

# CARBON FELT MODIFICATION WITH HNO<sub>3</sub> FOR THE ELECTRO-FENTON PROCESS OF DEGRADATION ORGANIC POLLUTANTS IN THE AQUATIC ENVIRONMENT

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## Abstract

In this study, the authors modified the surface of raw carbon felt (CF) using concentrated HNO<sub>3</sub> for durations ranging from 0 to 12 hours at temperatures between 30°C and 90°C. The structural morphology of both the raw CF and the modified CF was characterized using SEM, EDX, XRD, FT-IR, and contact angle methods. The results showed that the CF modified for 6 hours at 30°C exhibited the highest degradation efficiency for LFX. After 90 minutes of reaction, the degradation efficiency for LFX reached 79.65%. Additionally, the modified CF electrodes demonstrated flexibility and high applicability in degrading various organic pollutants such as oxytetracycline, tartrazine, rhodamine B, methyl orange, and methyl blue, achieving degradation efficiencies of 64.64-97.14% after 45 minutes of reaction. Furthermore, long-term stability tests confirmed its relatively stable performance after 3 cycles.

**Keywords:** *Electro-Fenton; carbon felt; levofloxacin; hydroxyl free radical.*

## 1. Introduction

Organic dyes and antibiotic waste are some of the key substances that can cause environmental pollution due to their widespread use in various industries and modern medicine. Levofloxacin (LFX) is a third-generation synthetic fluoroquinolone antibiotic extensively used to treat severe or life-threatening bacterial infections. However, traditional wastewater treatment processes are ineffective in efficiently removing residual LFX due to its difficult-to-biodegrade biological characteristics. Therefore, it is urgent and necessary to find an effective remediation technique to eliminate LFX from the water environment [1].

Advanced oxidation processes (AOPs) are considered an effective technology for wastewater treatment. The effect arises from the action of hydroxyl radicals ( $\bullet\text{OH}$ ), a

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highly reactive and non-selective oxidizing agent capable of quickly breaking down persistent organic pollutants that are difficult to degrade. Electro-Fenton (EF) technology has garnered widespread attention in recent years among advanced oxidation processes due to its effectiveness in degrading organic pollutants, high mineralization capacity, and relatively low operational costs [2], [3]. In EF technology, the electrochemical reduction of oxygen generates H<sub>2</sub>O<sub>2</sub> on-site, which then reacts with ferrous ions (Fe<sup>2+</sup>) to form •OH radicals (Eq. (1) and (2)). Subsequently, Fe<sup>3+</sup> can be reduced back to Fe<sup>2+</sup> according to Eq. (3). The •OH radicals are strong oxidants that react non-selectively with most pollutants, resulting in the formation of inorganic mineralization products [4], [5].



The efficiency of EF is influenced by several key factors, including catalyst concentration, material of cathode, solution pH, and current density. During the EF process, the cathode is a critical component that determines the mechanism and kinetics of electro-catalysis where Fe<sup>3+</sup> is reduced to Fe<sup>2+</sup> and H<sub>2</sub>O<sub>2</sub> is continuously generated on the cathode surface. The choice of cathode material significantly influences H<sub>2</sub>O<sub>2</sub> formation, with its yield often being determined by the surface properties of the cathode material. Therefore, the kinetics of pollutant mineralization in the electro-Fenton (EF) technology primarily relies on the choice of cathode material [3], [6]. Carbon felt (CF) is commonly used for cathode applications in the electrochemical Fenton process because of its notable benefits, such as good stability, superior electrical performance, large surface area, and affordability. However, due to its poor water wettability, surface activation of CF is necessary to enhance the reduction involving two electrons for H<sub>2</sub>O<sub>2</sub> production [7], [8].

Various chemical agents have been used to modify CF, aiming to enhance its electrocatalytic activity and improve H<sub>2</sub>O<sub>2</sub> production efficiency. The chemical agents consist of an ethanol-hydrazine solution, boiled at 60°C for 6 hours [9], concentrated HNO<sub>3</sub> at 60°C for 6 hours [4], HNO<sub>3</sub>-H<sub>2</sub>SO<sub>4</sub> [10] and HNO<sub>3</sub> and H<sub>2</sub>O<sub>2</sub> [11]. Chemical alteration is a powerful approach to enhancing the electrochemical performance of carbon electrodes by altering the physical, chemical, and surface reactivity characteristics of the electrodes. Oxygen-containing functional groups can increase charge transport and electrocatalytic performance by enhancing the water affinity of the carbon substrate.

