Lepidium sativum* were sown in each pot at a depth of 5 mm from the soil surface. Each plastic pot that had drainage holes at the bottom was filled with 100 g of soil that had been dried under sunlight conditions. Afterwards, a volume of 10 mL of each water mixture was applied to the respective pots at each day interval. The pots were kept at 22 °C ± 5 °C and placed under direct sunlight.

The plant samples were harvested on day 15, and the roots and edible parts of the plants were thoroughly washed with tap water and rinsed with distilled water. The lengths of the roots and stems were measured, and the plants were then left to dry in the oven at 105 °C for 24 h. Subsequently, the dry weights of the plants were determined.

2.7. Analytical methods

The composition of the sewage sludge was explored using a ZEISS (Imager A.2) fluorescence microscope coupled with scanning electron microscopy (TESCAN, VEGA, USA). The concentrations of phenol and CBZ were analyzed by an Agilent 1100 HPLC system (Agilent Technologies, Germany). For CBZ, a C18 column (Agilent Eclipse Plus, 4.6 × 250 mm, dp = 5 μm) was used at 30 °C. The mobile phase consisted of water and acetonitrile (CAN) at a ratio of 60:40 (*v*/*v*), and the UV detector was set at 284 nm. The sample injection volume was 30 μL. The total suspended solids (TSS) and volatile suspended solids (VSS) of the sludge were measured according to the standard methods (APHA, 2005).

3. Results and discussion

3.1. Biochar characterization

3.1.1. Crystallinity and elemental composition

The XRD analysis (Fig. 1a) of the BC revealed that the crystalline structures were related to their adsorption properties. The SBC shows peaks at 20.8° and 26.6°, which is consistent with the presence of quartz (SiO_2) in its structure, providing rigidity and stability to the matrix [17]. For SBC-Fe, additional peaks, such as those at 33.1° and 49.4°, correlate with α-Fe_2O_3 phases, indicative of successful Fe integration, enhancing the surface reactivity for adsorption [29]. XRF analysis (Fig. 1b) further confirmed the increased Fe_2O_3 content in SBC-Fe, which was correlated with its increased surface affinity due to iron oxide incorporation.

3.1.2. Morphology

SEM images (Fig. 2) revealed a distinct morphological transformation, with SBC-Fe presenting a flower-like structure, increasing the surface area available for CBZ interactions.

Particle size analysis revealed a smaller average size in SBC-Fe (75.2

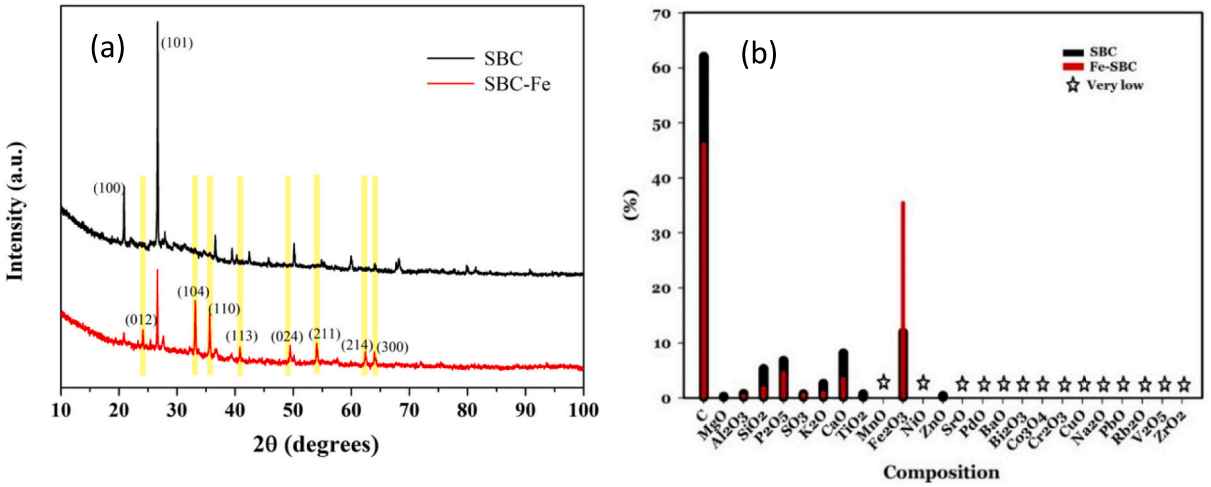


Fig. 1. (a) XRD spectra of the prepared SBC and SBC-Fe samples; (b) Composition of the SBC and SBC-Fe samples on the basis of the XRF results.

