

Hydrothermal Synthesis of Fenton Catalyst from Soybean Curd Residue Biochar for Tetracycline Degradation

Quang Sang Huynh¹, Quyen Kim Thi Doan², Nguyen Duy Dat^{3*}

¹Phuc Thien Long Services Trading Joint Stock Company, Ho Chi Minh City, Vietnam

²Ton Duc Thang University, Vietnam

³Ho Chi Minh City University of Technology and Education, Vietnam

*Corresponding author. Email: datnd@hcmute.edu.vn

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ABSTRACT

In this study, heterogeneous catalysts were synthesized by hydrothermal method to load nano goethite to biochar derived from soybean curd residue, which served as catalysts for the heterogeneous degradation of tetracycline hydrochloride (TCH) in an aqueous solution. The catalytic tests using this composite material demonstrated significant TCH degradation. After 90 min of reaction, the optimum degradation of TCH in the aqueous solution was achieved. The initial pH value and TCH concentration were set at 2 and 50 mg/L, respectively, and the ambient conditions were maintained. The results showed that 0.5 g/L of catalyst and 60.0 mM H₂O₂ were the ideal catalyst and reagent dosages. Experimental data showed that the second-order kinetic model accurately described the degradation process than the first-order kinetic model. The study showed that biochar-loading goethite could be prepared from soybean crud residue and used for the degradation of TCH in an aqueous solution. Additionally, these results also provide a new approach for catalyst generation by the hydrothermal method that might help reduce costs and be environmentally friendly.

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1. Introduction

Nowadays, one of the most common antibiotics, tetracycline hydrochloride (TCH), represents a hazard to both humans and other living things when it leaks into the water source [1]. The increasing demand for personal care and medicinal goods has an impact on the environment as well. It's interesting to note that even in low quantities, leftover drugs and genes associated with antibiotic resistance can be generated and diffused, leading to fetal harm or organ abnormalities [2]. Antibiotic residues and poor biodegradability make traditional wastewater treatment techniques unsuitable for removing antibiotics [3]. Consequently, it is imperative to look for a practical technique to remove organic antibiotic contamination. Numerous techniques, including adsorption [4], oxidation [5], solvent extraction [6], membrane separation [7], biodegradation [8], and others, have been used to remove TCH from wastewater. These techniques, however, are typically vulnerable to intricate processes and potential secondary contamination. One of the best ways to get rid of refractory organics is through the use of advanced oxidation processes (AOPs), which produce reactive hydroxyl radicals ($\bullet\text{OH}$). With its discovery, the Fenton reaction received a lot of interest as one of the most sophisticated and effective oxidation reactions.

The two types of Fenton reactions typically distinguished are homogenous and heterogeneous [9]. Fenton techniques are widely utilized because of their cost-effectiveness, ease of usage, and high $\bullet\text{OH}$ production efficiency [10]. However, there are multiple limitations on the utilization of Fenton processes. In other words, the homogeneous sort is not cost-effective and has particular problems with ion recovery and separation following catalyst treatment [11]. Additionally, it has been noted that this procedure may create metal hydroxide sludge and secondary pollutants like acid or metal ions [11]. The application of the heterogeneous Fenton system can help solve the issues that were previously discussed. The heterogeneous Fenton technology is a range of heterogeneous Fenton reagents, including Fe₃O₄

[12], Fe₂O₃ [13], FeOOH [14], and applying Fenton's catalyst to the surfaces of various carriers, such as biochar, activated carbon, graphene, and so on [15].

It has been established that goethite works effectively as a heterogeneous Fenton-like catalyst to degrade organic contaminants [16]. It can effectively activate H₂O₂ to generate hydroxyl radicals due to the presence of Fe²⁺, magnetite which initiates the Fenton reaction. Notably, Goethite could participate in the Fenton heterogeneous catalyst process; However, for Goethite-quantity saving purposes, the loading of iron on the surface could not only increase the catalyze efficiency but also save the amount of Fe. Therefore, the selection of the catalytic surface plays an important role [17], [18]. There are a variety of catalyze materials that require the cost-effectiveness, and they often use materials derived from agricultural debris and, through thermolysis, produce biocarbon [19]. Previous studies have also shown the use of biochar as a catalyst due to the attachment of iron ions to the biochar surface during the decomposition of pollutants [20], [21]. The transformative methods of attaching iron ions to the biochar surfaces of these studies usually use pyrolysis and impregnation. There are numerous approaches to loading iron in the form of oxides on biochar support, including hydrothermal, co-precipitation, and sol-gel processes [22].