In this study, the authors performed surface treatment of CF using a chemical method, employing concentrated  $\text{HNO}_3$  as a modifier under ambient conditions, applied as a cathode for the Fenton reaction to electrolyze and degrade organic pollutants (antibiotics and dyes). The effects of temperature and the time of CF modification have been thoroughly investigated. The morphological structure characteristics were measured using SEM, contact angle, EDX, XRD, and FT-IR methods. The modified CF electrode was utilized in the EF process to degrade a range of organic pollutants, including levofloxacin, oxytetracycline, tartrazine, rhodamine B, methyl orange, and methylene blue.

## **2. Experiments**

### **2.1. Chemicals and reagents**

All reagents were used directly without further purification. All solutions were prepared using double-distilled water. The commercial CF is provided by Hebei Xingshi New Material Technology Co., Ltd. (China). Reagents used in this study included concentrated nitric acid ( $\text{HNO}_3$ , 65-68% purity, analytical grade), iron (II) sulfate ( $\text{FeSO}_4$ , 98% purity, analytical grade), oxytetracycline ( $\text{C}_{22}\text{H}_{24}\text{N}_2\text{O}_9$ , OTC,  $\geq 98\%$  purity), levofloxacin ( $\text{C}_{18}\text{H}_{20}\text{FN}_3\text{O}_4$ , LFX,  $\geq 98\%$  purity, HPLC grade), methylene blue ( $\text{C}_{16}\text{H}_{16}\text{ClN}_3\text{S}$ , MB,  $\geq 98\%$  purity), methyl orange ( $\text{C}_{14}\text{H}_{14}\text{N}_3\text{NaO}_3\text{S}$ , MO,  $\geq 98\%$  purity), rhodamine B ( $\text{C}_{29}\text{H}_{31}\text{ClN}_2\text{O}_3$ , RhB,  $\geq 98\%$  purity), tartrazine ( $\text{C}_{17}\text{H}_9\text{N}_4\text{Na}_3\text{O}_6\text{S}_2$ , TTZ,  $\geq 98\%$  purity), concentrated sulfuric acid ( $\text{H}_2\text{SO}_4$ ,  $\geq 98\%$  purity, analytical grade), sodium hydroxide ( $\text{NaOH}$ ,  $\geq 99\%$  purity, analytical grade), and ethyl alcohol ( $\text{C}_2\text{H}_5\text{OH}$ ,  $\geq 100\%$  purity). All chemicals were provided by Sigma (USA).

### **2.2. Modified carbon felt**

To remove any organic substances attached to the surface, small pieces of commercial CF, measuring  $3\text{ cm} \times 4\text{ cm} \times 0.5\text{ cm}$ , were immersed in acetone for 30 minutes. To remove any residual acetone, the material was rinsed twice using distilled water and then dried for 12 hours at  $80^\circ\text{C}$ . The surface was treated using a chemical approach. Next, the CF electrode underwent a 6-hour treatment with concentrated  $\text{HNO}_3$  (65-68% purity) at room temperature ( $30^\circ\text{C}$ ). Following this, the electrode was rinsed twice with distilled water, then ultrasonically cleaned for 15 minutes each in distilled water, ethanol, acetone, and distilled water again. Finally, the CF electrode underwent a drying process at  $80^\circ\text{C}$  for a duration of 12 hours. The effect of activation temperature ( $30\text{-}90^\circ\text{C}$ ) was also investigated under similar conditions. The samples were labelled as CF $_x$ - $y$  where  $x$  is the modified time in concentrated  $\text{HNO}_3$ ,  $y$  is the temperature of modified process.

### 2.3. Material characterization

Using a Bruker D8 Advance diffractometer, powdered samples were analyzed through X-ray diffraction (XRD) with CuK $\alpha$  radiation ( $\lambda = 1.5406 \text{ \AA}$ ). The analysis was conducted at 40 kV/40 mA voltage/current, with a step size of  $0.02^\circ$  and a step time of one second. To characterize the morphology of the samples, field emission scanning electron microscopy (FE-SEM, JSM-IT 800, Jeol, Japan) was utilized, along with energy dispersive spectroscopy (EDS, Oxford ultimax 65) for elemental analysis. Additionally, a Fourier-transform infrared (FT-IR) spectrometer (Perkin Elmer, USA) was conducted FTIR spectroscopy (FTIR), covering the wavenumber range from 4000 to  $400 \text{ cm}^{-1}$ . A UV-Vis spectrophotometer (Biochrom model SP-60, Biochrom Ltd., UK) was used to analyze the concentrations of organic pollutants at various reaction times, with the measurement taken in 1.0 cm quartz cuvettes.

