

Iron-coated SS316L electrodes for the electrochemical degradation of tetracycline

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ABSTRACT

Tetracycline (TC) is another antibiotic widely used in hospital and livestock farm treatments. Once used, these drugs are excreted from the body along with other waste products. Biological treatment is the preferred system for reducing pollution levels in effluent before it is discharged into the environment. However, the quality of effluent treated by biological systems is still insufficient to remove low concentrations of antibiotic molecules, allowing them to persist and spread into the environment. This research aimed to develop and evaluate the efficiency of an electrochemical wastewater treatment process. This study investigated the effect of Fe-coated anodes on 316L stainless steel (SS316L) surfaces to reduce TC concentration in synthetic wastewater. Two factors were studied: initial TC concentration and electrical potential on TC molecular removal efficiency. The results showed that higher electrical potentials had higher TC molecular removal efficiency. At 24 volts (V) and an initial concentration of 30 mg/L, TC molecular removal reached 83%, followed by 18 V and 12 V with efficiencies of 67% and 50%, respectively. Furthermore, TC molecular removal efficiency was optimal at lower initial concentrations, reaching 70% at 10 mg/L.

Keywords: electrochemical, tetracycline, antibiotic, wastewater.

INTRODUCTION

Antibiotic contamination in the environment from hospital and livestock farm discharge is a global concern due to its threat to the quality of life and ecosystems. TC is a widely used antibiotic that is consumed in large quantities worldwide. Its annual consumption is estimated at approximately 200,000–300,000 tons, of which 70–90% is excreted in a bioactive form (Hou et al., 2015). TC concentrations in natural waters ranged from 0.01–200 µg/L in surface water and as high as 1–100 mg/L in wastewater from healthcare facilities and livestock establishments (Li et al., 2015).

The persistence of TC in wastewater poses multiple risks, including promoting the development of antibiotic resistance in microbial communities, disrupting the balance of aquatic ecosystems, and posing risks to human health through bioaccumulation in the food chain.

According to the report by the World Health Organization (WHO, 2019), antibiotic resistance could lead to up to 10 million deaths per year by 2050 if immediate corrective action is not taken. Several methods for removing TC contaminants from wastewater have been studied, particularly the application of biological wastewater treatment processes. However, biological wastewater treatment technologies are limited in their efficiency in dealing with TC contamination due to its high chemical stability and good water solubility. Matos et al. (2014) found that conventional biological treatment processes can remove TC with efficiencies of less than 30%, which highlights the need for electrochemical advanced oxidation processes (EAOPs) as a superior alternative for pharmaceutical removal. While physical-chemical methods generally achieve efficiencies of only 20–60% (Ganiyu and Martinez-Huitle, 2020). The application of advanced wastewater treatment processes, such as membrane

remediation (MBR) technology only achieves 45–65% elimination efficiency in removing TC from wastewater (Wang et al., 2020). Furthermore, most biological wastewater treatment technologies have several significant drawbacks, including high operating costs, residual pollution, and the inability to remove low concentrations. EAOPs are one application of this technology for removing pollutants from wastewater. This system can destroy antibiotic molecules by generating hydroxyl radicals ($\bullet\text{OH}$), leading to high oxidation and reducing the concentration of the toxic substance (Wang and Higgins, 2024; Sirés et al., 2014). The electrochemical wastewater treatment process using a boron-doped diamond anode (BDD) has been shown to remove over 90% of TC molecules (Brinzila et al., 2014; Chi et al., 2021). Advantages of the electrochemical wastewater treatment process include the elimination of the need for additional chemicals and precise process control. In addition, it can function under various environmental conditions (IWA, 2023). SS316L has been developed and applied as a cathode in electrochemical processes due to its attractive properties, such as corrosion resistance, availability, and surface treatment (Dudek and Lisiecka, 2018; Kumaran et al., 2021; Aliyu et al., 2023; Singh and Pandey, 2024). Fe, a component of SS316L, is highly effective in oxidizing and decomposing organic contaminants (Xu et al., 2013; Pan et al., 2019; Gao et al., 2012). Electrochemical methods can generate suitable surfaces, enhancing oxidation reactions (Liu et al., 2010). Surface modification of SS316L with an Fe coating can increase the specific surface area and enhance surface roughness, resulting in a significant improvement in catalytic efficiency (Weber et al., 2023; Gülsoy et al., 2015). Although research has been conducted on modification for contaminant removal, system research on SS316L surface modification with Fe and efficiency enhancement is still lacking.