The hydrothermal method is the most widely utilized since it is simple to perform and occurs at relatively moderate temperatures [23]. One benefit of the non-extreme reaction temperatures is that the hydrothermal process uses less energy. Simultaneously, it permits the use of low-cost, basic compounds. Because the hydrothermal treatment is done at a low temperature, the reactions happen quickly [23]. Moreover, the hydrothermal approach provides benefits for the creation of a strong catalyst. This method was chosen and utilized for modifying the precursor biochar created via pyrolysis of biomass sources (soybean crud residue). Many biomass feedstock sources and adaptable preparation techniques allow for the development of biochar with a range of uses and applications in the processes of water pollution, mineralization, and degradation [24]. Furthermore, this food waste is typically recovered by pyrolysis, which produces carbonaceous materials, in closed furnaces with or without very little oxygen present [25]. By releasing greenhouse gases into the atmosphere, this has the benefit of reducing air pollution [22].

This study used pyrolysis to create biochar from dried soybean crud residue [26]. The iron in the previously - made biochar was then immobilized using the hydrothermal process. It also investigated the way the particles worked under various pH and dose catalyst settings to aid in the Fenton oxidation of TCH. The kinetics of the TCH degradation were also modeled using kinetic models that had previously been published in the literature in order to determine the kinetic parameters related to the catalysis or degradation processes involved.

2. Materials and Methods

2.1. Chemicals

Tetracycline hydrochloride was given by Sigma Aldrich. Deionized (DI) water was utilized to create the solutions employed in this investigation, and all other reagents such as KOH, HCl, FeCl₃.6H₂O, and ethanol 96° were analytical grade.

2.2. Preparation of catalyst materials

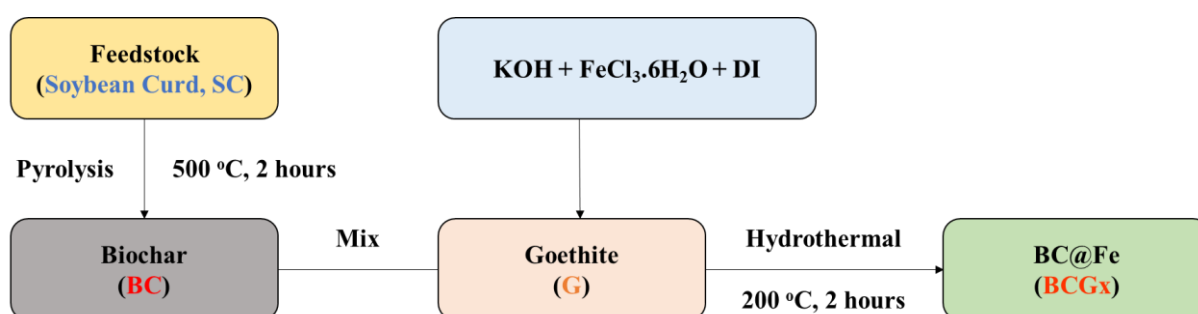


Figure 1. Materials synthesizing processes

Figure 1 highlights the materials synthesis techniques employed in this work. Soybean curd residue from the tofu-making process was collected at traditional markets. First, it was dried at 105 °C for 24 hours and sieved to a size of less than 0.25 mm for storage; the dried soybean curd residue was then referred to as SC. To make soybean curd biochar (BC), the sample was calcined in a furnace at 500 °C for 2 hours, with a heating rate of 10 °C/min. The BC substance was then sealed in an airtight container for further activation.

The Goethite (α -FeOOH) utilized in this investigation was produced using FeCl₃. Firstly, a magnetic stirrer was used to thoroughly mix KOH 5M and FeCl₃.6H₂O 1M for 10 min. Secondly, the freshly created ferrihydrite may be able to age to goethite by drying at 70 °C for 24 hours. Subsequently, the production was washed several times with ethanol (C₂H₅OH) and distilled water to adjust the final solution pH to approximately 7. After being dried for 24 hours at 105 °C, the finished product was ground to a size of 0.0105 μ m [27].

