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Research Article

Electrooxidation of Oxacillin on a Boron-doped Diamond Electrode: A Voltammetric Investigation

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Abstract

The effectiveness of electrochemical techniques in preventing and resolving wastewater contamination issues has been demonstrated. However, this method requires knowledge of the organic pollutant's (Oxacillin: OXA) electrochemical behavior before electrolysis. The aim of this study is to enhance comprehension of the electrochemical process of oxacillin oxidation on the non-active boron-doped diamond (BDD) electrode. These electrochemical properties, focusing on phenomena at the electrode/electrolyte interface, were analyzed by cyclic voltammetry. Effects of concentration of oxacillin, potential scan rate, number of potential scanning cycles, temperature and chloride ions that were investigated allowed for the acquisition of some parameters. This study showed that BDD electrode can be used to quantitatively determine the presence of this substrate in medicines and environmental samples. The process is irreversible and diffusion controlled and proceed in two ways: an indirect oxidation mediated by in situ oxidative species and a direct electron transfer at the surface of the boron-doped diamond electrode. Parameters of OXA electrooxidation, such as anodic transfer coefficient, heterogenous rate constant and activation energy were estimated as 1.09, 1.97×10^{3} s^{-1} and 17.632kJ mol^{-1}. The increase in temperature and the presence of chloride ions promote of OXA. This indicates electrochemical conditions adequate to oxidize oxacillin on boron-doped diamond anode.

Keywords

Cyclic Voltammetry, Oxacillin, Boron-doped Diamond, Electrooxidation

1. Introduction

In recent decades, the treatment of various pathologies has been the basis for the emergence of pharmaceutical products in both human and animal medicine. Thus, various pharmaceutical substances exist such as antibiotics, anti-inflammatories, analgesics etc. Antibiotics are drugs generally used to treat quickly and effectively some types of bacterial infectious diseases [1, 2]. However, studies have shown that these pharmaceutical components are resistant to microbial degradation [3]. They are present in surface water, groundwater and even drinking water for consumption [4, 5] in concentrations ranging from ng/L to mg/L [6, 7], causing water pollution. Indeed, like many foods and supplements that are consumed by humans and animals, pharmaceuticals are not completely absorbed or metabolized in the body and are

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eventually excreted into the environment [8]. Their accumulation in the environment induces a development of antibiotic resistance bacteria [9, 10] leading to inefficiency of these products in the treatment of infectious diseases [11]. This poses a threat to human health and that of aquatic organisms [8, 12]. A semi-synthetic penicillin that is part of the β lactam antibiotic class, oxacillin is one of the most often used medications worldwide. Conventional urban wastewater treatment plants have been shown to leak over 50% of OXA into the environment at a concentration of 10ng/L [13]. The non-biodegradable character of this compound excludes biological treatment as an efficient option.

In this condition and because of its ability to oxidize completely the non-biodegradable compound by the intermediate of extremely reactive and unselective hydroxyl radical, electrochemical techniques have been chosen for such an investigation [14, 15]. This method does not require any addition of toxic chemical reagents and does not induce the production of hazardous compounds. The pathway and efficiency of the electrochemical process for the oxidation of organics is influenced by the nature of the anode. In fact, in this method, the electrochemical oxidation of organic compounds could proceed via various reaction pathways such as via direct electron transfer at the anode surface and/or via mediated oxidation with powerful oxidant, the hydroxyl radical •OH or other oxidative species formed from water or other species discharge at the anode surface [16]. In this context, the boron doped diamond (BDD), a non-active electrode, has emerged as a promising option for treating organic substances. BDD electrode has a good stability and extremely high oxygen evolution, which leads to the formation of a large quantity of •OH. It has interesting electrochemical properties, including high thermal conductivity, high hardness, chemical inertness, wide electrochemical potential window in both aqueous and non-aqueous media [17].

Among electrochemical method, cyclic voltammetry (CV) is one of the powerful electrochemical techniques used to understand the oxidation and reduction mechanisms of organic compounds [18-22]. In a variety of research context, CV is a flexible electrochemical method for analyzing redox state [23]. In fact, this technique allows the qualitative and quantitative study of a processes taking place at the electrode/electrolyte interface [24, 25].

The aim of this work is to study the electrooxidation of OXA on BDD electrode in neutral media by cyclic voltammetry. The influence of different parameters such as OXA concentration, potential scan rate, temperature variation, number of potential scanning cycles and chloride ions was investigated.

2. Materials and Methods

2.1. Electrode Preparation

By using hot-filament chemical vapor deposition (HF-CVD) on low resistivity (1-3 mΩ.cm) p-Si wafers (siltronix, diameter 10cm, thickness 0.5mM), Boron doped diamond (BDD) electrodes were produced. The process gas was a mixture of 1% CH_4 in H_2 containing trimethylboron. Film growth occurred at a rate of 0.24µm.h^{-1}. The film thickness was about 1µm.