The research challenges involved enhancing the ability to break down or reduce drug residues from treatment processes using electrochemical processes that apply microwaves to modify metal surfaces to improve the catalytic efficiency of antibiotic degradation in water.

Therefore, this study aimed to develop and evaluate the efficiency of Fe-coated SS316L through electrochemical modification for the removal of TC from synthetic wastewater and to investigate the effects of key operational variables

on removal efficiency and degradation kinetics for the design of an effective and sustainable wastewater treatment system.

MATERIALS AND METHODS

Study design

This study employed a controlled experimental design to evaluate the performance of Fe-modified SS316L electrodes in the electrochemical degradation of TC. The experiments were designed to examine the effects of applied electric potentials (12, 18, 24 V) and initial TC concentrations (10, 20, 30 mg/L).

Preparation and Fe-SS316L–modified electrodes

Preparation of Fe-SS316L

The support material used was SS316L, supplied by Metha Metal LP Co., Ltd., Thailand, with a chemical composition of Fe 70–75%, Cr 18%, Ni 8%, and Mo 2.0–2.5%. The samples were cut to dimensions of $5 \times 15 \times 0.1$ cm using a hydraulic shearing machine and underwent a standard surface preparation process. The surface preparation procedure for SS316L before Fe coating begins with washing with 0.5 M acetone (99.5% purity, Sigma-Aldrich) for 10 minutes, followed by washing with 0.1 M methanol (99.8% purity, Merck) for 10 minutes, and a final wash with 0.1 M isopropanol (99.7% purity, Fisher Scientific) for 10 minutes to remove any residual organic matter and grease from the sample surface. Afterward, the sample is dried at room temperature, then oven-dried at 120 °C for 12 hours under normal atmosphere to remove moisture and prepare the surface for the subsequent coating process.

Synthesis of Fe coating layer

Fe coating on the SS316L surface was performed using a coating solution prepared by dissolving 0.5 grams of ferric nitrate ($\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$, 99.5% purity, Alfa Aesar) in 1 M HNO_3 (HNO_3 , 65% concentration, Fisher Scientific) and adding deionized water (18.2 M Ω ·cm, Millipore system) to a total volume of 1.000 mL. The solution was prepared in a fume hood under controlled conditions. The Fe coating process on the SS316L surface involves immersing a

surface-prepared sample in a solution for 6 hours, followed by drying for at least 2 hours. The heat treatment was carried out in two consecutive steps to enhance adhesion and the formation of a stable Fe layer on the surface of the substrate. The high-temperature heat-coating process is performed with controlled temperature at 600 °C for 2 hours. Afterward, the sample is allowed to cool to room temperature. The coated sample is then microwaved at 700 watts for 15 minutes. The sample is then coated three times, washed with deionized water, and dried before being used as an anode in the electrochemical process.

Electrochemical experiment

Preparation of TC synthesis

TC (CAS: 64-75-5, MW 480.90 g/mol, purity $\geq 95\%$, Sisco Research Laboratories Pvt. Ltd.) was used as a representative TC in this study. A stock solution of 1,000 mg/L was prepared by dissolving it in deionized water and stored at 4 °C in the dark to prevent photodegradation. Working solutions of 10, 20, and 30 mg/L were prepared by diluting the stock solution with deionized water on the day of use. The pH of the TC solution was adjusted to 7.0 ± 0.1 using NaOH (0.1 M) or HCl (0.1 M) and measured with a pH meter (Model: SevenEasy pH, Mettler Toledo). During the electrochemical treatment process, 10 mL water samples were collected at predetermined time intervals (0, 10, 20, 30, 40, 50, and 60 minutes). The physical and chemical properties of the tetracycline used in this study are summarized in Table 1.

