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Ampicillin Removal in Situ Oxidation Using a Double-layer-cathode Trickle Bed Electrochemical Reactor (TBER)

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Abstract

Ampicillin (AMP) is extensively used as an antibiotic in hospitals and veterinary clinics worldwide to combat bacterial infections. As a result, residues of this antibiotic are commonly identified in the wastewater released by these facilities. This study introduces an advanced oxidation process for efficiently removing ampicillin from wastewater using a double-layer-cathode trickle-bed electrochemical reactor (TBER). This system electro produces environmentally friendly hydrogen peroxide (H_2O_2) through oxygen reduction, which is then used to oxidize ampicillin in wastewater. The TBER features a high-porosity electrode (C-PTFE-SSM) with optimized operating parameters. The effectiveness of this electrochemical process was evaluated within a TBER measuring 120 mm wide, 110 mm high, and 40 mm deep. equipped with a stainless steel anode and a gas diffusion cathode made of carbon black polytetrafluoroethylene The evaluation focused on the removal of antibiotics, specifically ampicillin (AMP), using applied voltages ranging from 1 to 3 volts, electrolyte concentrations 0.5 M, electrolyte flow rates 25 to 250 mL/min, oxygen flow rates 2 to10 L/min, and ampicillin concentrations 100 ppm. The reaction temperature maintained constant at15°C and duration necessary for achieving maximum removal by electrochemical process within the TBER. The results indicate that the in-situ oxidation process conducted in the customized reactor holds significant potential for improving ampicillin elimination. Under optimal conditions, including a potential of 1 V, an Electrolyte concentration 0.5M H_2SO_4, electrolyte flow 200ml/min, oxygen flow 9L/min, ampicillin concentration 100ppm, reaction temperature 15°C, and a reaction time of 40 minutes, the highest achieved ampicillin removal rate was an impressive 93.6%. Under the ideal conditions.

Keywords: In-situ oxidation; TBER; Hydrogen peroxide; Ampicillin; Antibiotic degradation; Stainless steel anode

1- Introduction

Wastewater treatment plants (WWTPs) are pivotal in both receiving and releasing antibiotics into the environment, [1, 2] with a diverse array of detectable antibiotic types. Antibiotics present in WWTPs encompass β-lactams, tetracyclines [3] sulfonamides, Predominant [4]. and quinolones. The influents of municipal wastewater treatment plants typically contain antibiotic concentrations ranging from 1 ng/L to over 1 mg/L, as reported by [5] and [6]. However,

industrial wastewater, particularly from animal and pharmaceutical sources, tends to have higher concentrations, reaching up to 100 mg/L, as noted by [7]. Additionally, antibiotics have the potential to affect the behavior, reproduction, [8] this alarming situation has resulted in a staggering 4.95 million deaths in 2019, according to [9]. It is worth mentioning that antibiotics are widely prescribed as a broad-spectrum medication for treating bacterial infections, including gonorrhea, urinary tract infections, and ear, nose, and throat infections, as stated by [10-12]. The concentration of ampicillin in municipal and hospital wastewater usually falls within the micrograms per liter (μg/L) range, whereas pharmaceutical wastewater contains higher concentrations, typically in the milligrams per liter (mg/L) range. The impact of antibiotic addition on the performance of EAOPs (Electrochemical Oxidation Process). There is a wealth of research and literature available on the "electrochemical oxidizing method," encompassing studies and publications that delve into its principles, applications, and outcomes. Researchers investigate various aspects of electrochemical oxidation, including material deposition, surface modification, and wastewater treatment, with the primary aim of comprehending and optimizing this method for specific industrial purposes. In their study [13], Vidal and their team investigated Ampicillin (AMP) degradation and removal of its 34 antimicrobial properties. They tested different electrochemical advanced oxidation processes (EAOPs), such as electrooxidation with hydrogen peroxide (EO-H2O2), electro-Fenton (EF), and photo electro-Fenton (PEF). PEF showed remarkable efficiency, achieving significant degradation, mineralization, and complete elimination of antimicrobial activity in 120 minutes. This was due to rapid hydroxyl radicals (•OH) generation facilitated by UV radiation and direct photolysis of Fe^{3+}-organic intermediates. The study identified byproducts and intermediates, including inorganic ions, carboxylic acids, and aromatic compounds, using photometric and chromatographic techniques. The research also proposed an oxidation pathway for converting Ampicillin into CO2.. [14] used a boron-doped diamond (BDD) reactor to study ampicillin's electrochemical oxidation, finding that higher electrolyte concentration and current density improved ampicillin and COD removal. They introduced "electrochemical oxidation byproducts and intermediates (EOX)" and identified optimal conditions for 92.5% ampicillin removal, 71.7 kWh/kg COD removal, and 97.1% energy consumption. The study suggests BDD electrodes effectively treat pharmaceutical residues. [15] studied SS electrode shapes' effect on the electrochemical treatment of pharmaceutical pollutants. They coated SS plates and grids with PbO2 using various deposition methods. Their analysis revealed improved ampicillin degradation, reaching 100% removal after 120 minutes using SS/PbO2 electrodes.