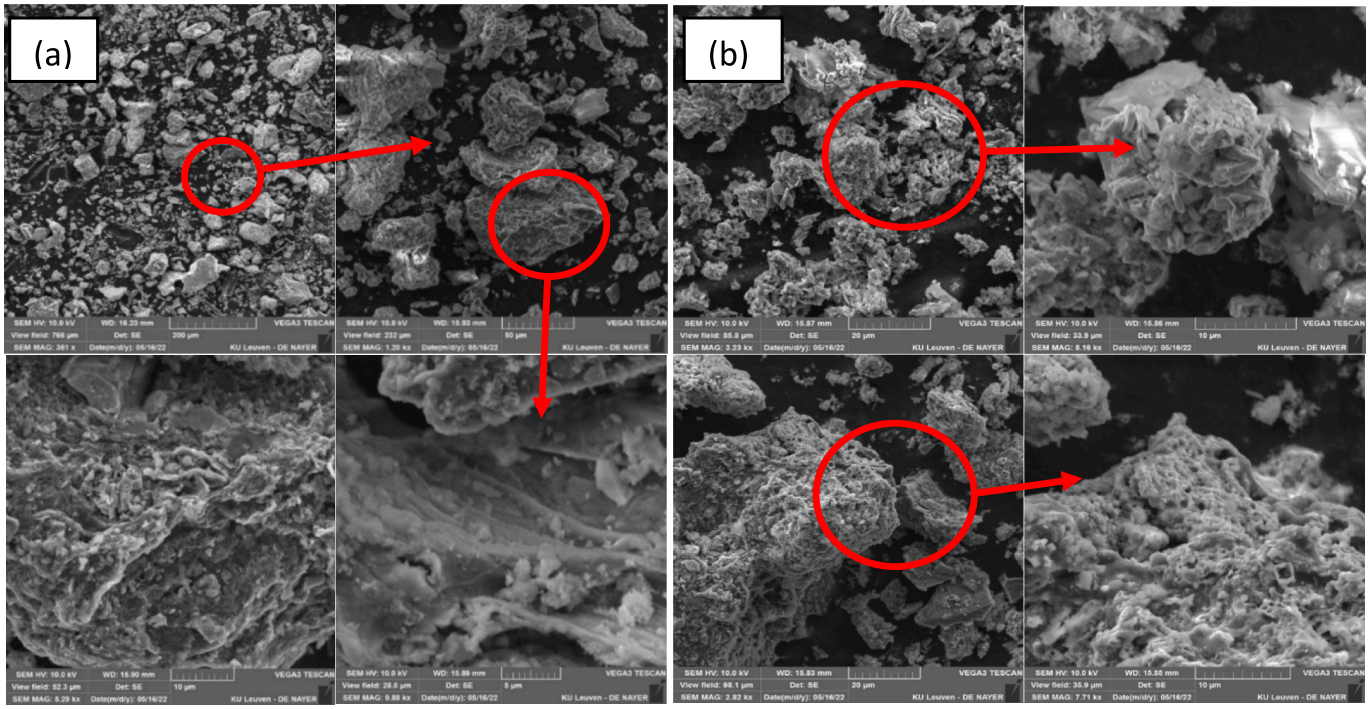


Fig. 2. Scanning electron microscopy images of the sludge-derived biochar (a); modified sludge biochar (b) at different magnifications.

μm) than in SBC (85.7 μm), indicating a finer dispersion of Fe-modified particles, which enhances diffusion and interaction with contaminants.

3.1.3. *Specific surface area and porosity*

Both SBC and SBC-Fe exhibit a mesoporous structure (IUPAC Type IV isotherm with H3 hysteresis), but SBC-Fe shows a decreased specific surface area and pore volume (25.44 m^2/g and 0.031 cm^3/g, respectively) relative to SBC (60.19 m^2/g and 0.070 cm^3/g) (Table 3), likely due to pore blockage by Fe_2O_3. This reduction in porosity aligns with studies showing that Fe incorporation can reduce the surface area while increasing the number of active adsorption sites, which is critical for CBZ binding [30].

Table 3
Specific surface area and pore size of different materials.

Samples	BET surface area(m^2/g)	Pore volume(cm^3/g)	Average pore size (nm)
SBC	60.1894	0.070469	7.5627
SBC-Fe	25.4364	0.031538	4.7327

According to the International Union of Pure and Applied Chemistry (IUPAC) classification, both SBC and SBC-Fe display a typical type IV isotherm with an H3 hysteresis loop [31], confirming the mesoporous structure of the materials.

The considerable decrease in surface area (60.19 → 25.44 m^2/g) and pore volume (0.07 → 0.03 cm^3/g) observed in SBC-Fe (Fig. 3 & Table 3) can be attributed to pore blockage by Fe_2O_3 nanoparticles during pyrolysis, as evidenced by (1) preserved mesoporosity (Type IV isotherm) with narrowed pore size distribution (7.56 → 4.73 nm), indicating partial pore filling rather than structural collapse; (2) XRF-measured Fe_2O_3 content matching the theoretical pore volume reduction (~58 % for 10–15 nm particles); and (3) maintained catalytic activity (100 %