### 2.4. Degradation experiments

At room temperature ( $30^\circ\text{C} \pm 2$ ), the electro-Fenton (EF) experiments took place in 500 mL glass beakers. The voltage was controlled using a power supply (QJE QJ3005XE, Ningbo Jiuyuan Electronics Co., Ltd., China). The cathode used was a CF modified according to the above process. The titanium mesh anode, coated with platinum, measured 10 cm by 5 cm. The electrodes were positioned 2.0 cm apart with a consistent gap maintained throughout the experiment. A 250 mL solution of a mixture containing Levofloxacin (LFX) at 10 mg/L,  $\text{Fe}^{2+}$  0.1 mM and  $\text{Na}_2\text{SO}_4$  at 50 mM was prepared. A pH of 3.0 was achieved by introducing 2 M  $\text{H}_2\text{SO}_4$ . The solution was stirred at a speed of 500 rpm and aerated continuously for 15 minutes before the EF process was initiated. A current intensity of 24 mA was set. Absorbance measurement at 295 nm was conducted by collecting 3.00 mL of LFX solution every 5 minutes. Other organic contaminants such as OTC, RhB, TTZ, MO, and MB were measured at wavelengths of 354 nm, 554 nm, 428 nm, 464 nm, and 664 nm, respectively.

The process of cathode reuse is carried out by washing the CF several times with double-distilled water and ethyl alcohol to remove degradation products after the electro-Fenton process has concluded, and then drying it at  $80^\circ\text{C}$  for 12 hours. The experiment is repeated under the same conditions as mentioned above. The degradation efficiency was determined using the following expression:

$$H (\%) = [1 - (C_t/C_o)] \times 100 = [1 - (A_t/A_o)] \times 100 \quad (4)$$

where  $C_o$  and  $A_o$  refer to the initial concentration and absorbance of the pollutants at the beginning of the experiment. The terms  $C_t$  and  $A_t$  represent the concentration and absorbance recorded at any subsequent time point. Each experimental set was conducted three times, with the final results presented as the mean values along with their corresponding standard deviations.

### 3. Results and discussions

#### 3.1. Structural characteristics of CF

Figure 1 presents the Scanning Electron Microscopy (SEM) images used to examine the morphological structure of raw carbon felt (CF) and modified CF. Observations from Figs. 1a, b revealed that the surface of the raw CF was relatively smooth and flat, which limited the material's interaction with water. In contrast, Figs. 1c, d demonstrated that following modification with concentrated  $\text{HNO}_3$ , the surface of the activated CF exhibited the increase of roughness and texture. This enhancement provided favorable conditions for the dissolved oxygen reduction reactions, leading to a higher formation of  $\text{H}_2\text{O}_2$  [4].

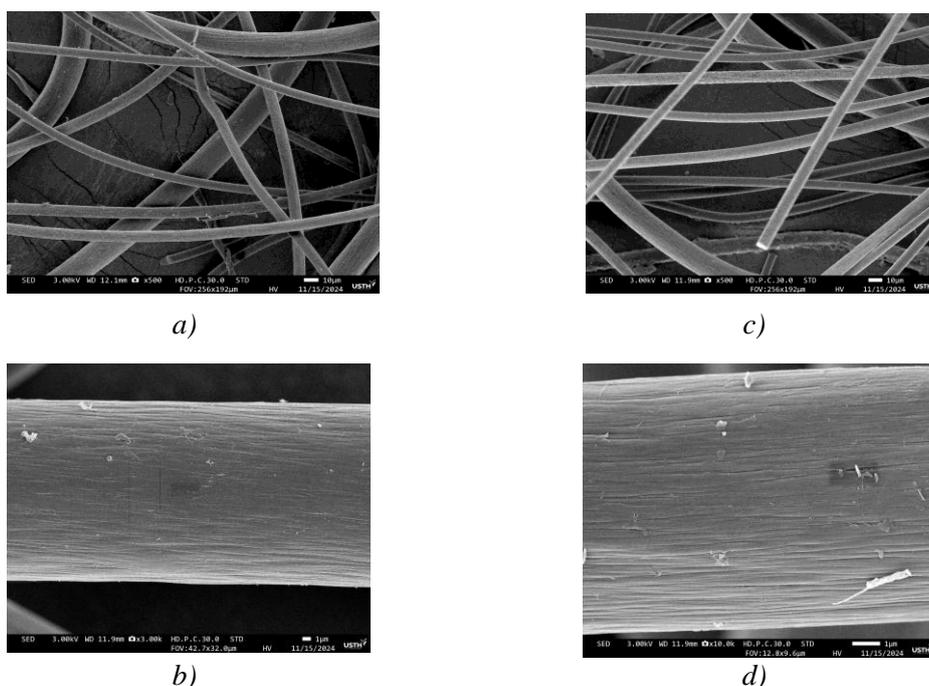


Fig. 1. Surface morphology captured via SEM: (a-b) for raw CF and (c-d) for CF6-30.