To create the potential in the oxidation ability of the material, the BC sample was paired with α -FeOOH to synthesize new materials for heterogenous oxidation process. BC is mixed with goethite in different proportions, denoted with BCGx in a Teflon-lined autoclave, where x is the percentage of goethite in the composite materials. The mixture was stirred at room temperature for 0.5 hours before heating to 200 °C for 2 hours.

2.3. Analytical methods

Numerous operating parameters, such as pH and contact time, were used to perform the heterogeneous Fenton process of TCH onto the composite material. Every experiment was conducted with the starting H₂O₂ concentration and the solid/liquid (m/V) ratio held at 0.5 g/L. The substance was added precisely 0.02 g to 40 mL of TCH-containing solution. The pH of solutions was adjusted both before and during the Fenton process using NaOH and HCl (1M). After that, the mixture was stirred for a predetermined amount of time at a regulated temperature at the speed of 250 rpm in an isothermal shaker. A 0.45- μ m filter paper was used to remove the loaded catalyst from the mixture after the specified contact time. Additionally, the same procedures were followed in the preparation of the controlled sample. Throughout the experiments, the laden solids were collected and dried for 24 hours at 105 °C to examine the material's properties. Using a UV-vis spectrophotometer (Hitachi U-2910, Hitachi Corp. Japan), the remaining medications in the solution were examined at the maximum absorbance of 357 nm for TCH.

The surface morphology of materials and main surface elements was examined using scanning electron microscope (SEM) images and the energy dispersive X-ray spectroscopy (EDS) data (JSM-6510 LV), respectively.

3. Results and Discussion

3.1. Effect of the goethite proportions on the final composite materials

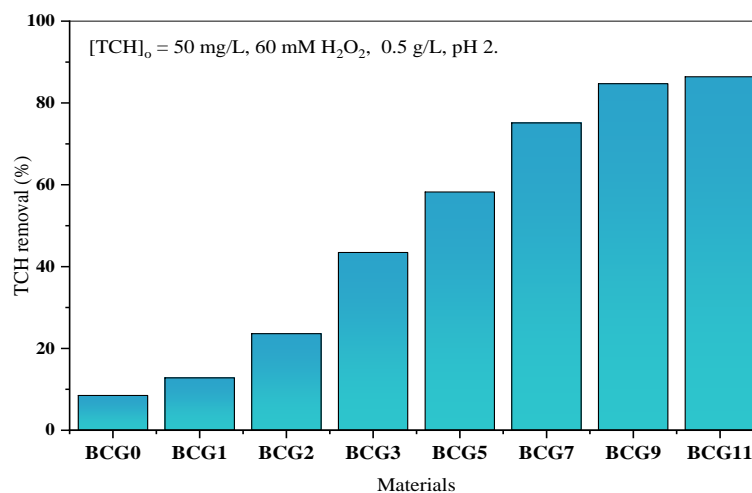


Figure 2. Effect of the goethite proportions on the composite materials

Fig. 2 shows that, under the same reaction conditions, different amounts of iron will result in TCH breakdown efficiency. The initial TCH concentration was 50 mg/L, pH 2, 0.5 g/L, 60 mM H₂O₂, and room temperature. The results indicated that increasing iron content increased TCH removal efficiency. The TCH removal efficiency was in the range of 12.8% to 58.2% when the iron content was changed from 1% to 7%. The loaded iron content was higher than 7%, and the TCH removal efficiency was significantly enhanced by more than 84%. However, adding too much iron (9%–11%) to the material surface did not lead to a noticeable increase in treatment efficiency. The catalytic surface only attaches Fe to the surface with a certain amount, so the increase in Fe content like BCG11 (11%) results in similar data compared to BCG9 (9%) due to the release of iron in BCG11 through the material washing process.

Besides, the TCH removal of BCG0 was 8.5%, the BCG0 material probably is one in which non-goethite loads onto the surface of the biochar. The performance mostly depends on the biochar's effective adsorption capability after being denatured by a hydrothermal procedure. Additionally, Dat et al. have observed that the rate constant of the Fe(III) and H₂O₂ reactions with a graphene catalyst is 100 times higher than that of the process without a graphene catalyst [17].