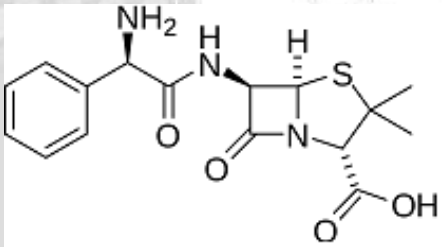
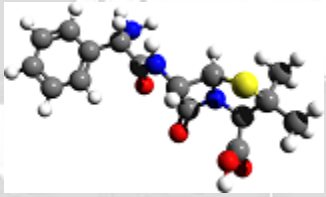
In this research, we have introduced an advanced in-situ oxidation method within a specially designed TBER to address the degradation of antibiotics, with a focus on ampicillin. The TBER is uniquely equipped with a stainless steel anode and a gas diffusion cathode made of carbon black polytetrafluoroethylene affixed to a stainless steel mesh, allowing on-site production of hydrogen peroxide for antibiotic oxidation. Our evaluation encompassed a range of operational parameters, such as applied voltages, electrolyte flow rates, oxygen flow rates, and the duration necessary for achieving maximum removal.

2-Materials and Methods

2-1 Chemicals and Analytical Procedure

The study utilized Ampicillin antibiotic with a purity of 99.99%. Details of its properties can be found in Table 1[16] The supporting electrolyte for the electrochemical process was pure sulfuric acid (H_2SO_4) with a molecular weight of 98.08 g/mol.To investigate the degradation of Ampicillin through electrochemical oxidation, a UV-VIS spectrophotometer was employed. The spectrophotometer operated at a wavelength of 350 nm, allowing for precise measurement and analysis during the electrochemical process.

Table 1: Properties of Ampicillin. [15]

Ampicillin	Properties
Molecular formula	$C_{16}H_{19}N_3O_4S$
2D structure	
3D structure	
Molecule weight (g/mol)	349.4
Physical shape	crystallized powder
Purity	97%
pH	3.5-5.5
Appearance	powder from white to bright yellow
Melting point (°C)	82-83
Storage temperature (°C)	2-8
Maximum wavelength (nm)	339

Ampicillin is marketed in three different forms: anhydrous, trihydrate, and salt. [17]

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Table 2: Commercially ampicillin is available in 3 forms: Ampicillin anhydrous, Ampicillin trihydrate and Ampicillin sodium salt.

Property	Ampicillin anhydrous	Ampicillin trihydrate	Ampicillin sodium salt
CAS Number	69-53-4	7177-48-2	69-52-3
Molecular formula	$C_{16}H_{19}N_3O_4$	$C_{16}H_{19}N_3O_4S.3H_2O$	$C_{16}H_{18}N_3O_4Na$
Molecular weight	349.4	403.4	371.4

2-2 Experimental Procedure and Setup

2-2-1 Electrochemical System

The electrochemical experimental setup involved a TBER figure 1, consisting of various components, including an anode and a cathode separated by a Cilgard membrane to prevent circuit breakage. Support was provided by two stainless steel plates, with two stainless steel nets and a cathode frame contributing to the structural integrity. The cathode itself was comprised of two C-PTFE block beds, and two rubber rings were strategically positioned between the cathode, anode, and membrane to ensure electrical separation and prevent gas and liquid leaks. AMP and sulfuric acid (H_2SO_4) were dissolved in deionized water within a glass flask, and the solution temperature was precisely controlled by immersing the flask in a water bath, maintaining a temperature constant 15 °C. The experimental procedures, depicted in Figure2 (graphically), encompass the dissolution of AMP and H_2SO_4in deionized water, maintaining a controlled temperature. For waste removal, a dosing pump and a liquid flow meter were employed to regulate the liquid flow at a constant rate of 200 ml/min. additionally, a gas flow meter was used to ensure a constant flow rate of 9 L/min.