The wettability of CF electrodes was assessed through contact angle measurements (Fig. 2). The results showed that the contact angle of CF before activation was relatively high at  $143.1^\circ$ , indicating a low wettability. After activation with concentrated  $\text{HNO}_3$ , the contact angle decreased to  $127.2^\circ$ . The addition of functional groups, such as carboxyl, carbonyl, and hydroxyl groups, to the surface, typically enhances the wettability of CF, resulting in a lower contact angle. These results indicate that the hydrophilic properties of CF have been significantly enhanced through the chemical modification process. Hydrophilicity is a crucial characteristic for

cathodes in the EF process; improved hydrophilicity leads to better diffusion of dissolved oxygen, which in turn increases the production of  $H_2O_2$  [12].

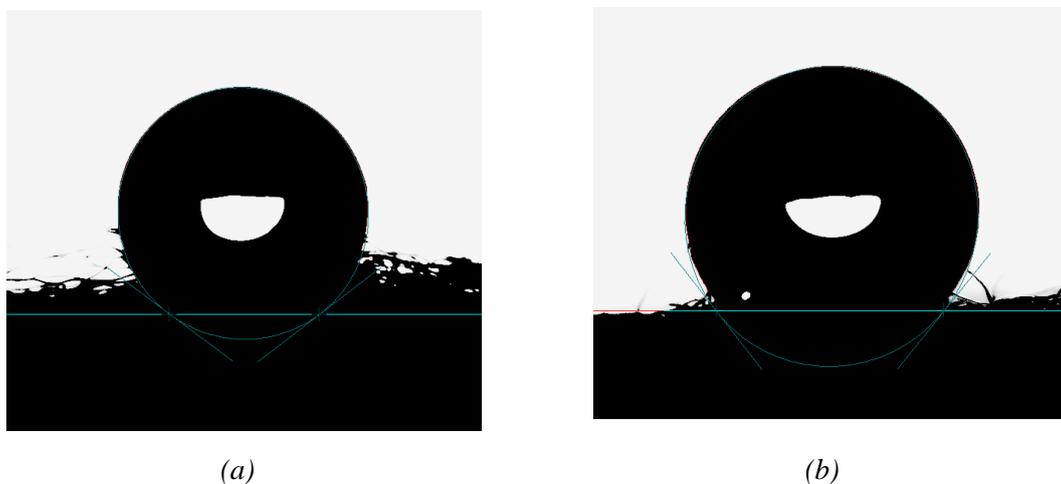


Fig. 2. Contact angles of raw CF (a) and (b) for CF6-30.

The chemical composition of the cathode materials after modification was determined through EDX analysis and is shown in Fig. 3.