3.2. Effects on the reaction parameters (pH, contacting time, and dosage catalyst) of TCH

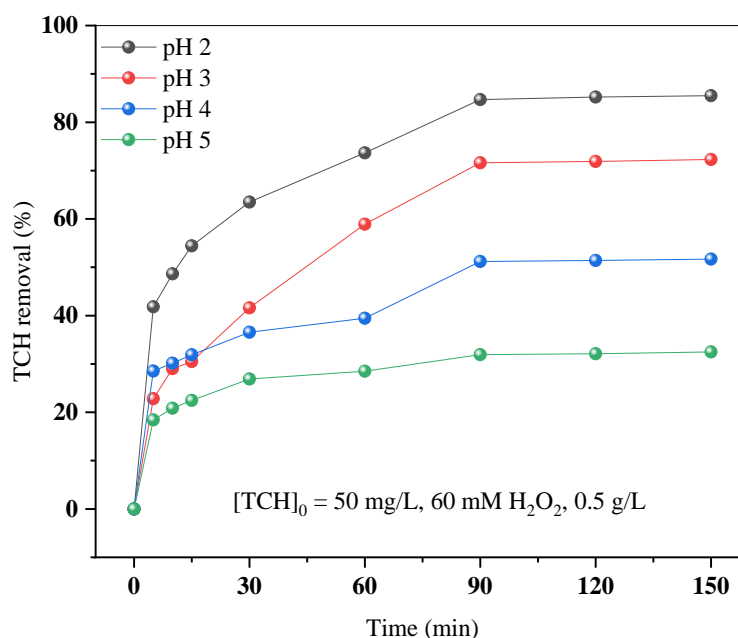


Figure 3. Effect of the solution pH on the TCH degradation

The solution pH is one of the most crucial factors in chemical oxidation processes. This was studied at pH values of 2, 3, 4, and 5 as this is a well-known factor that can remarkably affect the oxidation reaction (Fig. 3). This parameter can influence the activity of the oxidant and the material. Fig. 3 shows the influence of various pH ranges on TCH degradation in a Fenton reaction (BCG9/H₂O₂). As shown in Fig. 3, the TCH removal efficiency decreased from 84.7% to 31.91% when the pH values increased from 2 to 5 at 90 min. After 90 min, the TCH degradation was almost the same until 150 min.

The three acid dissociation constants of TCH (pK_a value) were 3.3, 7.7, and 9.7, respectively. The pH of the solution also affected the solubility rate of the iron components of the BCG catalyst [28]. Hence, the evaluation of the effect of pH in the initial solution varied from 2.0 to 5.0 for the removal of TCH in this study, and the pH variation was recorded in the degradation procedure aimed at better understanding the TCH degradation at different pH values. The decomposition of H₂O₂, the lower oxidation potential of •OH radicals, and the catalyst's deactivation as a result of ferric hydroxide complex formation are the reasons for the reduced degradation rate at higher pH values [28], [29]. On the other hand, at pH 2.0, the high activity of Fe²⁺ can produce OH on the catalyst surface. As a result, the highest degradation performance was observed at pH 2.0. At a pH of 4.0 to 5.0, the reaction of the catalytic (Fe²⁺) and H₂O₂ can produce •OH radicals that are not strong enough to break down TCH,

resulting in a low rate of mutual elimination. According to the findings of our earlier investigation, pH levels of about 2 are optimal for maximizing the generation of $\bullet\text{OH}$ during the Fenton oxidation process [30]. The traditional Fenton process was shown in multiple earlier studies to have strong catalytic activity in acidic environments with pH values between 2 and 4 [31].