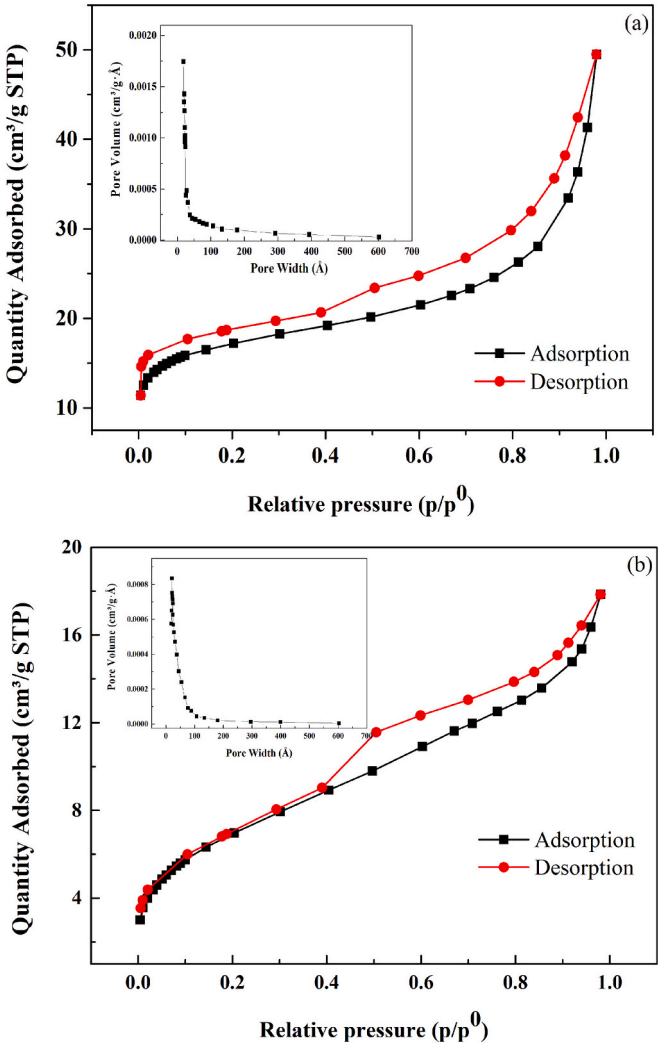


Fig. 3. N_2 adsorption–desorption isotherms and pore size distribution curves of (a) SBC and (b) SBC-Fe.

CBZ removal), confirming that Fe_2O_3 deposits remain accessible. The absence of micropore formation further supports pore blockage as the dominant mechanism over alternative explanations such as carbon matrix distortion. Similar results have been reported by Palansooriya et al. [30], who reported that the surface area of Fe-BC was lower than that of BC (7 m^2/g < 50 m^2/g). Other studies have confirmed that the addition of Fe decreases the specific surface area and porosity of carbonaceous materials.

3.2. Wastewater treatment

In this study, phenolic wastewater containing CBZ was treated via advanced treatment processes because of the resistance of CBZ to biodegradation and its persistence in conventional wastewater systems. The Fenton-like process was selected for its ability to generate hydroxyl radicals that can breakdown complex organic molecules such as CBZ, which are otherwise recalcitrant to biological treatment. Additionally, the AS process was applied, as it provides a biological pathway for the degradation of phenolic compounds. However, since CBZ is resistant to biological degradation, the combined use of Fenton-like oxidation and AS was implemented to increase the overall removal efficiency.

3.2.1. CBZ removal from synthetic wastewater via a Fenton-like process

SBC-Fe catalyzes CBZ removal primarily through oxidative degradation, with H_2O_2 serving as the primary oxidant. The incorporation of iron into BC enhances its catalytic efficiency, as it provides active sites for Fenton reactions.

Notably, SBC-Fe achieved complete degradation (100 % removal) of CBZ within 30 min under all tested conditions, whereas only 14 % removal was achieved with unmodified SBC after 60 min when SBC was paired with H_2O_2 (2 mmol/L) (Fig. 4). These results suggest that the oxidative capacity of SBC-Fe plays a critical role in CBZ degradation, leveraging H_2O_2 to generate highly reactive species, including hydroxyl radicals.

The degradation of CBZ by SBC-Fe may have a strong pH dependence and is governed primarily by three interrelated factors: Fe speciation, surface charge of the catalyst, and reactive oxygen species (ROS) generation. Under highly acidic conditions (pH < 3), increased Fe leaching may result in the loss of catalytically active iron species, reducing the overall system efficiency. In contrast, under alkaline conditions (pH > 6), ferric ions may tend to precipitate as $Fe(OH)_3$, limiting the availability of the surface-bound Fe^{2+} required for Fenton-like reactions [32]. Furthermore, at pH values below the point of zero charge ($pH_{PZC} \approx 5.2$), the surface of the BC becomes positively charged, enhancing the adsorption of CBZ via a combination of electrostatic attraction and hydrophobic interactions [33]. Overall, the superior catalytic performance of the SBC-Fe system at mildly acidic pH is attributed to a favorable balance between iron stability and efficient ROS generation. In contrast, extremely acidic or alkaline environments compromise the degradation efficiency through either iron loss or passivation by precipitation.

3.2.2. CBZ removal from phenolic wastewater via the activated sludge process

The biodegradation of synthetic wastewater containing phenol alone (100 mg/L) without nutrients (Ph_{100}), with nutrients (Ph_{100}-N), with phenol (100 mg/L) and CBZ (5 mg/L) without nutrients (Ph_{100}-CBZ_5), with nutrients (Ph_{100}-CBZ_5-N), and with Ph_{100}-CBZ_5-N in the presence of SBC-Fe was studied using an initial sludge dosage of 1 gVSS/L. The results revealed that 97 % and 96 % phenol biodegradation were obtained for the Ph_{100} and Ph_{100}-N conditions, respectively (Fig. 5).