2.2. Electrochemical Measurements

The voltammetric measurements were performed on the BDD electrode in a 100mL three-electrode electrochemical cell using an Autolab PGStat 20 (Ecochemie) connected to a potentiostat equipped with USB electrochemical interface. In addition to BDD as working electrode, the counter electrode (CE) was a platinum wire and the reference electrode (RE) was a saturated calomel electrode (SCE). All potentials reported were versus the SCE. To overcome the potential ohmic drop, these electrodes was placed close each over by a distance of 2mM and the reference electrode was mounted in a luggin capillary. The apparent exposed area of the BDD electrode was 1cm^2. The system is linked a data storage and processing computer equipped with GPES 4 software to launch and record the voltammograms.

Before the experiments, the BDD electrode was pretreated electrochemically in a 0.5mol/L H_2SO_4 solution. For this pre-treatment, an anodic pre-treatment (+2V, 15s) is followed by a cathodic pre-treatment (-2V, 90s). In this way, the BDD surface was first cleaned of all impurities and then mainly converted to hydrogen [26].

2.3. Chemicals

Oxacillin [(2S,5R,6R)-5-hydroxy-3,3-dimethyl-6-{[(5-methyl-3-phenyl-1,2-oxazol-4-yl) carbonyl] amino}-4-thia-1-azabicyclo [3.2.0] heptane-2-carboxylic acid] with the empirical formula $C_{19}H_{21}N_3O_5S$ was purchased from pharmacies in Abidjan. It was prepared by dissolving an accurate mass of the drug in an appropriate solution of 0.1M of potassium sulfate (K_2SO_4) used for the supporting electrolyte. Ultrapure distilled water was used to prepare the supporting electrolyte.

3. Results and Discussion

3.1. Effect of OXA Concentration on BDD

The voltammetric responses presented in Figure 1 show the behaviour of the BDD electrode in the presence and absence of OXA in the K_2SO_4 (0.1M) electrolyte support under a scan rate of 50mV/s. In the absence of OXA (K_2SO_4 alone: color black), a normal cyclic voltammogram of the BDD in neutral media is observed as in acid media with an increase in current density to 1.45V vs. SCE followed by oxygen evolution reaction [20, 27]. After addition of OXA (color red), we notice a current increase rapidly from 1.23 V followed by an oxidation peak at 1.6V vs. SCE in the forward potential scan (Figure 1). This finding indicates the oxidation of OXA on

BDD. In the reverse potential scan, no characteristic peak of OXA reduction was observed. That indicates that the oxidation of the OXA on DDB undergoes an irreversible electrochemical process.

This oxidation peak appears before oxygen evolution of the electrolytic media on the electrode of BDD. This reflects the direct oxidation of OXA which results from an electronic exchange to the interface electrolyte/BDD. Within the high potential domain, the recorded current is weak in the presence of OXA that in its absence suggesting that the oxidation of OXA takes also place via oxidative species in the reaction medium resulting from water electrochemical decomposition of water could involve that in the OXA oxidation in that domain is indirect [16]. Thus, these results indicate that the OXA degradation on this electrode can be done either by a catalytic oxidation mechanism involving direct oxidation of OXA on electrode surface, i.e. consumption of OXA on the surface of the electrode, or by indirect oxidation involving the adsorbed hydroxyl radicals or other oxidative species produced in-situ.

The variation in concentration is a kinetic parameter frequently used in electrooxidation of organic compounds. The study of the variation of the OXA concentration from 0.62 to 3.74mM on the BDD electrode was carried out under a scan rate of 50mV/s in a potential domain from -1.5V to 2.2V vs. SCE in K_2SO_4 (0.1M) media at ambient temperature. Figure 2A presents the results obtained. We progressively note an increase in the current density of the peak of oxidation with the addition with the concentration in OXA. Also, the oxygen evolution reaction becomes faster. These results show that during the oxidation of the OXA on the BDD in this medium, the electrochemical process could be controlled by diffusion [28]. Thereafter a potential was selected on the level of the peak of oxidation (1.6V/SCE) and the density of the anode current was studied according to the OXA concentration (Figure 2B). The layout obtained is a line of slope and determination coefficient $R^2 = 0.9959$ (equation (1)).

$$j_p = 0.0923C + 0.0466 \tag{1}$$

With j_p: the current density ($mAcm^{-2}$) and C: the OXA concentration (mM).