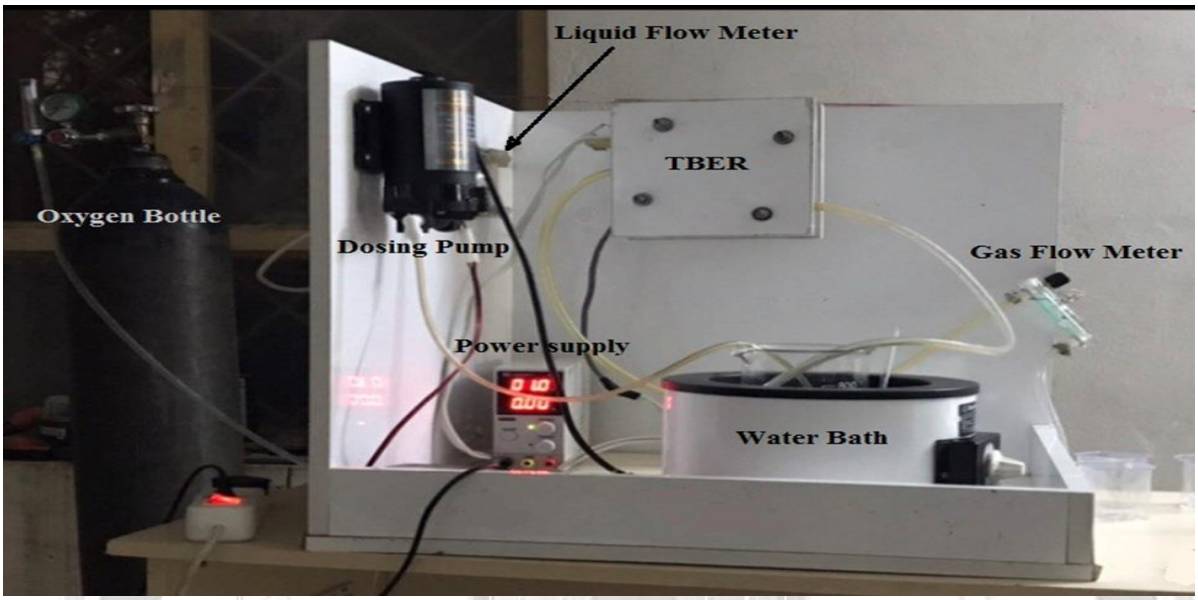
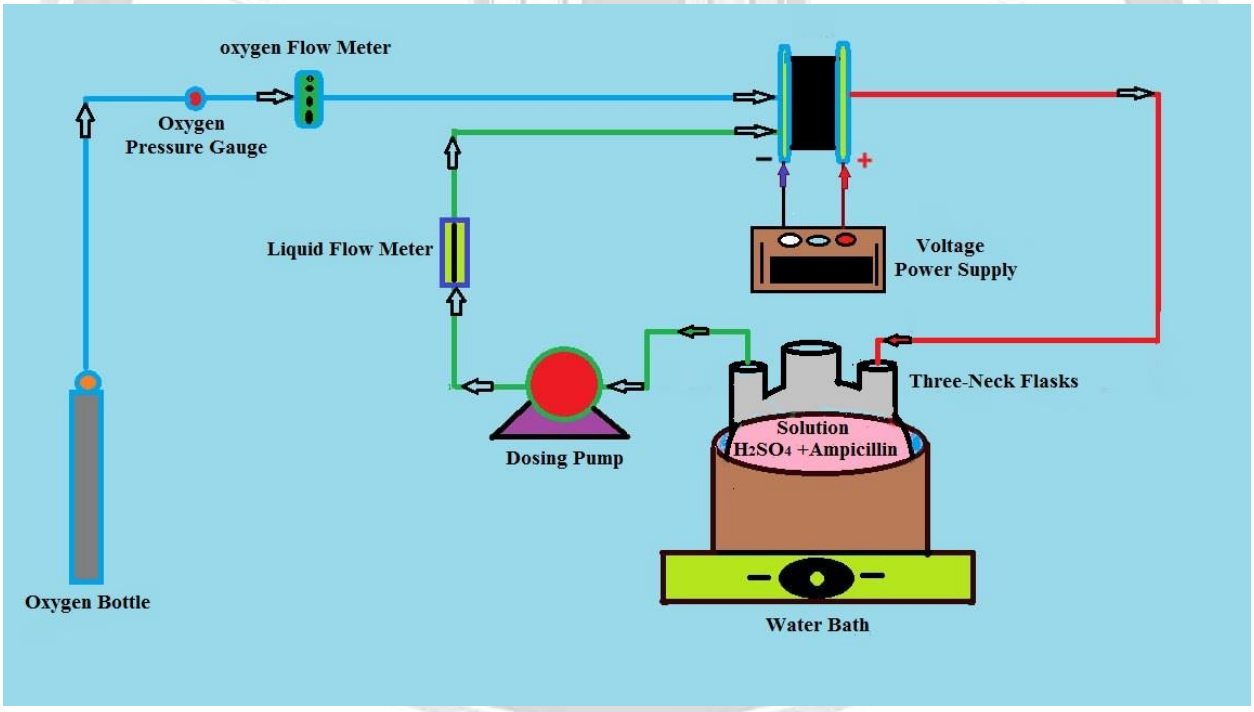


Figure1. Experimental Setup (real picture)



Figuer2.Experimental Setup (graphically)

2-3 Process and Mechanism of Ampicillin Oxidation:

Initially, the electrolytes and ampicillin were incorporated into the three flasks with a specific amount in the TBER process, the temperature of which was controlled. The oxygen was derived from the bottle of oxygen gas, and the flow was controlled by the meter that measures gas flow.