According to this figure, the AS process was efficient for the biodegradation of phenol alone (100 mg/L) with and without supplementation of the additives, reaching complete phenol removal after 120 min. However, the reactor supplemented with the nutrients resulted in faster biodegradation kinetics than did the non-supplemented reactor (0.0089 min^{-1} and 0.0069 min^{-1}, respectively). This is because in the presence of nutrients, the metabolic activities of microorganisms can be facilitated [34]. Under these conditions, the lack of nutrients decreased

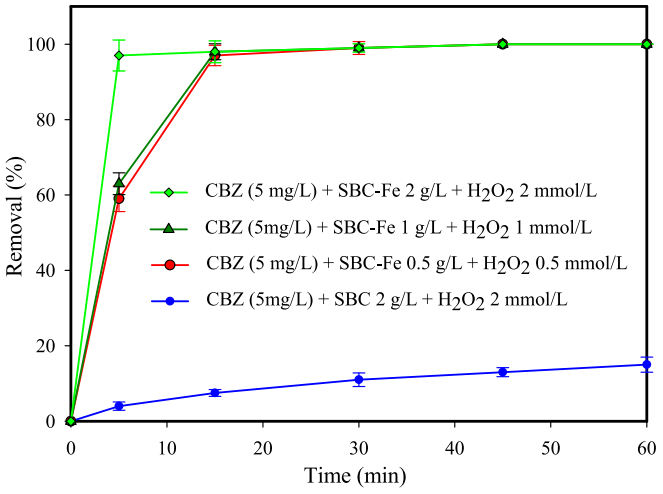


Fig. 4. The efficiency of the combination of the SBC-Fe system and H_2O_2 as an oxidant for the removal of CBZ from synthetic effluents.

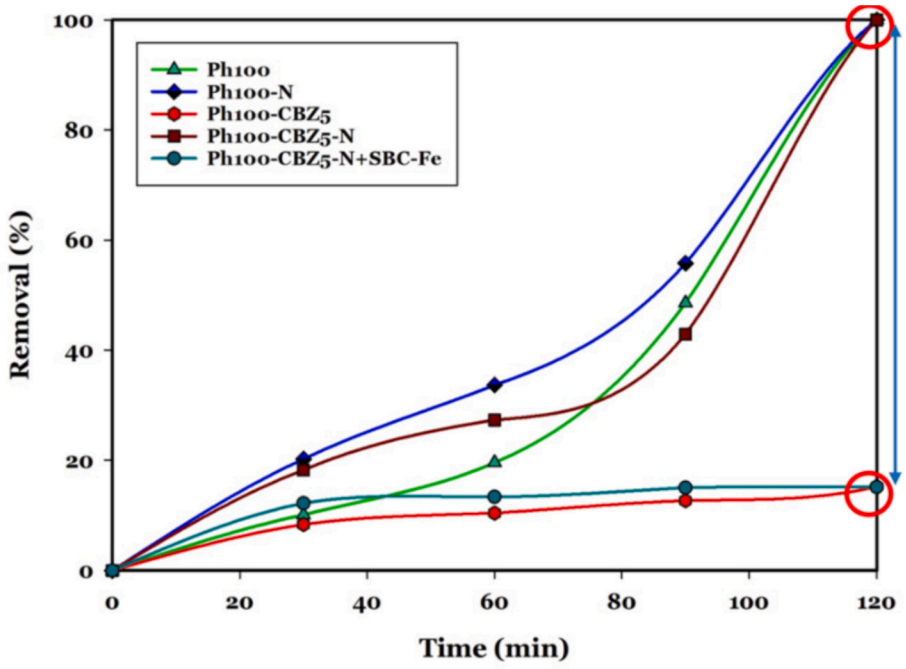


Fig. 5. Biodegradation of phenol in synthetic effluents containing phenol alone (100 mg/L) without nutrients (Ph_{100}), with nutrients (Ph_{100}-N), with phenol (100 mg/L) and CBZ (5 mg/L) without nutrients (Ph_{100}-CBZ_5), with nutrients (Ph_{100}-CBZ_5-N), and with Ph_{100}-CBZ_5-N in the presence of SBC-Fe.

the performance of the system in the presence of CBZ, leading to a low biodegradability rate of phenol (15 %) and CBZ (<5 %).

The optical density (OD) at 600 nm (OD600) indicator was also used to measure the growth of the microorganisms under various synthetic effluent compositions. According to the results, phenol in the reactor favored the growth development of the microorganisms, resulting in a nearly 27 % increase in their growth. Similarly, the growth rate of the microorganisms slightly increased over time, reaching 29.69 % after 120 min when nutrients were added to the reactors. Similar results have been reported in the literature, indicating that the availability of nutrients can determine the structure and composition of the microbial communities in AS [35]. In addition, CBZ resulted in a sharp decline in the cell growth density in all reactors (<9.48 %). Previous studies, such as the study of Vasiliadou et al. [36], reported that PhACs such as caffeine, sulfamethoxazole, and CBZ inhibit the growth of microorganisms in the AS process (especially sulfamethoxazole and CBZ). Although acclimatization strategies can enhance aerobic microbial community resistance against such PhACs, low biodegradability (K_{biol}) can be expected for these compounds [9]. Very low adsorption coefficient (K_d) values have been reported for pharmaceuticals such as CBZ, diclofenac, and naproxen [37].