Electrochemical system setup

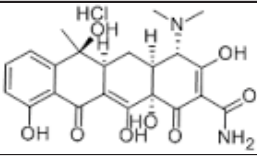
Electrochemical degradation of TC molecules was carried out in a two-electrode electrochemical cell inside a 1.000 mL glass reactor. The working electrode (anode) was Fe-coated SS316L, and the

counter electrode (cathode) was a $5 \times 15 \times 0.1$ cm. Ru-Ir-coated titanium mesh (Baoji Co., Ltd., China), with a chlorination potential <1.13 V and a lifetime $>3,000$ minutes at a current density of 1.000 A/m². The positive (Fe-coated SS316L) and negative (Titanium mesh) terminals are spaced 0.3 cm apart to minimize short circuits and facilitate power supply from the GPR-6060D (GW Instek) source. The experimental procedure began with preparing synthetic wastewater at a TC concentration of 200 mL (according to the experimental tank volume) under specified conditions. The pH was adjusted according to the parameters of each experimental factor. The two electrodes were then installed in their designated positions by connecting the electroplated titanium mesh plate to the negative electrode and the Fe-coated SS316L plate to the positive electrode. These were then placed in the reaction tank and connected to the power supply. During the experiment, the TC solution was stirred at 150 rpm using a magnetic stirrer to promote mass transfer between the solution and the electrode surfaces. Once the system was set up, an electrical potential was applied, and the timer was started. 2 mL of solution samples were collected at 0, 10, 20, 30, 40, 50, and 60 minutes using a syringe, and filtered through a 0.45 μ m glass fiber filter. At the end of the experiment, the power supply was turned off, the electrodes were removed from the solution, and the electrodes were rinsed with deionized water. During the experiment, key parameters including solution temperature, pH, current, and voltage were monitored and recorded every 10 minutes.

Characterization of coated stainless steel

The physical and structural analyses of materials were performed using a variety of techniques. For crystal structure analysis, XRD (XRD-6100 model, SHIMADZU) was used to identify the crystalline phase and structural composition of

Table 1. Characteristic of the TC hydrochloride used in this study

Chemical structure: 38614 TC Hydrochloride (TC), 95%	
Chemical formula:	$C_{22}H_{24}N_2O_8 \cdot HCl$
CAS number:	64-75-5
Molecular weight:	480.90
Company:	Sisco Research Laboratories Pvt. Ltd., Maharashtra, India

the material. For the determination of TC concentration in solution, a UV–visible spectrophotometer, Evolution 201 model (Thermo Scientific) was used, with the absorbance measured at the maximum wavelength ($\lambda_{\max}$) of 355 nm.

Calculation of tetracycline removal efficiency

The treatment efficiency of TC in the synthesis wastewater was evaluated to measure the efficiency of the electrodes under different operating conditions. The treatment efficiency was calculated using Equation 1 as follows:

$$\text{Treatment efficiency (\%)} = [(C_o - C_e)/C_o] \times 100(1)$$

where: C_o – TC concentration in wastewater before treatment (mg/L); C_e – TC concentration in wastewater after treatment (mg/L).

RESULTS AND DISCUSSION

Morphological changes of SS316L surface

Figure 1 shows the appearance of stainless steel. The surface morphology analysis shows significant changes in SS316L electrodes after Fe modification. The originally smooth and shiny surface was transformed into a highly rough, rust-colored surface, reflecting the successful coating and formation of an Fe layer on the support material. The increased roughness indicates a larger specific surface area, which is an important factor for electrocatalytic efficiency because it increases the number of active sites for the adsorption and oxidation of TC, consistent with the report by Weber et al. (2023). The obvious color change from silver to rust confirms the formation of the Fe phase with uniform distribution over the surface, indicating a dense coating that adheres tightly to SS316L with no uncoated areas. Figure 2 shows the image of a titanium mesh used as the anode, with dimensions of $5 \times 15 \times 0.1$ cm, coated with a ruthenium-iridium alloy. The anode was manufactured by Baoji Co., Ltd., China.