The wastewater [ampicillin and electrolyte (H_2SO_4)] was supplied from the top of the reactor, and the gaseous oxygen was supplied from the side of the reactor. The reactor was armed with enough power to create the requisite potential.

Figure 3 depicts the mechanism of electrochemical hydrogen peroxide production and ampicillin oxidation in TBER.

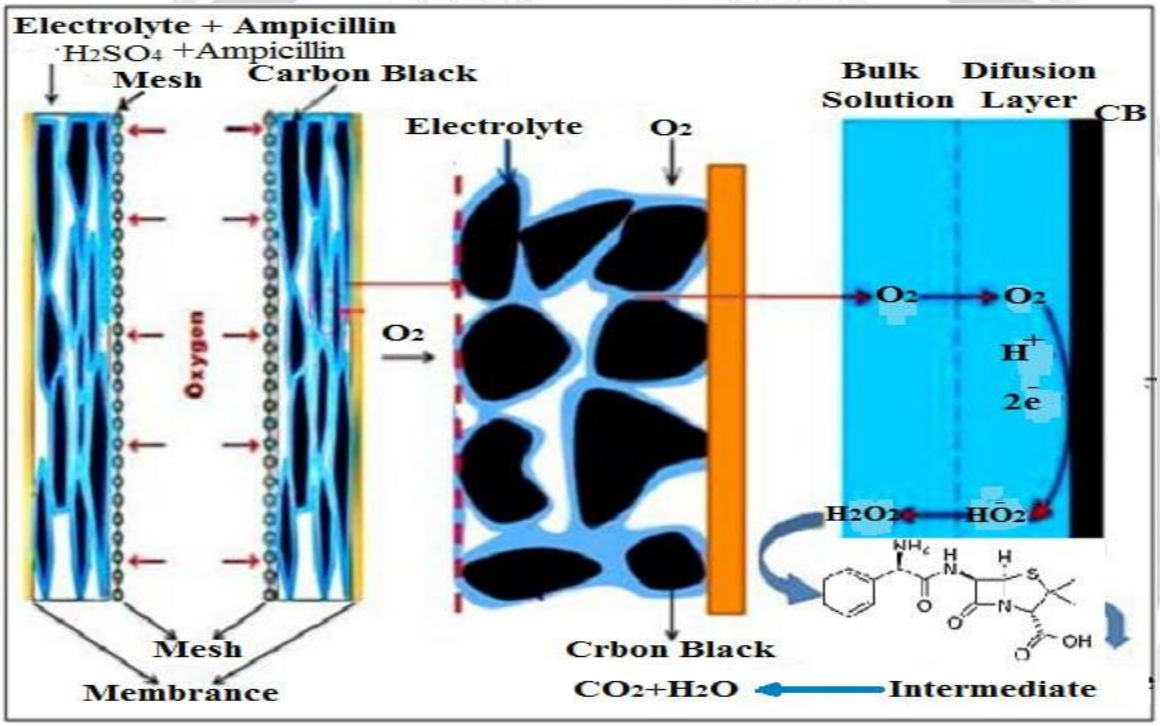


Figure3. The Reaction Mechanism through TBER

This block diagram (Figure 4) provides a visual overview of the key stages in the ampicillin oxidation process, illustrating the sequential flow of actions and interactions between different components.

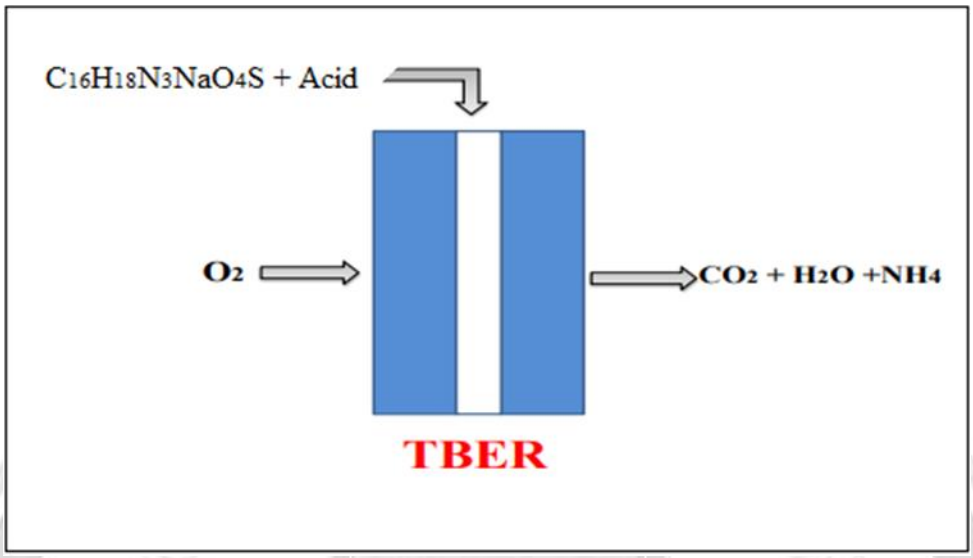


Figure 4. Block Diagram of Ampicillin Oxidation

In acidic media, the reduction of oxygen (O_2) to hydrogen peroxide (H_2O_2) at the cathode can be described by the following overall reaction:

Cathode: $O_2 + 2H^+ + 2e^- \rightarrow H_2O_2$

The presence of protons from the sulfuric acid facilitates the reduction of oxygen. The detailed mechanism involves multiple steps, including the reduction of oxygen to superoxide radical anions (O2^-), followed by protonation to form hydrogen peroxide. This can be represented by the following steps:

1. $O_2 + 2H^+ + 2e^- \rightarrow$ O2^- + H_2O

2. O2^- + $H^+ \rightarrow$ HO2^- (superoxide radical anion)

3. $2HO2^- + 2H^+ \rightarrow H_2O_2 + O_2$

The electrons (e^-) necessary for the reduction are supplied by an external circuit connected to the cathode. The cathode material is typically chosen to promote the reduction of oxygen to hydrogen peroxide.

The acidic medium is crucial in this process as it helps in protonation of intermediates and stabilizes reaction intermediates.

The oxidation of ampicillin at the anode may involve breaking specific bonds within the ampicillin molecule, leading to the formation of various oxidized products. The detailed mechanism of ampicillin oxidation in acidic media would depend on the specific conditions and electrode materials used in the electrochemical cell.

One possible oxidation reaction of ampicillin can be represented as follows:

Anode: Ampicillin $\rightarrow$ Oxidized products + e^-

The released electrons (e^-) flow through the external circuit to the cathode, completing the electrical circuit. The presence of sulfuric acid not only provides protons but also helps in maintaining the conductivity of the electrolyte solution, ensuring efficient electron transfer during the oxidation process.

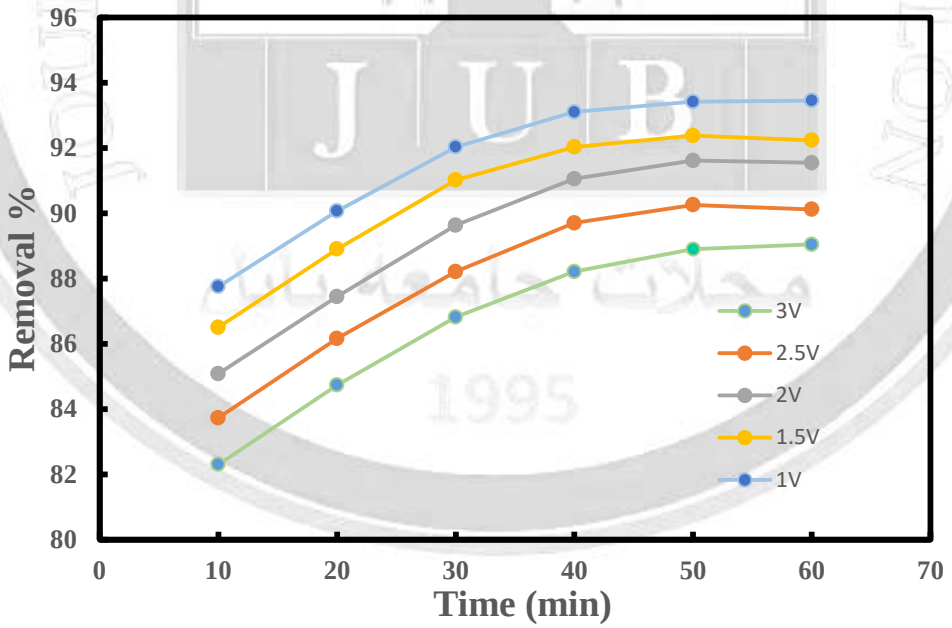
3-Results and Discussion

Factors critical in molding the breakdown of antibiotics through Enhanced Advanced Oxidation Processes (EAOPs)

3.1 Effect of the Voltage Applied Across a System

The research investigated the impact of varied applied potentials (ranging from 1 V to 3 V) on the oxidation of ampicillin in a modified TBER setup. The experimental conditions included a fixed the electrolyte concentration is 0.5M H_2SO_4, the electrolyte flow rate is 200ml/min, the oxygen flow rate is 9L/min, the ampicillin concentration is 100ppm and the reaction temperature is maintained 15°C.