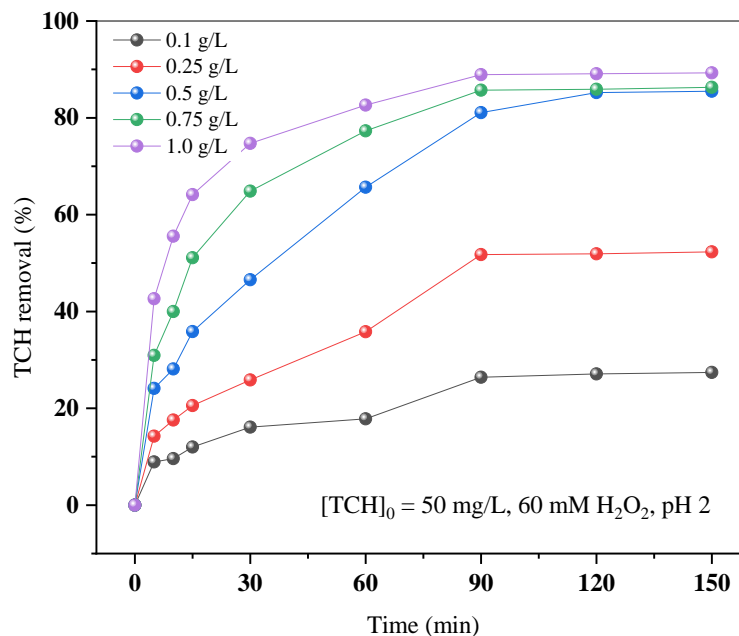


Figure 4. Effect of dosage catalyst on the TCH degradation

The study assessed the impact of different BCG9 dosages (i.e., 0.1 to 1.0 g/L) on the TCH degradation in the Fenton-like system. The initial TCH concentration was 50 mg/L, and the H_2O_2 dosage was 60 mM at pH 2. The removal of TCH with BCG9 and H_2O_2 was demonstrated to be effective in Fig. 4. According to the experimental data, the TCH removal efficiency tended to rise with increased contact times. The maximum removal efficiency in the Fenton oxidation process with a reaction time of 150 min was 26.4% (0.1 g/L); 51.7% (0.25 g/L); 81.1 (0.5 g/L); 85.7 (0.75 g/L); and 88.9% (1.0 g/L). The catalyst dose rose in proportion to the efficiency of TCH decomposition. The rate of degradation increased as a result of the production of higher hydroxide radicals and more active sites when more catalysts were applied [32]. The removal efficiency of TCH notably increased for a catalyst dose of 0.5 to 1.0 g/L. According to the research, a large dose of the catalyst may cause the coagulation reaction, which decreases the concentration of hydroxide radicals and, as a result, reduces the removal effectiveness of TCH [33], [34]. Furthermore, the increased removal effectiveness of TCH in this situation may be due to boosting the degradation of H_2O_2 and raising the number of active sites, which can produce more $\bullet\text{OH}$ radicals. Higher concentrations of heterogeneous catalysts offer more surface area for the increased quantity of iron species needed to produce $\bullet\text{OH}$ radicals, and quenching of $\bullet\text{OH}$ radicals by excess iron ions in the reaction process (Equation 1) [17]. Additionally, a contact time of 90 min and a catalyst dosage of 0.5 g/L were identified as the optimal conditions for the Fenton oxidation process utilizing a heterogeneous BCG9 catalyst.



3.3. Kinetic study of TCH degradation

The BCG9/ H_2O_2 procedure of TCH degradation required numerous steps, and the dynamics of TCH degradation were challenging. Based on prior research, the BCG9/ H_2O_2 system's degradation kinetics of TCH have been examined using two common kinetic mathematical models, the first-order and the second-order models.

$$C_t = C_0 e^{-k_1 t} \quad (2)$$

$$\frac{1}{C_t} = \frac{1}{C_0} + k_2 t \quad (3)$$

where C_0 (mg/L) is the initial concentration of TCH, C_t (mg/L) represents the TCH concentration at reaction time t (min), k_1 (min^{-1}) and k_2 (L/mg.min) are reaction rate constants of the first-order and second-order models, respectively.