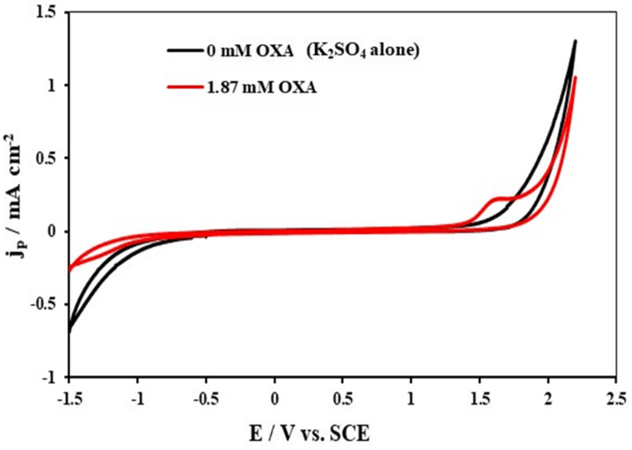

Figure 1. *Cyclic voltammograms of BDD in 0.1M K_2SO_4 in presence of 1.87mM OXA and absence of OXA (K_2SO_4 alone) at $50mVs^{-1}$.*

The linear curve obtained with a determination coefficient close to 1 indicates that the increase in the current density of the OXA oxidation peak is strongly dependent on the OXA concentration. This assure us that the OXA present in the medium is responsible for the modifications observed on the voltammogram. According to the results obtained, the BDD electrode would be useful for the quantitative determination of OXA.

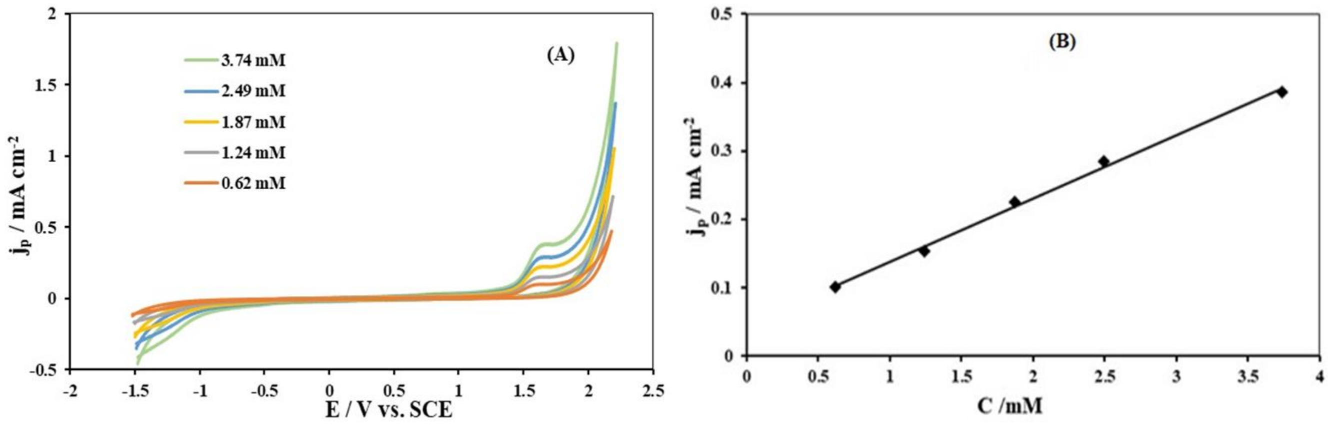

Figure 2. *(A) Cyclic voltammetry of BDD in K_2SO_4 (0.1M) in the presence of different OXA concentrations at $50mV\ s^{-1}$; (B) plot of current density of the OXA oxidation peak versus OXA concentrations.*

3.2. Investigation of the Potential Scan Rate

Scan rate is one of parameters significantly affecting electrooxidation of various compounds. It provides the information about the electrochemical processes such as reversibility and kinetic regime (adsorption or diffusion). Figure 3 shows the voltammetric response of the BDD at different scan rates (from 5 to 100mV/s) and for a concentration of 1.87mM OXA in K_2SO_4. These curves show that the intensity of the current oxidation increases with the scan rates.

For obtaining information on the reversibility, we analyzed two approaches: the dependencies of peak current density (j_p) on the square root of the scan rate ($v^{1/2}$) [29] and the logarithm of peak current density (log j_p) on the logarithm of the scan rate (log v) [30].

The first approach, the current density recorded at 1.6V/SCE and plotted against square root of the potential scan rates (Figure 4A). This curve leads to a linear relationship between j_p versus $v^{1/2}$ with the following equation:

$$j_p\ (\mathrm{mA\ cm^{-2}}) = 1.2139\ \mathrm{v^{1/2}}\ (\mathrm{V\ s^{-1}}) + 0.0012\ (\mathrm{R^2} = 0.9949) \tag{2}$$

This straight line does not intercept the origin of the axes, indicating that the oxidation process of OXA could be an adsorption-controlled process and preceded or followed by a homogeneous chemical reaction [29, 30].

The second approach, recorded at same potential, the logarithm of the current density as a function of the logarithm of the potential scan rates is summarized in Figure 4B. The resulting curve describes a line characterized by the equation (3).

$$\log\ j_p(\mathrm{mA\ cm^{-2}}) = 0.4969\ \log \mathrm{v}\ (\mathrm{V/s}) + 0.0782\ (\mathrm{R^2} = 0.9945) \tag{3}$$

The slope of this straight line has a value of 0.4969 close to 0.5, so the electrooxidation process of the OXA is limited by diffusion [31]. According the results of both approaches, we can deduce that an adsorption-diffusion phenomenon is occurring [20, 31, 32].