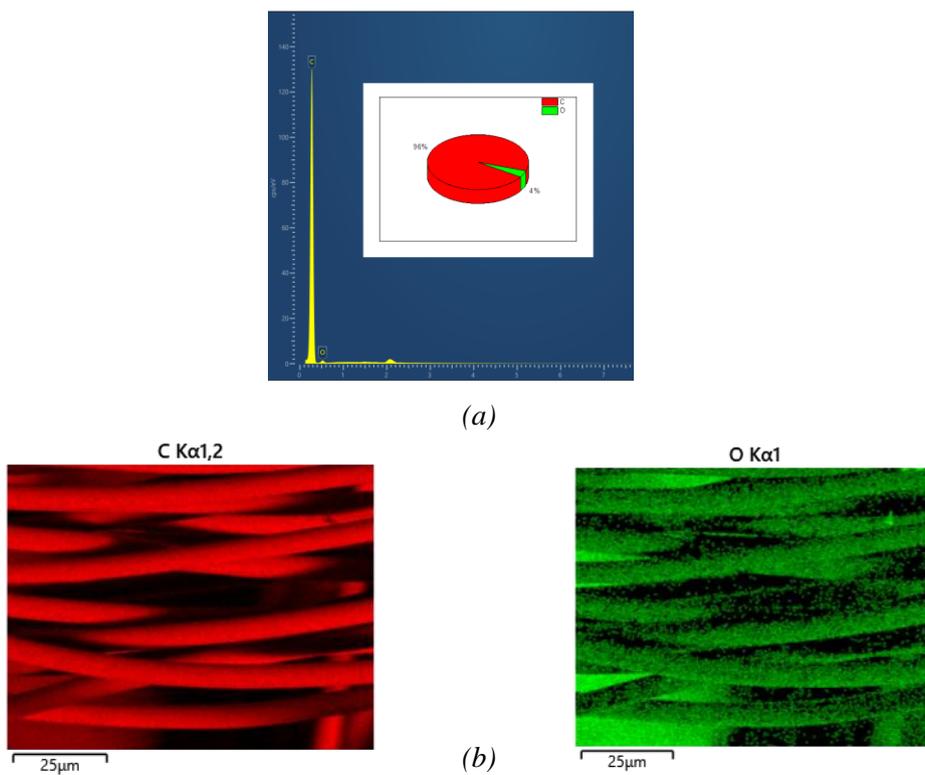


Fig. 3. The EDX spectrum (a) and EDX mapping (b) of CF6-30.

The chemical composition of the cathode materials after modification was determined through EDX analysis (Fig. 3). Analysis showed that CF6-30 consisted mainly of carbon (96%) and oxygen (4%), confirming the presence of oxygen-bearing surface groups, which enhanced electrical conductivity and increased the electrode's hydrophilicity. Additionally, the EDX mapping analysis results showed that these elements were evenly distributed within the electrode composition [13].

X-ray diffraction (XRD) analysis was conducted to investigate the material's crystal structure. In Fig. 4a, at the position of  $2\theta \approx 25$ , the presence of the 002 plane is observed, which was an important indicator of the development of carbon's crystalline structure [14]. This relates to the purity, crystal size, and electrochemical properties of activated carbon (AC), thereby influencing its performance in industrial applications and research. The 002 plane is associated with the layered structure of carbon, particularly in materials with a graphitic structure. It indicates how carbon layers were arranged and the distance between them. The crystal structure affected the movement of electrons within the material, thus influencing its electrical conductivity [15].

In Fig. 4b, FT-IR spectra were measured on raw CF and CF activated at  $30^\circ\text{C}$  for 6 hours. For CF, a weak signal band at  $3458\text{ cm}^{-1}$  corresponding to the -OH group vibration can be observed. After modification, the signals of the functional groups in CF6-30 are more pronounced. In addition to the presence of the -OH group, the peak at  $1632\text{ cm}^{-1}$  suggests a stretching vibration of C=O in -COOH, indicating a significant formation of carboxyl-containing functional groups [16]. Moreover, the peak at  $1379\text{ cm}^{-1}$  also indicates the presence of hydroxyl groups.

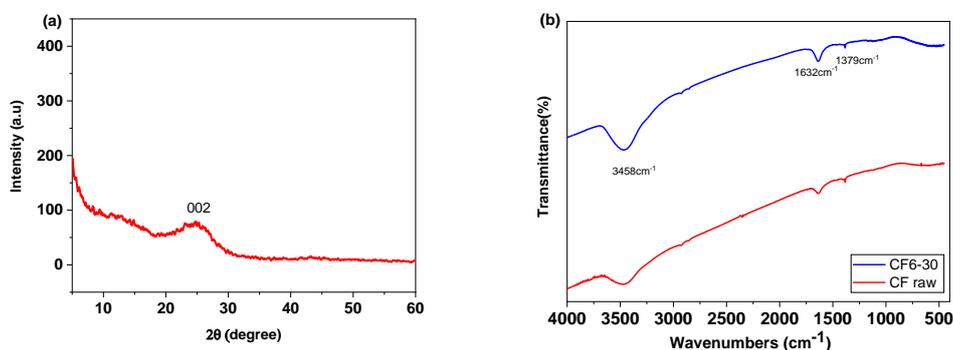


Fig. 4. XRD spectrum (a) and FT-IR of raw CF and activated CF and CF6-30 (b).

### 3.2. Influence of temperature and processing time

The authors conducted a study to investigate the effect of the activation temperature of CF (between  $30^\circ\text{C}$  and  $90^\circ\text{C}$ ) on the degradation capacity of LFX (as shown in Fig. 5a). The results indicated that after 90 minutes of treatment, the

degradation efficiency of LFX decreased in the following order: CF6-30 at 79.65%, CF6-60 at 65.78%, and CF6-90 at 31.64%. This trend can be attributed to the strong oxidizing nature of HNO<sub>3</sub>. As the activation temperature increased, the structure of CF became damaged, leading to a reduced degradation efficiency for LFX.