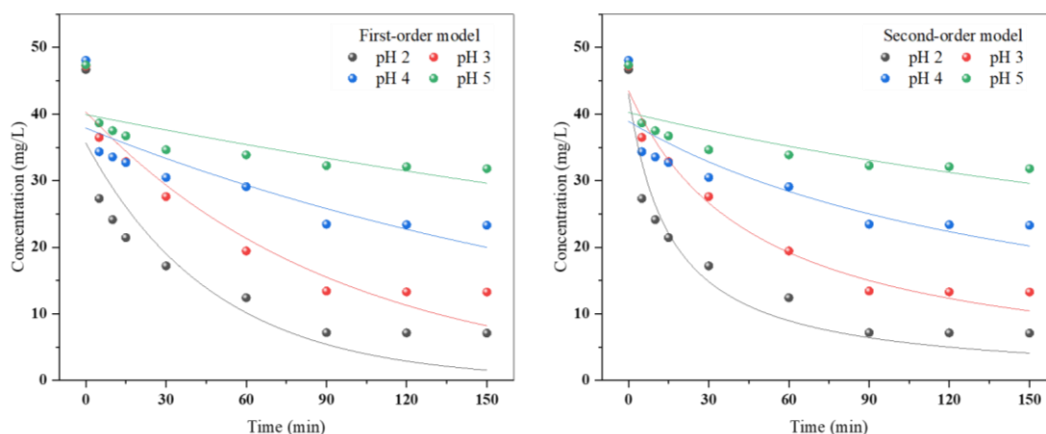


Figure 5. Effect of contact time and modeling fit of TCH degradation on BCG9

The kinetics of TCH degradation at pH 2 were assessed in 150 min and 60 mM H_2O_2 under room temperature conditions. There was a lot of agreement between the generated lines and the experimental data in Fig. 5. However, as shown in Table 1, the second-order kinetic model's correlation coefficients (R^2) tend to be greater than those of the first-order model. Consequently, the best fit for predicting the TCH degradation process by the BCG9/ H_2O_2 system under the reaction conditions is the second-order kinetic model. It is clear that the k_2 values gradually increased from pH = 5 to 2 (0.0002 to 0.0036 L/mol.min) (Table 1). The value of k_2 at pH 2 is three times greater than the one at pH 3. This can be explained by the fact that acidic factors promote the synthesis of $\bullet\text{OH}$, so the value of k_2 increases when the pH decreases [35]. In addition, when compared with other studies with the same condition as pH 2 and the initial concentration of TCH = 50 mg/L, $k_2 = 0.0004$ L/mg.min [35] is lower than those compared to this study. The results supported the previously stated tendency for TCH deterioration [22].

Table 1. Kinetic parameters of first-order and second-order models for the degradation of TCH.

pH	k_1 (min^{-1})	R^2	k_2 (L/mg.min)	k_2 (L/mol.min)	R^2
2	0.027	0.77	0.00159	0.0036	0.92
3	0.014	0.92	0.00054	0.0012	0.96
4	0.006	0.57	0.00022	0.0005	0.62
5	0.003	0.49	0.00010	0.0002	0.52

3.4. Catalyst characterization

The amount of iron on the surface of the BCG9 catalyst was 6.69% based on the EDS analysis (Table 2). There was small difference between the calculated and expected iron contents of the samples because of the high degree of solid hydration found throughout the preparation procedure, which made it difficult to reach the appropriate iron values. Losing iron from the synthesis process (washing material) can also be the reason for lower Fe content on the surface compared to the estimated content. In addition, the reaction for BCG9/ H_2O_2 happened at a pH of 2, and a small quantity of iron from the material was released into the reaction solution.

SEM morphological (Fig. 6) observations demonstrate the surface morphology of three types of materials (Biochar, Goethite, and BCG9 catalyst). The SEM image of BCG9 indicates that the small-sized particles of goethite were coated on the surface of biochar. Fig 6.a, showing clear pores and

specific shapes on the biochar surface. The cause may occur due to the development of internal voids after tar is removed from the biochar by the activating agent method through physicochemical activation (pyrolysis). For images of goethite (Fig 6.b), the synthesized goethite shows agglomerations of needle-like particles, which appear as elongated particles, whose surface is flat, sometimes covered with terraces and straight edges, consistent with crystalline morphology [36]. The results of the catalytic material synthesized from BC and goethite through the hydrothermal process (Fig 6.c) show a similar surface to biochar, but the presence of small needle-like crystals such as goethite present on the surface of the catalytic material. This emphasized that hydrothermal synthesis succeeds in attaching goethite to the surface but the hydrothermal process does not change the surface structure of the catalytic material.