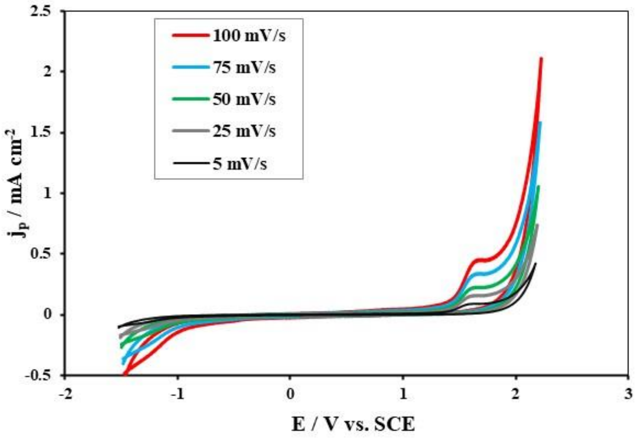

Figure 3. *Cyclic voltammograms of BDD in 0.1 M K_2SO_4 containing 1.87mM OXA at different scan rate.*

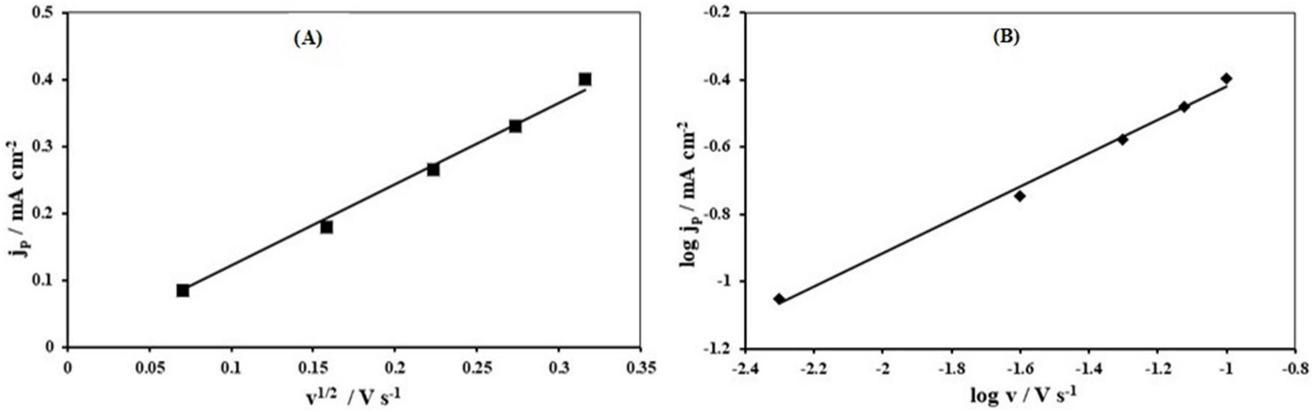

Figure 4. *(A) Current density as a function of the square root of the potential scan rate; (B) Plot of log j vs. log v.*

Figure 5A represents a dependence of the potential of the oxidation peak Ep on v potential scan rate determined from cyclic voltammograms recorded for the OXA electrooxidation. The potential of the oxidation peak increases and shifts slightly to the right with the scan rate. It can be concluded that during the electrooxidation of OXA at BDD in the electrolyte medium used, the heterogeneous electron transfer is irreversible [32]. i.e., there is a slow exchange of electrons of the redox species with the BDD electrode [33].

Additionally, a study on the potential of peak Ep as a function of log (v) has been conducted and presented in the Figure 5B. The dependence of Ep vs. log v is linear with a determination coefficient 0.9872 and described by the equation.

$$\mathrm{Ep} = 0.0531 \log(\mathrm{v}) + 1.7199 \qquad (4)$$

With E_P: peak potential (V) and v: scan rate (V s^{-1}).

Applying Laviron's equation for an irreversible process at the electrode described by the following equation [34]:

$$E_p = E^0 + \left(\frac{2.303RT}{\alpha nF}\right) \log\left(\frac{RTk^0}{\alpha nF}\right) + \left(\frac{2.303RT}{\alpha nF}\right) \log v \qquad (5)$$

With Ep is the peak potential in V, E^0 formal potential in V, v is the potential scan rate (V s^{-1}), R is the universal gas constant (8.314J K^{-1} mol^{-1}), n is the number of electrons involved, T is the temperature in Kelvin (298 K), k^0 is the standard heterogenous rate constant of the reaction.

According to equations (4) and (5), αn was determined to be 1.09. For a totally irreversible electrode process, the electron transfer α is taken to be 0.5 [30, 35]. Therefore, it was (s^{-1}), F is Faraday's constant (96487 C mol^{-1}) and α is the anode transfer coefficient determined that there were 2.18 ≈ 2 electrons involved. The electrochemical oxidation of OXA at BDD involved two electron transfer phenomenon. E° and k° can be deduced respectively from the extrapolation of the trend curve from data obtained from the curve Ep = f (v) and Ep = f (log v) using equation (5). The values of E° and k° are respectively 1.61V and $1.97.10^3$ s^{-1}.