Characteristics of Fe-coated SS316L

The structural characterization of the stainless-steel surface coated with Fe through the surface modification process by XRD analysis revealed significant structural changes while maintaining

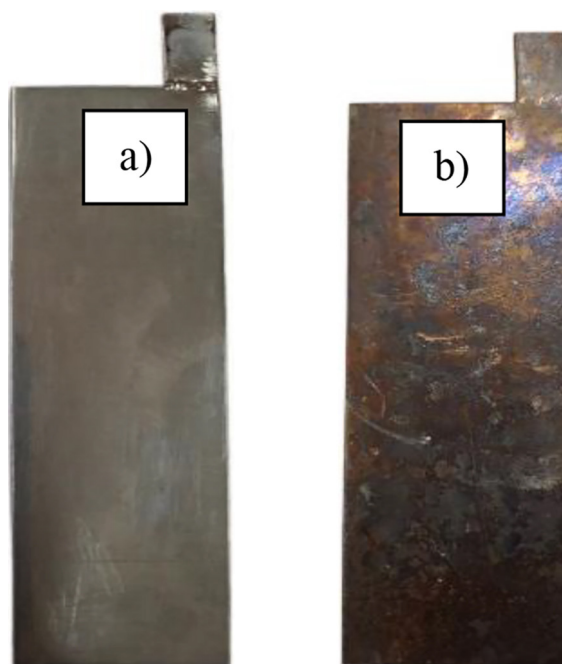


Figure 1. Images of stainless-steel samples before coating (a) and after coating (b) and calcination at 600 °C for 3 hour



Figure 2. Image of electroplated titanium mesh (Baoji Co., Ltd., China)

the integrity of the support material. XRD analysis of the SS316L sample after surface modification with Fe showed that the synthesis method can clearly form an Fe coating. For the uncoated SS316L sample (Figure 3a,3b), sharp and high-intensity diffraction peaks were observed, consistent with the austenitic structure of stainless-steel grade 316L. XRD analysis of the surface area of SS316L before (Figure 3a) and after ferric nitrate coating (Figure 3b) revealed peaks in the 12–14° range in both samples. However, the peaks on the ferric nitrate-coated SS316L were wider and more intense than those on the regular SS316L.

Effects of the initial TC concentration

A study on the effect of initial TC concentration on TC removal efficiency in synthetic wastewater. Experimental conditions were controlled using different initial concentrations: 10, 20, and 30 mg/L, with a constant voltage of 6.0 Volts and 293.15 K in all studies. Samples were collected at: 0, 10, 20, 30, 40, 50, and 60 minutes to evaluate TC removal efficiency at each time interval. All experiments were conducted in a batch system. The results showed that the initial concentration of TC significantly affected removal efficiency. The lowest concentration of 10 mg/L achieved a maximum removal of approximately 70%, while concentrations of 20 mg/L and 30 mg/L decreased the efficiency to approximately 60% and 50%, respectively. These results indicate that the electrochemical process can remove TC more efficiently when the contaminant concentration is at a low level (Figures 4 and 5). This finding is consistent with the research of Chi et al. (2019), who found that using a Ti-anode for TC degradation provides higher removal efficiency at lower TC

concentrations, as TC molecules can fully access the active sites on the anode surface. Liu et al. (2020) reported that the removal rate decreases as the initial TC concentration increases because TC molecules compete for access to reactive species, such as hydroxyl radicals ($\bullet\text{OH}$). Brinzila et al. (2014) found that the electrochemical degradation of TC is negatively correlated with the initial concentration. That is, lower concentrations yield higher degradation because oxidized species in EAOPs can fully react with TC. Wang et al. (2020) supported this idea, pointing out that the reaction rate of hydroxyl radicals with contaminants is negatively correlated with the initial concentration (Table 2).

Effects of voltage

This research aimed to study the degradation efficiency of TC using an electrochemical process under an initial TC concentration of 30 mg/L. The experimental variables were controlled by varying the electrical potential levels to three different levels: 12, 18, and 24 volts. The temperature was kept constant at 293.15 K, and the initial TC concentration was 30 mg/L. The results showed that increasing the electrical potential significantly reduced the TC concentration, from 30 mg/L to 5 mg/L, with a maximum removal efficiency of 83%. While the 18-volt potential reduced the concentration from 30 mg/L to 10 mg/L with an efficiency of 67%, the 12-volt potential reduced it from 30 mg/L to 15 mg/L with an efficiency of only 50%. The experimental results indicate that increasing the electrical potential results in greater electrical current and oxidizing agents that degrade TC molecules more effectively. Similarly, Chi