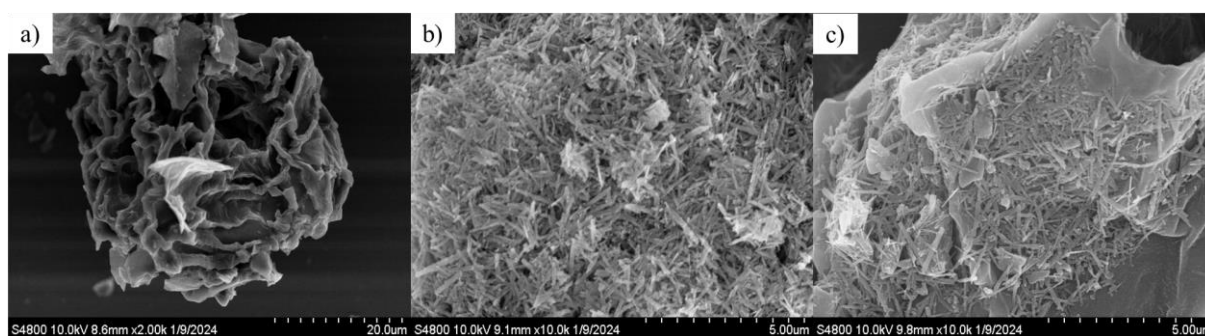


Figure 6. SEM images of (a) Biochar (b) Goethite, and (c) BCG9 catalyst

Table 2. Elemental composition of Biochar and BCG catalyst

Materials	C	O	Mg	Al	P	K	Ca	Fe
Biochar (in wt%)	78.34	16.4	0.5	0.65	0.96	1.87	1.64	-
BCG9 catalyst(in wt%)	67.59	16.8	-	2.17	0.87	2.60	3.31	6.69

4. Conclusions

Based on the results, it was discovered that the degradation processes are involved in the heterogeneous Fenton oxidation of TCH with biochar-loaded goethite. The ratio of Goethite appearing on the biochar surface of the study displayed the optimal result at 9% (for BCG9 material) due to the fulfilled Fe ion on its surface. TCH was virtually eliminated under optimal conditions (pH 2, 60 mM H₂O₂, and 0.5 g/L catalyst dose) after 90 min. According to BCG9 results, TCH can be degraded to 84.7% of its original state in 90 min. Therefore, it can be conclusively stated that biochar-loading goethite can be prepared from food waste to effectively remove TCH using the Fenton process. Moreover, investigating the TCH degradation kinetics of BCG9 material is suitable for the second-order model. This can be seen as the unique characteristic of material synthesized by the hydrothermal method. This study is the basis for future research to focus on the kinetic mechanism of the TCH degradation process. Further research might focus on evaluating the mineralization of TCH by analyzing not only the total organic carbon (TOC) but also intermediate compounds through decomposition from free radicals (\bullet OH) to determine the decomposition ability of TCH with H₂O₂. It is also important to perform the material characteristics; For instance, calculating the total amount of iron lost after each reaction is necessary to assess the economic feasibility and application.

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Conflict of Interest

The authors declare no conflict of interest

Data Availability Statement


The data that support the findings of this study are available from the corresponding author upon reasonable request.

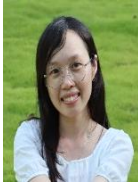
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Huynh Quang Sang: Huynh Quang Sang is currently pursuing a master's degree at Ho Chi Minh City University of Technology and Education, where he is majoring in Environmental Engineering. Set to graduate in 2024, His research interests are focused on innovative and sustainable methods for wastewater treatment. His work delves deeply into the development and application of biosorbents, exploring their potential to effectively remove contaminants from water at Phuc Thien Long Services Trading Joint Stock Company, Ho Chi Minh City, Vietnam. Additionally, he is investigating the Fenton oxidation process, a powerful advanced oxidation technique, to enhance the breakdown of organic pollutants. His expertise also extends to biological processes in wastewater treatment, aiming to harness natural biological mechanisms to treat and purify water. Through his studies and research, He is committed to contributing to the advancement of environmental engineering and promoting cleaner, more sustainable water treatment solutions. Email: huynhquangsang1906@gmail.com. ORCID:  <https://orcid.org/0009-0002-2601-2723>



Doan Thi Kim Quyen: She received her PhD from the National Central University, Taiwan (2023). She is working as a lecturer at Ton Duc Thang University. Her research remit is in converting biomass waste into high-valued add material employed to remediate the environment, such as CO₂ capture, and wastewater treatment. Email: doanthikimquyen@tdtu.edu.vn. ORCID:  <https://orcid.org/0000-0002-9307-5698>



Nguyen Duy Dat: Nguyen Duy Dat has been a dedicated lecturer and researcher at HCMUTE since 2019. His scientific contributions primarily focus on micropollutants, air pollution control, and wastewater treatment. In 2019, he earned his Ph.D. from National Central University (NCU), further solidifying his expertise in these critical environmental fields. Email: datnd@hcmute.edu.vn. ORCID:  <https://orcid.org/0000-0001-5965-2060>