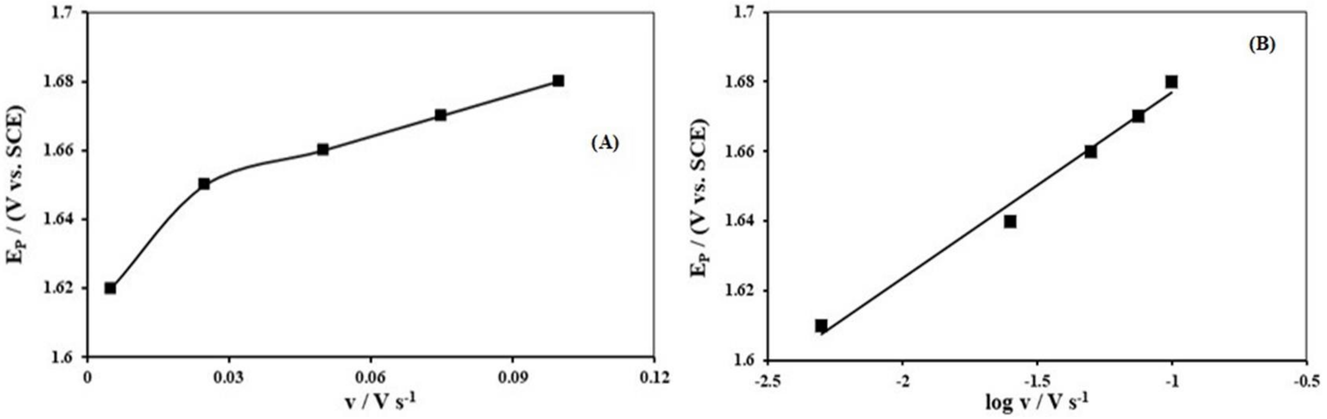

Figure 5. *(A) Dependence of peak potential (Ep) on the potential scan rate (v); (B) Plot of Ep vs. log v.*

3.3. Effect of the Number of Potential Scanning Cycles

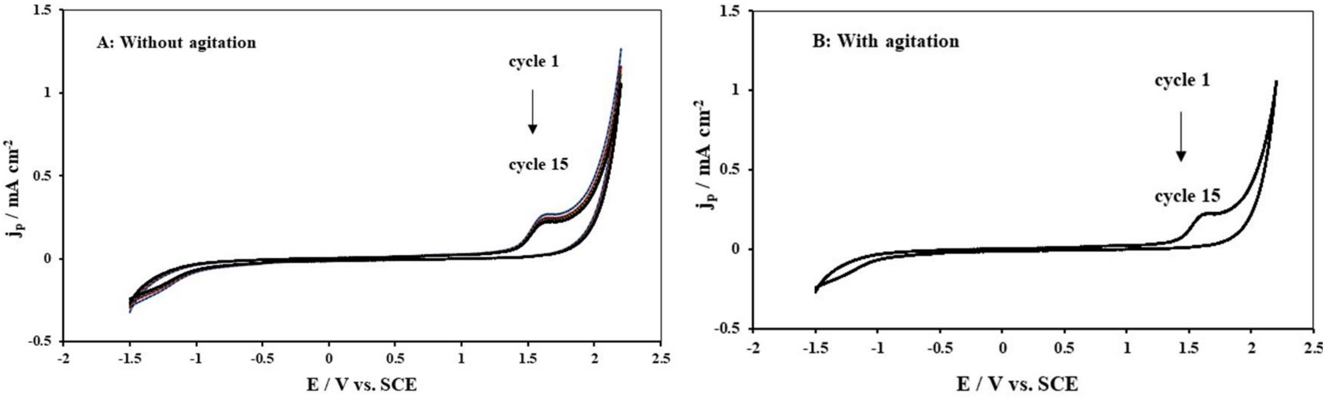

Figure 6. *Cyclic voltammogram of BDD in 0.1M K_2SO_4 for 15 successive Cycles in scans performed under a potential scan rate of 50mV s^{-1}; (A) without agitation; (B) with agitation.*

To further investigate this phenomenon, a study on the effect of successive cycling on the oxidation of OXA was conducted using a BDD electrode in 1.87mM OXA dissolved in K_2SO_4. Figure 6A presents the voltammograms recorded over 15 consecutive cycles within a potential window from -1.5V to +2.2V vs. SCE, at a scan rate of 50mV s^{-1}; under unstirred conditions. A decrease in the oxidation peak current is observed from cycle 1 to cycle 4, followed by the overlapping of curves from cycle 4 to cycle 15. This behavior suggests passivation of the BDD surface, likely due to the formation of a polymeric film, similar to phenomena reported during phenol oxidation on platinum [36] or methyl orange oxidation on BDD [37].