As depicted in Figure5, the influence of applied potential on ampicillin conversion is highlighted. The graph displays an increase in ampicillin conversion as the applied potential peaked at 1 volt. However, with a subsequent rise in applied potential to 3 volts, there was a noticeable decrease in ampicillin conversion. To be specific, the conversion, starting at 1 volt, decreased from 93.5 %, and further elevation of the applied potential to 3 volts led to a reduction in the conversion to 89.1%



Figuer5. Influence of Applied Potential on Ampicillin Removal

When the voltage value was elevated from 1 v to 3v, the time required for the complete treatment of Ampicillin was reduced from 10minutes to 60minutes, as reported by [18]. The investigation additionally revealed that the deterioration of antibiotics increased as the current value increased, this was increasing linearly process. Additionally, [19.] The findings of the study indicate that as the current density increases, the degradation rate of metronidazole (MNZ) also increases. Specifically, at current densities of 1.6 mA/cm2, 4.8 mA/cm2, 8.0 mA/cm2, and 12.8 mA/cm2, the degradation efficiency is recorded as 40.53%, 59.81%, 68.83%, and 73.21% respectively. These results align with the predictions of an increases linearly.

3.2 The Duration Necessary for Achieving Maximum Removal

Although the first-order kinetic model is commonly applicable to antibiotic removal dynamics [20, 21] there is notable variability in the required operational time to achieve optimal removal efficiency, as illustrated in Figure6. Specifically, a shorter duration (less than 40 minutes) suffices in approximately 28% of studies, while a majority (42%) necessitate a more extended time frame (40 minutes to over 120 minutes). The extended treatment time appears unfavorable for the application of EAOPs due to increased reactor size and elevated energy consumption. Moreover, prolonging the treatment duration does not consistently result in improved removal efficiency, given the influence of various factors on the maximum removal efficiency of the treatment (Figure 6). Notably, a negative correlation was observed for treatment times longer than 70 minutes (80.6% ± 19.30%). As a result, strategies to reduce the response time while increasing the efficiency of removal are also advocated for, such as. Incorporating oxygen into the base electrolyte [22] improving the design of the reactor and its operating conditions [23].

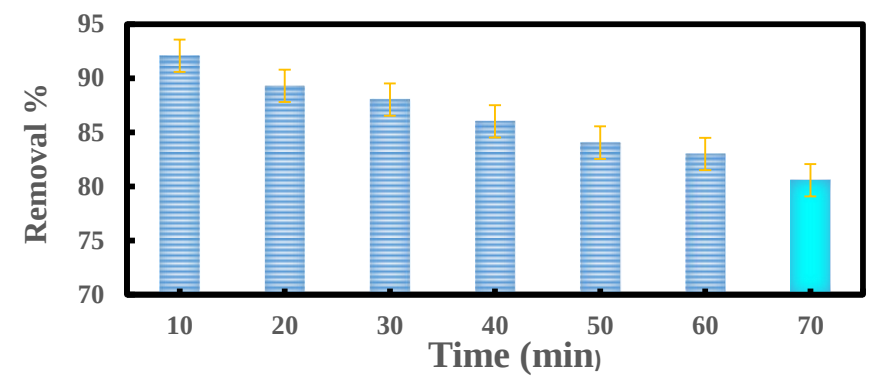


Figure 3.2. The duration necessary for achieving maximum removal

3.3 The Influence of Oxygen Flow Rate

The effect of different oxygen flow rates on ampicillin oxidation removal in TBER was studied, exploring flow rates of 2, 4, 7, 9, and 10 L/min. The conditions included an applied voltage of 1V, electrolyte concentration at 0.5 M H_2SO_4, electrolyte flow set at 200 ml/min, ampicillin concentration of 100 ppm, and a constant temperature of 15 °C.

Figure 7 illustrates the impact of oxygen flow rates on the removal of ampicillin oxidation. The data indicates a rise in ampicillin removal with increasing oxygen flow rates. Specifically, the conversion increased from 91.2% to 94.4% within the oxygen flow rate range of 2 to 10 L/min. However, further increases beyond 7 L/min led to a decline in ampicillin removal, dropping from 94.4% to 89.9%.