Moreover, the results of this study revealed that the phenol biodegradation rate in the presence of SBC-Fe was very low (0.0012 min^{-1}). This can be explained as a possible shock to the microbial communities induced by the introduction of BC to the system due to the presence of organic and inorganic compounds [38]. The results were in line with the findings observed by $OD_{600,}$ where only 9.4 % microbial growth was recorded. There are few reports in the literature on the inhibitory and toxic effects of α-Fe_2O_3 on gram-positive (*Staphylococcus aureus*, Bacillus) and gram-negative (Klebsiella pneumonia, *E. coli*) bacteria [39]. SiO_2 can also have inhibitory effects on microorganisms [40], mainly through the regeneration of reactive oxygen species in the medium [41].

3.2.3. Combined activated sludge and Fenton-like oxidation process

The applied AS process was very efficient for removing phenol from synthetic wastewater, especially in the presence of nutrients. However, no significant removal of CBZ (<5 %) was observed, indicating the presence of this pharmaceutical at the same concentration as that in the influent. Hence, the AS process should be coupled with efficient complementary treatment to prevent the release of CBZ into the environment in real wastewater treatment applications. In the present study, the combination of SBC-Fe (0.5 g/L) and H_2O_2 (0.5 mM) was selected as the post-treatment method for removing contaminants from effluents that had already been subjected to the AS process (Ph_{100}-CBZ_5-N) (Fig. 6).

As shown in Fig. 6, 97 % of CBZ with an initial concentration of 5 mg/L was removed after 30 min. Full degradation of CBZ was achieved after 45 min of reaction (k_{obs} = 0.22 min^{-1}).

The SBC-Fe/H_2O_2 system is presumed to operate via both radical and non-radical mechanisms on the basis of the following evidence: (1) The observed pseudo-first-order kinetics (k = 0.22 min^{-1}) and the rapid depletion of H_2O_2 are indicative of •OH-mediated oxidation, which is consistent with Fe^{2+}/Fe^{3+} redox cycling, as previously reported for ferric-nitrilotriacetate (Fe(III)-NTA/H_2O_2) systems at neutral pH (k ≈ 0.0419 min^{-1}) [42]. (2) Persistent catalytic activity under circumneutral pH conditions combined with minimal iron leaching (<1.7 %) suggests the involvement of surface-mediated heterogeneous pathways, which is consistent with mechanisms reported for similar Fe–BC composites [43]. Although direct identification of reactive species via electron paramagnetic resonance (EPR) was beyond the scope of this study, previous work has confirmed the presence of persistent free radicals (PFRs) in BC that generate •OH upon reaction with H_2O_2 [43]. Additionally, trace Fe(III) loading on BC has been shown to increase •OH production by up to twelvefold without significantly increasing H_2O_2 consumption, suggesting a synergistic Fe–PFR interaction [42]. Furthermore, hydrogen bonding and electrostatic interactions likely contribute to CBZ enrichment near catalytically active sites, facilitating synergistic adsorption–oxidation processes.

A slight decrease in the efficiency of the system can be seen compared with the identical conditions used but without nutrients (Fig. 6). This could be due to the presence of nutrients from the AS process. Similar results have already been reported in the literature.

For example, Bacardit et al. [44] reported that anions can capture hydroxyl radicals, which results in the production of fewer reactive anion radicals that can then react with hydrogen peroxide. For example, chloride ions react with Fe to form complexes (Eqs. 3–6), or they can act as scavengers, reacting with hydroxyl radicals (Eq. 7) [45]. Other anions