Under identical conditions but with stirring (Figure 6B), the voltammograms are nearly perfectly superimposed, indicating that the previously observed current decay was probably due to depletion of OXA near the electrode surface. This quasi-superposition confirms the stability of the BDD electrode response and supports its suitability for the electrochemical degradation of OXA in aqueous media.

3.4. Effect of Temperature Variation

The effect of temperature on the oxidation of OXA was investigated in a sodium sulfate solution (0.1 M) in presence of 1.87mM OXA at 50mVs-1. The results obtained are presented by Figure 7A. The oxygen evolution potential moves towards lower values with the increase in the temperature. Indeed, a decrease in this potential is observed from 1.56 V vs. SCE at 298 K to 1.45 V vs. SCE at 353 K. Also, an increase in the oxidation current density with temperature. This indicates an acceleration of OXA oxidation process with increasing temperature. The improvement of the kinetic of the OXA oxidation can be also explained by the in-situ production of more than hydroxyls radicals by electrochemical decomposition of water in the high potential range. The plot of the logarithm of the peak current density as a function of the inverse of temperature (ln j = f($\frac{1}{T}$) is shown in Figure 7B.

The curve ln j_p = f($\frac{1}{T}$) obtained is a straight line showing a linear relationship between ln j and the inverse of the temperature with j_p peak current density (Acm^{-2}) and T temperature (K):

$$\ln j_p = -2120.8\,\frac{1}{\mathrm{T}} + 6{,}433\ (\mathrm{R}^2 = 0.9948) \qquad (6)$$

The value of the activation energy (E_a) which gives information about the kinetic regime was estimated using the slope of equation (3) and the following relationship [38]:

$$E_a = -\mathrm{R}\ \left(\frac{\partial \ln j}{\partial(\frac{1}{T})}\right) \qquad (7)$$

We obtain a value of 17.632kJ/mol. This value of E_a lower than 40kJ /mol indicates that the OXA is oxidation controlled by diffusion [39, 40].

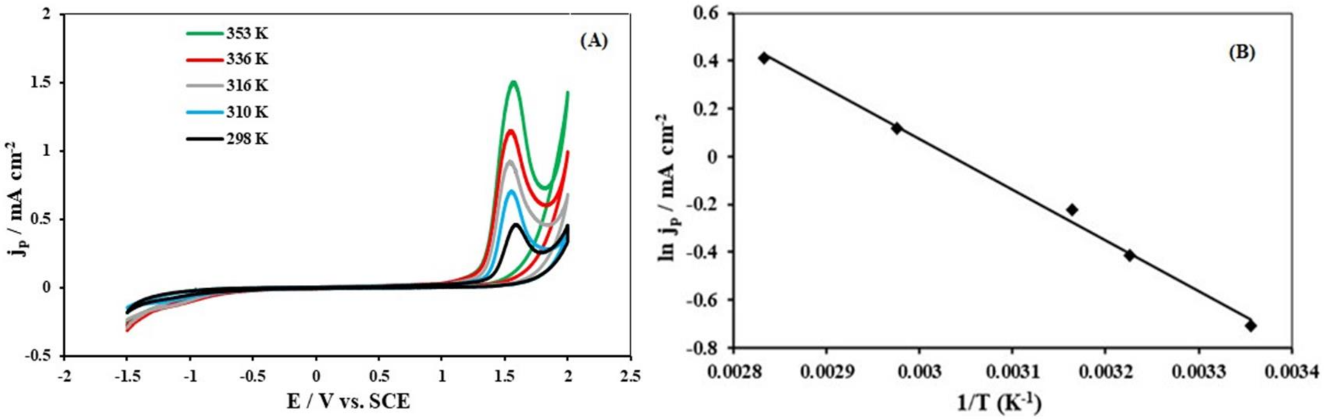

Figure 7. *A) Cyclic voltammetry of BDD at several temperatures in 0.1M K_2SO_4 containing 1.87mM OXA under the potential scan rate: 50mV s^{-1}, (B) plot of ln j_p = f ($\frac{1}{T}$).*

3.5. Effect of Chloride Ions

Figure 8 below, represents the voltammetric response performed in K_2SO_4 (0.1M) media containing 50mM of KCl and in presence of 1.87mM OXA. In this figure, in absence of KCl, current density increases from 1.23V/ECS, followed by gas evolution. However, in presence of KCl, this rapid increase in current density starts at 1.19V, also followed by the evolution of gas. Based on the work of Kambiré et al, in the presence of chloride ions Cl^-, the chlorine (Cl_2) evolution is faster than that of oxygen [41]. Thus, the gas evolving after 1.19V is linked to the evolution of chlorine. Furthermore, in presence of KCl, the oxidation peak of oxacillin is more accentuated.