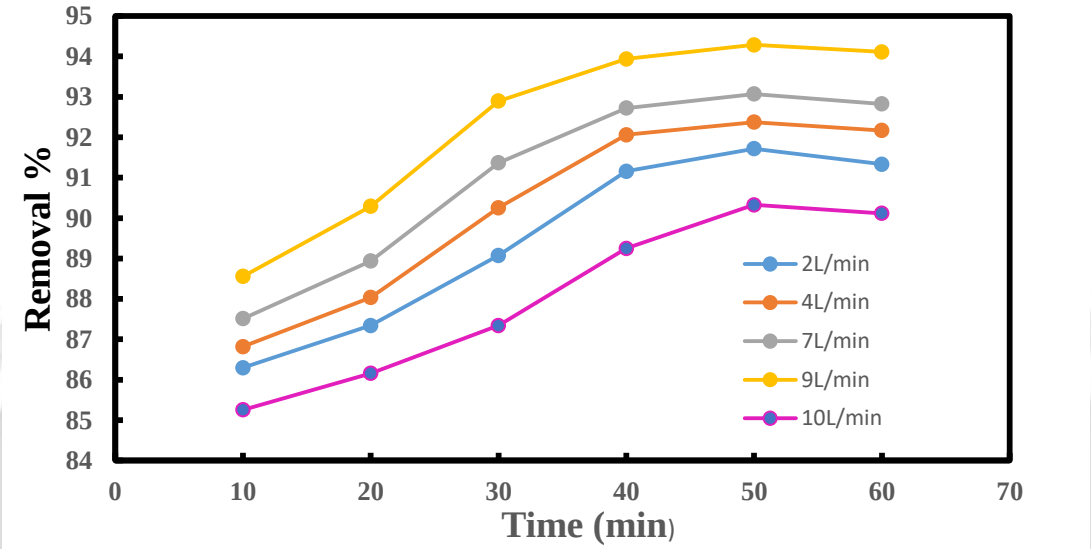


Figure 7 Impact of Oxygen Flow Rates on the of Ampicillin Removal

The increased removal rate of ampicillin is associated with the heightened concentration of generated H_2O_2 resulting from the increased oxygen flow rate. This elevation in oxygen flow facilitates the transmission of a greater quantity of oxygen through the liquid layer to the interphase, leading to an increased amount of reduced oxygen that produces peroxide ions, enhancing H_2O_2 production [24]. However, exceeding an oxygen flow rate of 9 L/min caused a reduction in the liquid phase's stoppage in the distillate layer, subsequently decreasing the liquid film thickness on the C-PTFE bed's surface and reducing active areas [25]. As a consequence, there is a decline in the rate of H_2O_2 generation and the electrochemical oxidation of ampicillin.

3.4 The Effect of Changing Electrolyte Flow Rates

The influence of different electrolyte flow rates on the removal of ampicillin oxidation in the modified TBER was examined at flow rates of (25, 75, 125, 200, and 250) ml/min. The applied voltage was 1V, electrolyte concentration was 0.5 M, oxygen flow rate was 9 L/min, ampicillin concentration was 100 ppm, and the temperature was maintained at 15°C.

The conversion of ampicillin is influenced by electrolyte flow rates, as illustrated in Figure 8. With an augmentation in the flow rate of electrolytes from 25 to 200 ml/min, the conversion of ampicillin experienced a notable surge, escalating from 90.1% to 93.6%. Nevertheless, surpassing the threshold of 200 ml/min resulted in a decline in the elimination of ampicillin, plummeting from 93.6% to 89.3%.

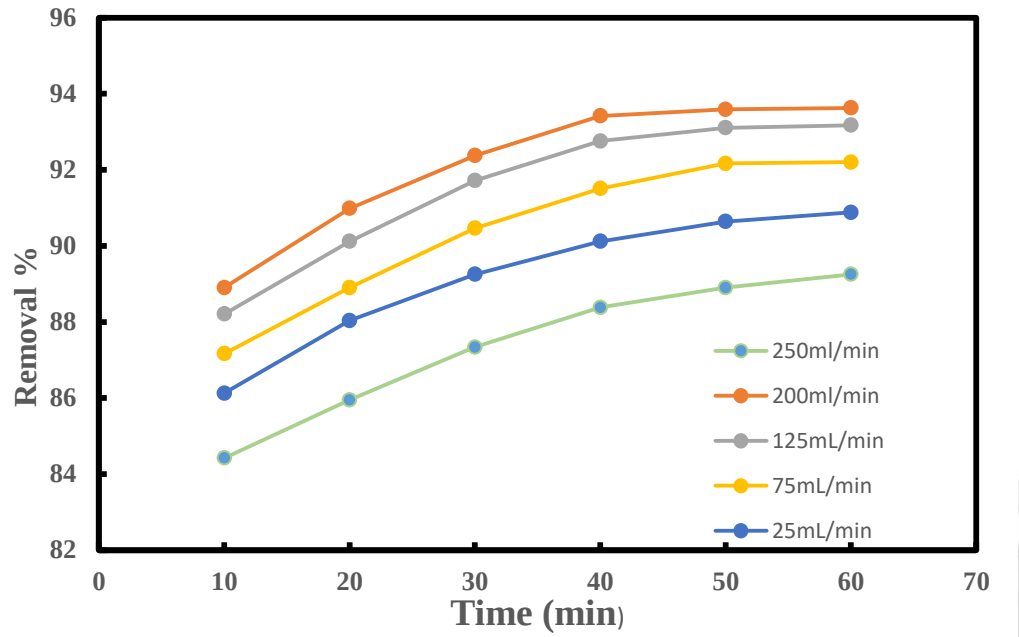


Figure 8.Influence Electrolyte Flow Rates on the Ampicillin Removal

The rise in ampicillin conversion is attributed to the increased concentration of hydrogen peroxide generated with the higher electrolyte flow rate, resulting from enhanced C-PTFE wetting and increased electrode effective area [26]. However, passing 200 ml/min of electrolyte water leads to an increase in the liquid volume in the trickle bed, this results in a thicker liquid layer forming on the C-PTFE surface. This impedes and delays the transfer of oxygen from the gas phase to the electrolyte-cathode interface, this diminishes the production of hydrogen peroxide in situ and thus decreases the amount of ampicillin that is oxidized.