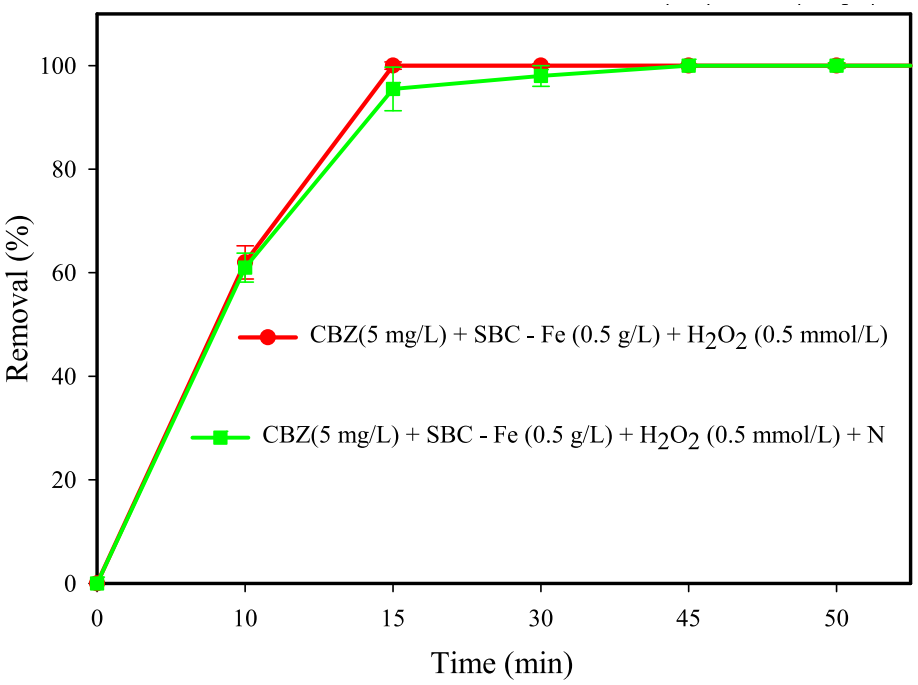


Fig. 6. The efficiency of the combination of the SCB-Fe system and H_2O_2 as an oxidant as a post-treatment for the removal of CBZ from synthetic effluents treated by the activated sludge process.

have similar effects.

$$Fe_2^+ + Cl^- \leftrightarrow FeCl^+ \quad (3)$$

$$FeCl^+ + Cl^- \leftrightarrow FeCl_2^0 \quad (4)$$

$$Fe_3^+ + Cl^- \leftrightarrow FeCl_2^+ \quad (5)$$

As indicated by Sima & Sipos [46], Fe_3^+ forms complexes

$$Fe_3^+ + 2Cl^- \leftrightarrow FeCl_2^+ \quad (6)$$

$$Cl^- + OH^* \rightarrow ClOH^{*-} \quad (7)$$

While synthetic wastewater was used in this study to isolate CBZ removal mechanisms, evidence suggests that the SBC-Fe/H_2O_2 system remains effective in complex matrices containing moderate chloride concentrations or natural organic matter (NOM) [47].

3.2.4. Stability and reusability of SBC-Fe and scale-up considerations

The long-term applicability of SBC-Fe was evaluated over five successive reuse cycles. During these cycles, the CBZ removal efficiency decreased by approximately 20 %, which was attributable mainly to pore blockage and partial oxidation of Fe^{2+} to Fe^{3+}. Similar trends have been observed in Fe-BC systems, where structural changes and iron oxidation degrade performance upon reuse [48]. Importantly, a simple acid wash using 0.1 M HCl effectively restored the removal efficiency to 92 %, demonstrating robust catalyst regeneration. Comparable studies report that acid treatments can successfully recover both adsorption and catalytic performance in Fe-modified BCs by clearing blocked pores and remobilizing active iron species [49].

Iron leaching was negligible (<1.7 % of total Fe), confirming the minimal risk of secondary iron pollution. This aligns with observations in other Fe-BC systems, where iron dissolution remains below 1–3 % under similar conditions, preserving environmental safety and catalyst integrity [50].

While this study demonstrated efficient CBZ removal (100 % in 30 min) at the laboratory scale, practical implementation would require (1) a two-stage continuous system coupling CAS (for bulk organics) with a stirred-tank SBC-Fe/H_2O_2 reactor (for polishing), leveraging magnetic recovery, and (2) process optimization to address challenges such as catalyst fouling mitigated via acid washing and H_2O_2 dosing control (automated ORP/pH sensors). Pilot-scale studies should evaluate long-term stability under realistic conditions (e.g., variable flow rates and seasonal water matrix changes). The system's modular design and use of waste-derived BC align with circular economy principles, but fouling monitoring and iron leaching thresholds require field validation.

As summarized in Table S1 (Supplementary Material), the proposed SBC-Fe/H_2O_2 system achieves 100 % CBZ removal with better scalability (magnetic recovery) than conventional methods such as ozonation (~90 % removal; high cost) or homogeneous Fenton processes (>90 % removal; sludge disposal challenges).

3.3. Plant growth and phytotoxicity tests

In a 15-day phytotoxicity study, pots were irrigated with synthetic wastewater containing 5 mg/L CBZ and effluents from an AS process with the same CBZ concentration. A total of 5.5 mg/kg CBZ was introduced into the soil of each pot. Although this study assessed the phytotoxic effects of CBZ using synthetic wastewater containing 5 mg/L CBZ, conventional irrigation sources such as tap water or groundwater typically contain CBZ at concentrations 10^3–10^4 times lower (i.e., in the ng/L range). The use of elevated CBZ concentrations in this study was intended to evaluate the plant response under worst-case exposure scenarios.

Reduced plant growth was observed in all pots treated with synthetic wastewater containing 5 mg/L CBZ, regardless of whether the pH was acidic (3.5) or neutral. This reduction in growth was also noted in plants exposed to the effluents from AS processes under the same CBZ concentration and pH conditions. The findings in Fig. 7 show that the greatest plant growth occurred in pots irrigated with wastewater treated with AOPs, which included nutrient additions applied during the AS process.