The effect of CF modification time was also investigated (0-12 hours). The results were presented in Fig. 5b. The findings indicate that as the activation time increased from 0 to 6 hours, the degradation efficiency of LFX rose from 33.37% to 79.65%. The surface of the material might have undergone an increase in additional oxygen-containing groups, leading to an increased formation of H<sub>2</sub>O<sub>2</sub> and hydroxyl radical [4]. However, when the CF activation time was extended from 6 to 12 hours, the LFX degradation efficiency decreased from 79.65% to 47.90%. This decline might be due to the structural degradation of CF with prolonged activation time.

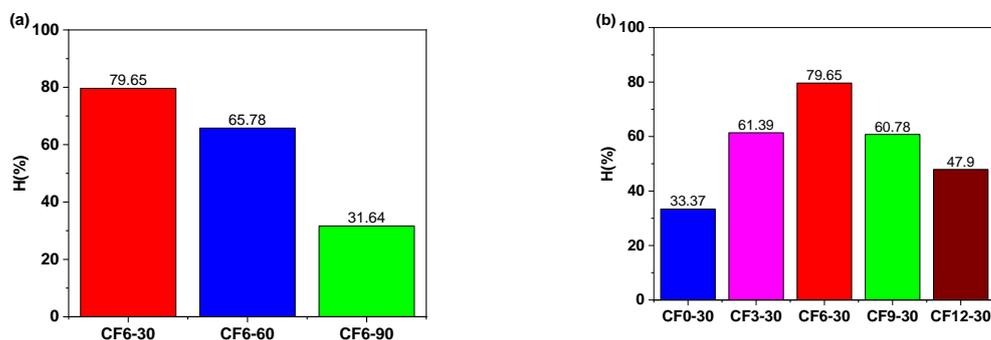


Fig. 5. Influence of modification time and temperature of CF on LFX degradation efficiency.

### 3.3. Application potential of the CF6-30 cathode

In wastewater treatment, the Electro-Fenton (EF) method was widely used to degrade persistent organic pollutants in wastewater. This technique relied on the strong non-selective oxidation capabilities of hydroxyl radicals ( $\bullet\text{OH}$ ), which have an oxidation potential ( $E^\circ$ ) of 2.80 V. These radicals were produced through the decomposition of hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>) and were catalyzed by Fe<sup>2+</sup>, enhancing their oxidative capacity. The potential and efficacy of modified CF in the EF process were evaluated on various pollutants, including antibiotics (LFX and OTC) and dyes (MB, MO, TTZ, and RhB) (Fig. 6). After 45 minutes of reaction, the degradation efficiency of the pollutants was observed to increase in the following order: LFX (64.64%) < OTC (81.07%) < TTZ (92.58%) < RhB (93.49%) < MO (96.53%) < MB (97.14%). After 90 minutes, the degradation percentages for LFX, OTC, and RhB were 79.65%, 88.74%, and 99.1%, respectively. The UV-Vis spectrum of the pollutants over time, shown in Fig. 7, indicated that degradation occurred gradually, as evidenced by a decrease in absorbance

intensity at the maximum wavelength. Overall, the dyes were readily degradable during the EF process, with MO and TTZ - two azo dyes - being quickly dismantled. Remarkably, over 50% degradation of these dyes was achieved within just 5 minutes of reaction. The degradation of the other pollutants was initially slower but accelerated significantly after the first 10 minutes. Modified CF demonstrated both flexibility and effectiveness in degrading various pollutants, and these results align with findings from previous studies [17], [18].