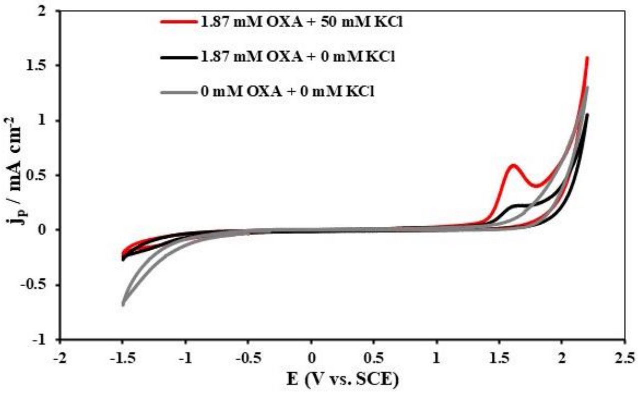

Figure 8. *Cyclic voltammograms of BDD in 0.1M K_2SO_4 in presence or absence of OXA and KCl at 50mV/s.*

In Figure 9A, the rapid increase of the current intensity observed at around 1.58V, before the evolution of gas, is linked to a direct OXA oxidation through an electronic charge transfer reaction. In the high potential domain, this current density increases with the addition of Cl^-. The chloride ions contribute to the OXA oxidation. It appears clearly that chlorides contribute in enhancing the kinetic of the OXA oxidation. In addition, the higher current density recorded in the presence of chlorides could result from the oxidation between Cl_2 and the OXA leading to the generation of oxidized by product on the electrode surface leading to indirect oxidation.

j_p plotted as a function of the KCl concentration for a potential set at 1.56V gives a straight line with $R^2 = 0.997$ (Figure 9B).

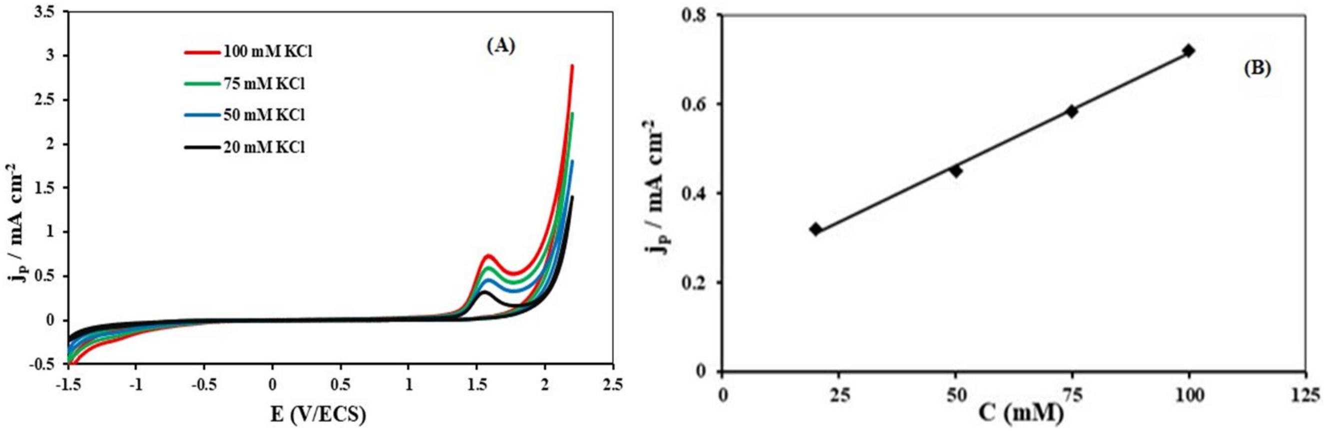

Figure 9. *(A) Cyclic voltammetry of BDD in K_2SO_4 (0.1M) containing 1.87mM OXA in the presence of different KCl concentrations at 50mV s^{-1}; (B) plot of $j_p = f(C)$.*

Figure 10 illustrates the obtained results following an investigation of the effect of the potential scan rate on the BDD electrode in the electrolyte support K_2SO_4 (0,1M) containing 50mM of chloride ions in the presence of 1,87mM of OXA. We note a stressing of the current density of the OXA oxidation of as well as a slightly shift of the gas evolving potential in the negative direction. The current density has been plotted against square root of the potential scan rate (Figure 11A). The curve $j_p = f(v^{1/2})$ is linear with a determination coefficient $R^2 = 0.9922$. These results suggest a diffusion-controlled mechanism in that domain of potential [29, 30].

To check the preceding assumptions of the adsorbent or diffusive character of the electrooxidation, the dependence of $\log j_p$ as function of $\ln v$ was studied. The curve $\log j_p = f(\log v)$ are presented on the Figure 11B. The layout obtained is a line of equation (8).

$$\log j_p = 0{,}4988 \log \mathrm{v} + 0{,}2982\ (\mathrm{R2} = 0{,}9967) \qquad (8)$$

The slope is equal to 0.4988. This indicates that the OXA oxidation process on the BDD electrode in the presence of chloride ions occurs by diffusion [31].