The observed reduced growth in pots irrigated with synthetic wastewater and AS effluents containing CBZ is consistent with the findings of a previous study [51]. These findings show that CBZ

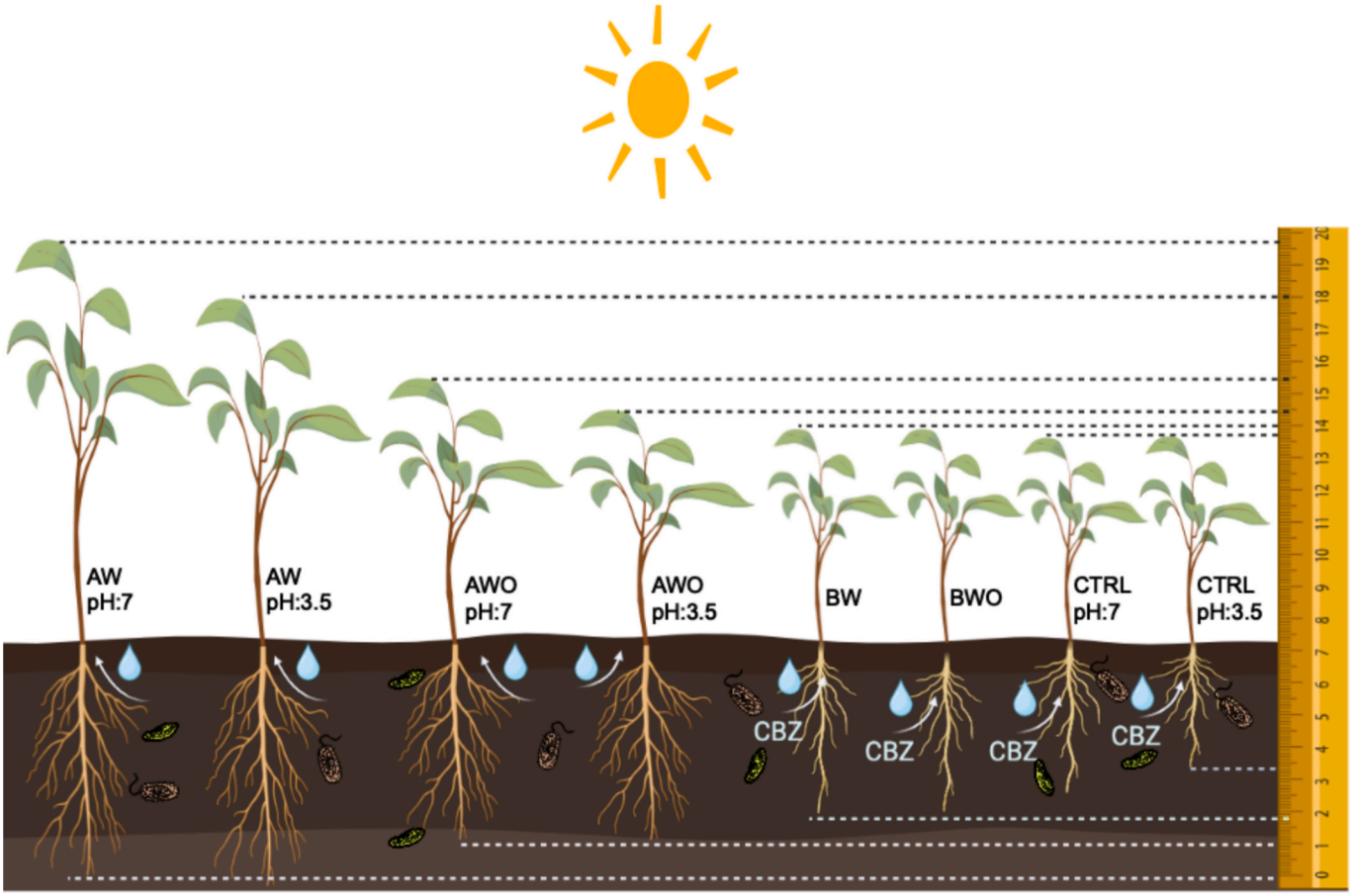


Fig. 7. Effect of wastewater effluent on plant growth. CTRL: synthetic wastewater containing 5 mg CBZ/L; BW: effluent from the activated sludge process with nutrients; BWO: effluent from the activated sludge process without nutrients; AW: effluent from the advanced oxidation process with nutrients; AWO: effluent from the advanced oxidation process without nutrients.

predominantly remains in the aqueous phase due to its hydrophilic properties and low soil sorption capacity, which facilitate its uptake by plants. As a nonionic compound, CBZ readily crosses plant root membranes via passive diffusion [52], bypassing the energy requirements typical of ionic compounds. As plants lack specific transporters for xenobiotics such as CBZ, passive uptake leads to accumulation within plant tissues, potentially impacting growth by targeting ion channels and enzymes that are critical for transporting essential nutrients [52,53].