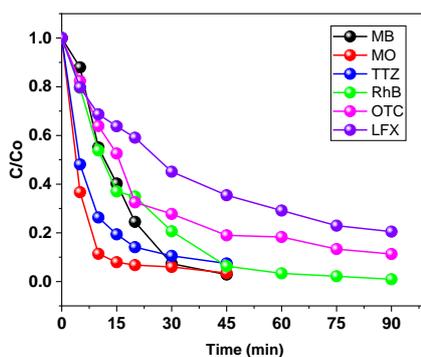
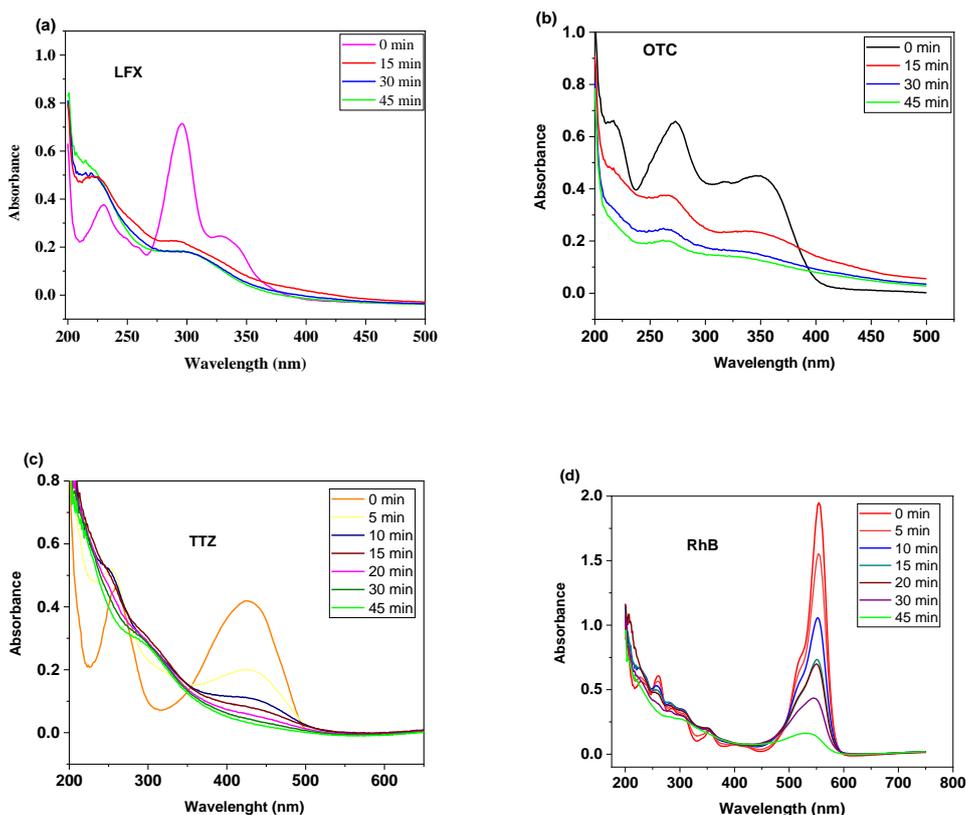


Fig. 6. Degradation of antibiotics and dyes by electro-Fenton process.



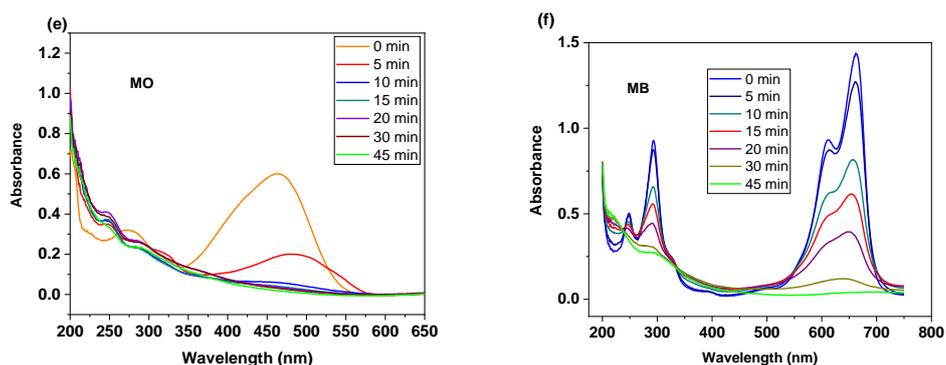


Fig. 7. UV-Vis spectra of pollutants over the reaction time.

(a) LFX, (b) OTC, (c) TTZ, (d) RhB, (e) MO, and (f) MB

The stability of the cathode and its reusability have been tested through 3 consecutive cycles. It is evident that after 3 consecutive uses, the CF6-30 cathode still maintains good performance in removing LFX (Fig. 8). The results indicate that after 3 reuse cycles, the LFX degradation efficiency slightly decreased from 79.65% to 72.23%, demonstrating the high application potential of the cathode in the electro Fenton (EF) process. The effects of activation methods using different activating agents are presented in Tab. 1. The results indicate that our study has the advantage of the CF activation method using  $\text{HNO}_3$ , which occurs under room conditions and achieves relatively high pollutant degradation efficiency.