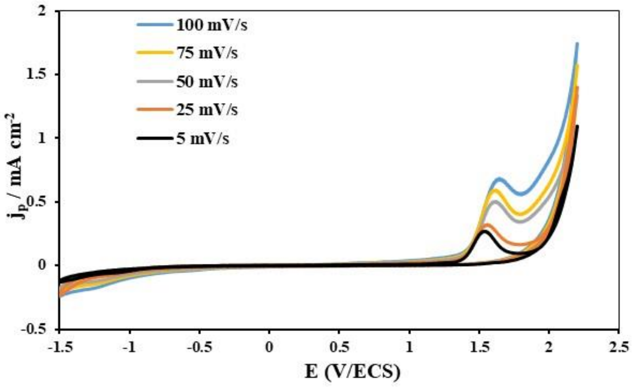

Figure 10. *Cyclic voltammograms of BDD in 0.1M K_2SO_4 containing 1.87mM OXA and 50mM KCl at different scan rate.*

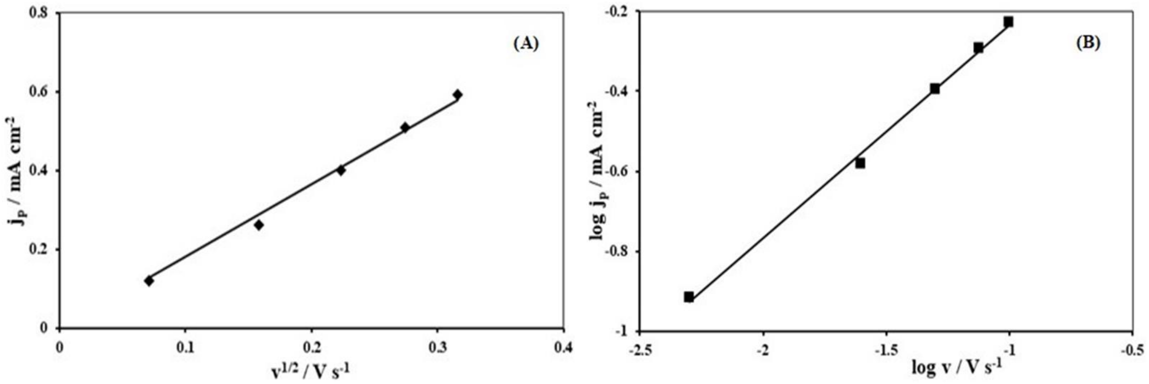

Figure 11. *(A) Current density as a function of the square root of the potential scan rate; (B) Plot of log j vs. log v.*

4. Conclusion

The electrooxidation of OXA has been studied on BDD in a potassium sulfate media. The electrooxidation of OXA is irreversible and proceeds by diffusion of OXA onto the BDD. The linearity of the dependence of the oxidation peak of the current density on the OXA concentration shows that BDD can be applied to the determination of OXA concentration in environmental and pharmaceutical samples. The OXA oxidation on the BDD electrode occurred directly via electron exchange at the electrode/solution interface and / or through oxidative species produced in solution. The study of the temperature and chloride ions influence showed that the process of OXA electrooxidation is accelerated on the BDD. The value of the activation energy, anodic transfer coefficient and the standard heterogeneous rate constant are respectively, 17.632kJ mol^{-1}, 1.09 and $1.97\times 10^3 s^{-1}$. This suggests that the boron-doped diamond anode has electrochemical conditions suitable for oxidizing oxacillin.

Abbreviations

OXA	Oxacillin
BDD	Boron-doped Diamond
SCE	Saturated Calomel Electrode

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Author Contributions

Souleymane Koné: Investigation, Validation, Formal Analysis, Methodology, Software.

Jean-Claude Meledje: Writing – original draft, Data curation, Writing – review & editing, Formal Analysis, Methodology.

Kouakou Jocelin Kimou: Writing – original draft, Data curation, Writing – review & editing, Formal Analysis, Methodology.

Lassine Ouattara: Conceptualization, Supervision, Data curation, Validation, Methodology, Visualization.

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Conflicts of Interest

The authors declare no conflicts of interest.

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Biography

Jean-Claude Meledje holds a Master's degree in Nanoscience and Energy Chemistry from Université Paris Diderot, completed under the supervision of Prof. Cyrille Costentin. He earned a Ph. D. in semiconductor electrochemistry in 2020, under the joint supervision of Prof. Ouattara Lassiné and Prof. Anne-Marie Gonçalves. He is currently a researcher at the Laboratory of Matter Constitution and Reaction at Université Félix Houphouët-Boigny in Abidjan. His research focuses on the electrochemical detection and degradation of organic pollutants.

Research Field

Souleymane Kone: Physical chemistry, Electrochemistry, Electrooxidation, Water treatment, Organics and inorganics detection.

Jean-Claude Meledje: Physical chemistry, Nanoscience, Energy Chemistry, Electrochemistry Electrochemical detection, electrochemical degradation.

Kouakou Jocelin Kimou: Physical chemistry, Electrochemistry, Electrooxidation, Water treatment, Organics and inorganics detection.

Lassine Ouattara: Physical chemistry, Electrochemistry, Electrooxidation, Water treatment, Organics and inorganics detection.