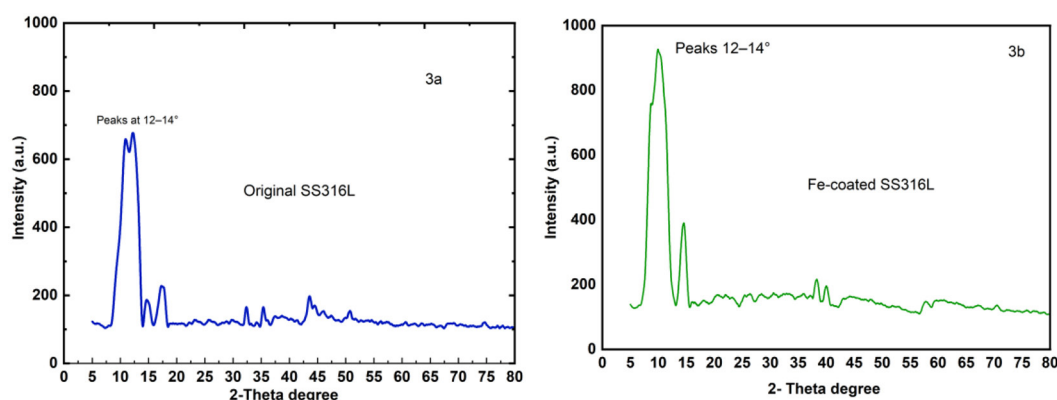
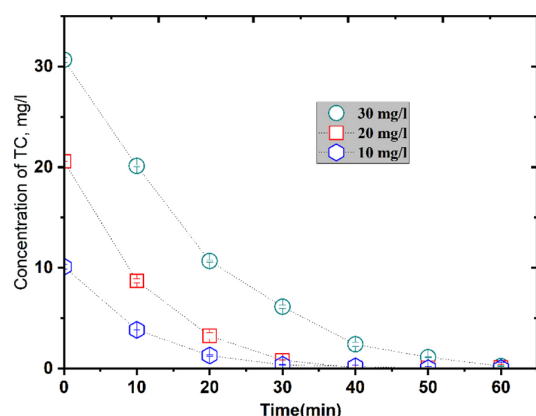
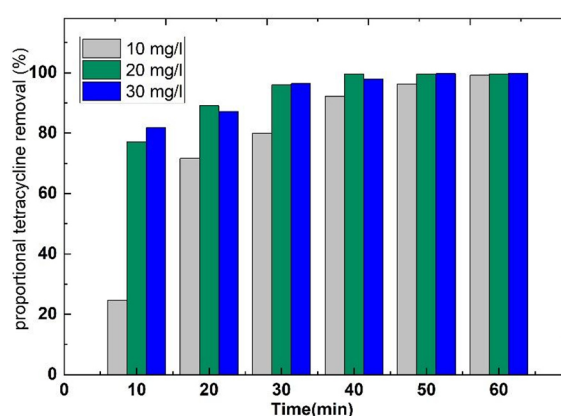


Figure 3. XRD patterns of uncoated (3a) and Fe-coated (3b) SS316L

Table 2. The residual concentrations of TC in synthetic wastewater under 12 volts

Time (min)	TC Conc.		
	30 mg/l TC	20 mg/l TC	10 mg/l TC
0	30.66±0.25	20.57±0.01	10.10±0.23
10	20.11±0.10	8.71±0.21	3.84±0.02
20	10.68±0.12	3.24±0.32	1.30±0.08
30	6.13±0.16	0.83±0.03	0.36±0.04
40	2.40±0.22	0.07±0.02	0.20±0.12
50	1.12±0.03	0.07±0.01	0.03±0.17
60	0.23±0.02	0.10±0.31	0.02±0.58

**Figure 4.** Reduction of TC under different initial concentrations of 10, 20, and 30 mg/L**Figure 5.** Percentage of TC removal under different initial concentrations of 10, 20, and 30 mg/L**Table 3.** The residual concentrations of TC in synthetic wastewater at different electrical currents