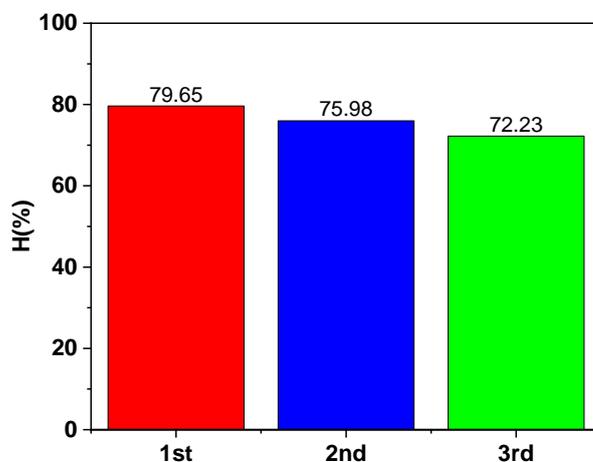


Fig. 8. Stability test of CF6-30 cathode in 3-times continuous.

Tab. 1. Comparison of CF modification efficiency by HNO<sub>3</sub> with other chemical agents

Chemical agent	Polutants	Current intensity/ Voltage	pH	Concentration Fe <sup>2+</sup>	Efficiency	References
HNO <sub>3</sub> 30°C	Levofloxacin (LFX) 10 mg/L	2 mA/cm <sup>2</sup>	3	0.1 mM	79.65% (90 min)	This study
HNO <sub>3</sub> 90°C	Levofloxacin (LEV) 80 mg/L	8 mA/cm <sup>2</sup>	3	0.05 mM	94.85% (60 min)	[4]
Ethanol- hydrazine 60°C	Para nitro phenol (PNP) 50 mg/L	-0.65 V/SCE	3	0.2 mM	78.7% (20 min)	[9]
HNO <sub>3</sub> - H <sub>2</sub> SO <sub>4</sub>	Rhodamine B (RhB) 20 mg/L	-0.7 V/SCE	3	0.2 mM	96% (120 min)	[10]
NaOH 400°C	Oxytetracycline (OTC) 20 mg/L	5.17 mA/cm <sup>2</sup>	3	0.2 mM	83.75% (30 min)	[19]

#### 4. Conclusion

Carbon felt was successfully modified by concentrated HNO<sub>3</sub> at different temperatures and durations. The optimal modification process was performed at 30°C for 6 hours. The CF electrode after modification exhibited roughness with deep grooves, which facilitated the dissolved oxygen reduction reactions, leading to an increased formation of H<sub>2</sub>O<sub>2</sub> and hydroxyl radicals, thereby promoting the breakdown of organic pollutants. The pollutants' degradation efficiency of LFX, OTC, TTZ, RhB, MO, and MB reached 64.64-97.14% after 45 minutes of reaction. Furthermore, the stability of the electrode was tested after 3 consecutive reuse cycles where the degradation efficiency for the target pollutant LFX decreased from 79.65% to 72.23% after 90 minutes of reaction.

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## BIẾN TÍNH CARBON FELT BẰNG HNO<sub>3</sub> CHO QUÁ TRÌNH FENTON ĐIỆN HOÁ PHÂN HỦY CHẤT Ô NHIỄM HỮU CƠ TRONG MÔI TRƯỜNG NƯỚC

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**Tóm tắt:** Trong nghiên cứu này, các tác giả đã tiến hành biến tính bề mặt của carbon felt (CF) bằng cách sử dụng HNO<sub>3</sub> đặc trong thời gian từ 0 đến 12 giờ ở nhiệt độ từ 30°C đến 90°C. Hình thái cấu trúc của cả CF thô và CF biến tính được đặc trưng bằng các phương pháp SEM, EDX mapping, XRD, FT-IR và góc tiếp xúc. Kết quả cho thấy CF biến tính trong 6 giờ ở 30°C (CF6-30) có hiệu suất phân hủy LFX cao nhất. Sau 90 phút phản ứng, hiệu suất phân hủy LFX đạt 79,65%. Hơn nữa, điện cực CF biến tính thể hiện tính linh hoạt và khả năng áp dụng cao trong việc phân hủy nhiều chất ô nhiễm hữu cơ khác nhau như oxytetracycline, tartrazine, rhodamine B, methyl dacam và xanh metylen, đạt được hiệu suất phân hủy từ 64,64% đến 97,14% sau 45 phút phản ứng. Ngoài ra, các thử nghiệm tính ổn định đã xác nhận qua hiệu suất phân hủy LFX ổn định sau 3 chu kỳ tái sử dụng.

**Từ khóa:** Fenton điện hoá; carbon felt; levofloxacin; gốc hydroxyl.

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