4-Conclusion

To summarize, this manuscript introduces an innovative approach to address the degradation of ampicillin using an advanced in-situ oxidation technique within a specially designed TBER. The reactor, equipped with a stainless steel anode and a gas diffusion cathode made of carbon black polytetrafluoroethylene, enables on-site production of hydrogen peroxide for efficient antibiotic oxidation. The study systematically evaluates the impact of various operational parameters, such as applied voltage, electrolyte flow rates, oxygen flow rates, (ampicillin concentrations, electrolyte concentrations, reaction temperature 15°C)remain constant, and duration necessary for achieving maximum removal by electrochemical process within the TBER. The results indicate that the in-situ oxidation process conducted in the customized reactor holds significant potential for improving ampicillin elimination. Under optimal conditions, including a potential of 1 V, an Electrolyte concentration 0.5M H_2SO_4, electrolyte flow 200ml/min, oxygen flow 9L/min, ampicillin concentration 100ppm, reaction temperature 15°C, and a reaction time of 40 minutes, the highest achieved ampicillin removal rate was an impressive 93.6%.

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إزالة الأمبيسلين في موقع الاكسدة بواسطة المفاعل الكهروكيميائي ذوطبقة مزدوجة من كاثود (TBER)

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الخلاصة

يستخدم الأمبيسلين (AMP) على نطاق واسع كمضاد حيوي في المستشفيات والعيادات البيطرية في جميع أنحاء العالم لمكافحة الالتهابات البكتيرية. ونتيجة لذلك، يتم تحديد بقايا هذا المضاد الحيوي بشكل شائع في مياه الصرف الصحي الصادرة عن هذه المرافق. تقدم هذه الدراسة عملية أكسدة متقدمة لإزالة الأمبيسلين بكفاءة من مياه الصرف الصحي باستخدام مفاعل كهروكيميائي ذو طبقة مزدوجة من الكاثود .(TBER) ينتج هذا النظام الكهربائي بيروكسيد الهيدروجين (H_2O_2)الصديق للبيئة من خلال تقليل الأكسجين، والذي يستخدم بعد ذلك لأكسدة الأمبيسيلين في مياه الصرف الصحي. يتميز TBER بقطب كهربائي عالي المسامية (C–PTFE–SSM) مع معلمات تشغيل محسنة. تم تقييم فعالية هذه العملية الكهروكيميائية داخل TBER يبلغ عرضه 120 مم، وارتفاعه 110 مم، وعمقه 40 مم. مجهزة بأنود من الفولاذ المقاوم للصدأ وكاثود نشر غاز مصنوع من بولي تترافلوروإيثيلين أسود الكربون. ركز التقييم على إزالة المضادات الحيوية، وتحديدًا الأمبيسيلين(AMP) ، باستخدام جهد مطبق يتراوح من 1 إلى 3 فولت، وتركيزات الإلكتروليت 0.5 م، ومعدلات تدفق الإلكتروليت 25. إلى 250 مل/دقيقة، ومعدلات تدفق الأكسجين 2 إلى 10 لتر/دقيقة، وتركيز الأمبيسلين 100 جزء في المليون. ظلت درجة حرارة التفاعل ثابتة عند 15 درجة مئوية والمدة اللازمة لتحقيق أقصى قدر من الإزالة بواسطة العملية الكهروكيميائية داخل .TBER تشير النتائج إلى أن عملية الأكسدة في الموقع التي تتم في المفاعل المخصص تحمل إمكانات كبيرة لتحسين التخلص من الأمبيسيلين. في ظل الظروف المثالية، بما في ذلك جهد 1 فولت، وتركيز إلكتروليت 0.5 مولار H_2SO_4، وتدفق إلكتروليت 200 مل / دقيقة، وتدفق الأكسجين 9 لتر / دقيقة، وتركيز الأمبيسيلين 100 جزء في المليون، ودرجة حرارة التفاعل 15 درجة مئوية، وزمن التفاعل 40 دقيقة، أعلى وكان معدل إزالة الأمبيسيلين الذي تم تحقيقه مثيرًا للإعجاب بنسبة 93.6٪. في ظل الظروف المثالية.

الكلمات الدالة: الأكسدة في الموقع؛ تيبر؛ بيروكسيد الهيدروجين الأمبيسلين. تدهور المضادات الحيوية. الأنود الفولاذ المقاوم للصدأ