Time (min)	TC Concentration under various electrical currents		
	12 Volt	18 Volt	24 Volt
0	30.66±0.25	30.66±0.35	30.66±0.39
10	17.93±0.04	14.06±0.01	11.03±0.06
20	9.72±0.01	7.63±0.09	2.99±0.04
30	3.67±0.05	1.56±0.13	0.26±0.05
40	1.15±0.21	0.07±0.07	0.22±0.02
50	0.38±0.12	0.13±0.18	0.07±0.03
60	0.07±0.01	0.12±0.07	0.05±0.02

et al. (2018) found that a high electric potential on the anode promotes better TC degradation. The characteristics of the TC removal rate exhibited a two-phase trend. In the first phase (0–30 minutes), the concentration decreased rapidly due to the availability of oxidant species. In the second phase (30–60 minutes), the reduction was slower and more stable due to

the decreasing contaminant concentration. This pattern is consistent with the studies of Sirés et al. (2014) and Oturan and Aaron (2014) on TC degradation by electrochemical advanced oxidation processes. Thus, the treatment in the first 30 minutes is both sufficient and efficient in terms of removal rate and energy consumption (Table 3, Figures 6–7).

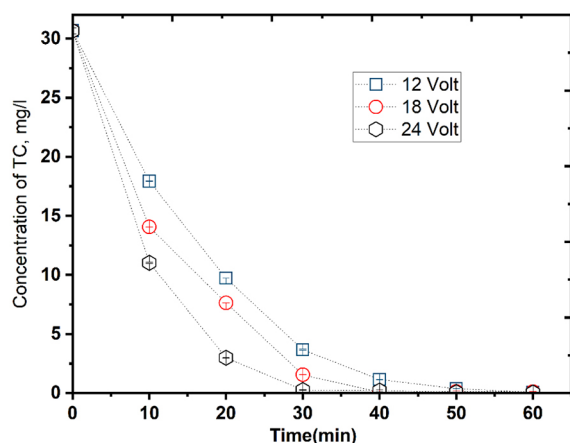


Figure 6. Reduction of TC under different voltages of 12, 18, and 24 V

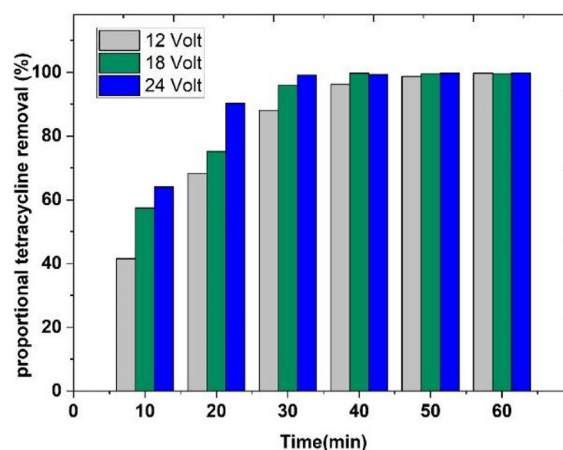


Figure 7. Percentage of TC removal under different voltages of 12, 18, and 24 V

CONCLUSIONS

This research aimed to study the development of SS316L electrodes modified with Fe for degrading tetracycline (TC) in electrochemical wastewater treatment processes. The electrodes were coated with Fe and calcined at 600 °C to form a stable Fe layer without damaging the structure of the support material. Analysis of the Fe molecular coating on the SS316L surface using XRD revealed peak formation at the same locations before and after coating. However, differences in peak height and width were observed in the samples coated with ferric nitrate. Furthermore, high TC removal efficiency was achieved with a low initial concentration (10 mg/L of TC), reaching 70%, and with a higher voltage (24 volts), an efficiency of 83% was attained under the same study time and conditions. From these results, it can be concluded that coating the SS316L surface with ferric nitrate enhances the electrochemical oxidation process for TC degradation. This can be adapted for use in wastewater treatment to reduce the spread of antibiotics in the environment.

However, further studies are recommended to comprehensively evaluate the practical applicability of the proposed electrode system. Future research should include electrical energy consumption assessments, kinetic modeling of tetracycline degradation reactions, and advanced material characterization techniques, such as SEM, EDS, and XPS to better understand surface morphology, elemental distribution, and reaction mechanisms. These investigations would provide a more comprehensive understanding of the performance, sustainability, and scalability of the Fe-coated SS6L electrode system for real-world wastewater treatment applications.

Acknowledgment

Kasetsart University Research and Development Institute, Suwan Vajokkasikij Building, 50 Ngam Wong Wan Rd, Lat Yao Chatuchak, Bangkok 10900.

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