In the CBZ-spiked pots, nutrient uptake was likely restricted, and the plant hormone balance may have been disrupted, both of which are important factors for plant growth. Carter et al. [54] reported that CBZ induced changes in plant nutrient and hormone homeostasis, which became detectable in plants exposed to CBZ concentrations as low as 0.005 mg/kg, with significant adverse effects between 4 and 10 mg/kg. They noted that CBZ may disrupt ion transport and alter membrane potentials, which are essential for sugar translocation within the plant. Disruptions in the membrane potential may lead to symptoms of sugar deficiency, contributing to necrosis in CBZ-exposed plants. The lowest-observed-effect concentration (LOEC) was approximately 2 mg/kg, with $p < 0.002$, indicating that even low CBZ levels can interfere with plant physiological processes. Moreover, exposure to CBZ at 0.01 to 1 mg/kg was associated with significant chlorophyll reduction, further highlighting its phytotoxic effects [52,55].

The increased growth observed in pots irrigated with AOP-treated wastewater supplemented with nutrients suggests that nutrient availability plays a crucial role in mitigating the phytotoxic effects of CBZ. The added nutrients likely support soil microbial activity, which in turn enhances the availability of essential nutrients for plant uptake, promoting growth. Additionally, residual H_2O_2 concentrations were noted in the treated wastewater: 0.89 mM with nutrient supplementation and 2.09 mM without nutrient supplementation. This difference in H_2O_2 concentrations can be attributed to the interactions between H_2O_2 and the nutrient components present in the wastewater. Elevated H_2O_2 levels in nutrient-free wastewater may induce oxidative stress in plants, triggering the accumulation of ROS, which can damage cellular structures and further inhibit growth [56].

In this study, while analysis of degradation byproducts was not conducted, the literature confirms that the Fenton-like oxidation of CBZ primarily yields low-toxicity intermediates [57]. Moreover, the observed 80 % increase in L. *sativum* growth indicates the potential suitability of the treated water for irrigation purposes. Nonetheless, additional ecotoxicological assessments would strengthen this conclusion. Notably, *L. sativum* is recognized as a sensitive bioindicator for pharmaceutical contaminants, with previous studies reporting a strong correlation between its response and that of standard aquatic bioassays [58,59].

These findings emphasize the potential of supplementing AOP-treated wastewater with essential nutrients to improve plant growth by increasing nutrient bioavailability and reducing oxidative stress. However, the persistence of CBZ in the environment and its phytotoxic effects on plants underscore the importance of advanced treatment approaches to effectively remove pharmaceutical contaminants from wastewater intended for agricultural reuse.

3.4. Regulatory implications

The SBC-Fe/H_2O_2 system reduced CBZ concentrations in the treated effluent from CAS to levels below 0.1 μg/L, which meets or surpasses the precautionary thresholds proposed for water reuse applications (0.1–1 μg/L). Furthermore, the effluent complied with key quality criteria for unrestricted irrigation as defined by the World Health Organization (WHO), including phenol levels below 0.1 mg /L. Although CBZ is not currently regulated by explicit threshold values for agricultural reuse, the absence of phytotoxic effects, as evidenced by an 80 % increase in L. *sativum* growth, aligns with the WHO guideline criterion of 'no observable adverse effects' on crop health.

For field-scale deployment, two operational considerations are recommended: (1) regular monitoring of residual CBZ with a limit of detection (LOD) of 0.05 μg /L to ensure compliance with future regulatory developments and (2) periodic catalyst regeneration via acid washing (0.1 M HCl) to maintain long-term performance. Given the system's compatibility with conventional treatment infrastructure, it offers a practical and cost-effective solution for wastewater reuse, particularly in regions aligning with the WHO's 2023 water reuse framework.

4. Conclusions

This study investigated the potential of BC derived from municipal sewage sludge for CBZ removal from wastewater, complementing CAS treatment, which fully degrades phenol. SBC-Fe demonstrated a relatively high adsorption efficiency, achieving up to 90 % CBZ removal (2 g/L BC, 1 mg/L CBZ) within 120 min, whereas non-modified BC removed less than 20 % of CBZ under similar conditions. When combined with H_2O_2 (2 mM), SBC-Fe achieved complete CBZ removal within 5 min under acidic conditions, which served as a polishing step after AS treatment. These findings highlight a synergistic two-stage strategy: AS for biodegradable organics (e.g., phenol) and SBC-Fe/H_2O_2 for recalcitrant PhACs (e.g., CBZ). While these findings suggest a promising application for CBZ mitigation, further studies are necessary to evaluate the economic feasibility and operational implications of using SBC-Fe and H_2O_2 for wastewater treatment. Addressing such parameters could advance the potential for integrating this treatment approach into sustainable water management practices, supporting both environmental and agricultural outcomes.

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CRediT authorship contribution statement

Rahim Molaey: Writing – original draft, Visualization, Methodology, Investigation, Formal analysis, Data curation. **Mohammadreza Kamali:** Writing – original draft, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Bodhi Laats:** Methodology, Investigation, Formal analysis. **Najmeh Askari:** Writing – review & editing, Investigation, Data curation. **Raf Dewil:** Writing – review & editing, Supervision, Methodology, Conceptualization. **Lise Appels:** Writing – review & editing, Supervision, Methodology, Conceptualization.

Declaration of competing interest

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests: Rahim Molaey reports financial support was provided by Horizon Europe. Najmeh Askari reports financial support was provided by Horizon Europe. If there are other authors, they declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Data availability

Data will be made available on request.

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