



## Behavior of boron-doped diamond anode on methyl orange oxidation

Kambiré Ollo<sup>1\*</sup>, Pohan Lemeyonouin Aliou Guillaume<sup>2</sup>, Konan Kouadio Franck<sup>3</sup>,  
Mohamed Berté<sup>3</sup>, Gnamba Corneil Quand-même<sup>3</sup> and Lassiné Ouattara<sup>3</sup>

<sup>1</sup>UFR Sciences et Technologies, Université de Man, BP 20 Man, Côte d'Ivoire.

<sup>2</sup>UFR Sciences Biologiques, Université Peleforo Gon Coulibaly de Korhogo, BP 1328 Korhogo, Côte d'Ivoire

<sup>3</sup>Laboratoire de constitution et réaction de la matière, UFR SSMT, Université Félix Houphouët-Boigny de Cocody, Abidjan, 22 BP 582 Abidjan 22, Côte d'Ivoire

\*Corresponding author, Email address: [kambireollo@yahoo.fr](mailto:kambireollo@yahoo.fr)

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[kambireollo@yahoo.fr](mailto:kambireollo@yahoo.fr)  
Phone: +225 0707416783

### Abstract

The presence of dyes such as methyl orange in wastewater discharged into the environment poses a risk to human health. Thus, our objective is to study the electrochemical oxidation of methyl orange (MO) on boron-doped diamond (BDD) electrode. The BDD electrode morphology was studied by scanning electron microscopy (SEM). As for the electrochemical properties of the electrode, they were studied by cyclic voltammetry. The effects of several parameters such as the supporting electrolyte nature (acidic medium and basic medium), the initial concentration of methyl orange and the potential scan rate were determined. SEM results revealed a polycrystalline structure, randomly oriented crystals stuck together. The electrochemical characterization of the electrode, carried out in acidic and basic media, showed that it is stable against corrosion, whatever the medium chosen. Also, the methyl orange oxidation on BDD electrode starts faster in acidic medium (0.04 V/MSE) than in basic medium (0.2 V/MSE). The voltammetric study showed that the MO oxidation takes place in the stability range of the supporting electrolyte. Thus, the MO oxidation occurs directly through an electronic exchange at the electrode-solution interface. This oxidation is controlled by a mixed adsorption-diffusion process and preceded or followed by a homogeneous chemical reaction in acidic and basic media. In the acidic medium, an oxidation peak is observed at 0.22 V/MSE followed by two oxidation waves at 0.46 and 0.68 V/MSE respectively, in the forward potential scan. In the backward potential scan, we observe a peak and a reduction wave at -0.053 V/MSE and -0.521 V/MSE, respectively. In contrast, in the basic medium, a single oxidation peak is observed at 0.43 V/MSE, in the forward potential scan. In the backward potential scan, we have a reduction wave at -0.64 V/MSE. This study also showed that the BDD anode can be used for a quantitative determination of methyl orange in acidic and basic media. Thus, the electrochemical method proves to be an adequate means to oxidize methyl orange and thus treat wastewater containing this compound.

## 1. Introduction

The textile industry, like the printing industry, is considered one of the most polluting sectors due to the high volume of colored wastewater discharged into the environment. Among the most used dyes in industrial environment, azo compounds (-N=N-) occupy one of the first places and represent about 70% of the world production of dyes [1,2]. The presence of this type of dye in wastewater discharges constitutes an important environmental problem and, therefore, imposes technological challenges for the treatment and purification of these wastewaters [3-6].

Many methods, physical-chemical, chemical and biological, have been used to treat wastewater containing synthetic dyes [7,8]. Taken individually, these methods have many advantages but also

some limitations that lead to insufficient efficiency in their applications. For example, physical-chemical methods [9-11] such as precipitation, coagulation, filtration, adsorption, etc., although effective in decolorizing wastewater, have the disadvantage of forming sludge which in turn requires treatment. Also, despite the relatively low cost of biological methods, they are less suitable for the industrial wastewater treatment, due to their complexities [12]. Thus, advanced oxidation processes (AOPs) have been proposed as alternative and effective methods for wastewater treatment containing toxic and hardly biodegradable pollutants. The AOPs generally used are: wet air oxidation, ozonation, electrochemical oxidation, photo-catalysis, Fenton process oxidation and their combined processes [13-17]. Their easy implementation and environmental compatibility have increased the interest of scientists in AOPs [18,19]. Compared to other methods of the AOP family, electrochemical oxidation is the most suitable for biorefractory pollutants degradation. Indeed, this method has some significant advantages such as moderate temperature and pressure requirements, high degradation efficiency, avoided secondary pollution and no use of chemical reaction [20-24].

The efficiency of electrochemical oxidation depends on the nature of the material used as anode. Due to their high surface loads, excellent mechanical and chemical resistance, dimensionally stable anodes (DSA type anodes) have been used in the field of wastewater treatment [25-29]. For example, in the studies carried out by our research team, the electrooxidation of stimulated and real wastewater was performed on iridium and/or ruthenium oxide electrodes. However, the use of DSA type electrodes does not lead to a total mineralization of organic pollutants, with or without the mediation of active chlorine (mixture of chlorine ( $\text{Cl}_2$ ), hypochlorous acid ( $\text{HOCl}$ ) and hypochlorite ion ( $\text{ClO}^-$ )) [26, 27]. Thus, another type of anode, the boron-doped diamond electrode (BDD) has received much attention in electrochemistry [30, 31]. It has been shown that BDD electrodes generate a significant amount of hydroxyl radicals on their surface which can lead to 100% decolorization and even total mineralization of organic pollutants [16, 32-33]. This electrode will therefore be used in this study.

The objective of this work is to contribute to the study of the electrochemical oxidation of methyl orange on BDD electrode using cyclic voltammetries. More specifically, it will be a question of characterizing by physical and electrochemical methods the BDD surface. Then to study the methyl orange oxidation using BDD electrode in acid and basic medium.

## **2. Methodology**

### **2.1. Measurement methods**

The voltammetric measurements were carried out using an ECHOCHEMIE Autolab Potentiostat (PGSTAT 20) connected by interface to an electrochemical cell with three electrodes: a working electrode, a counter electrode and a reference electrode. The working electrode is a BDD electrode with a geometrical contact area of  $1 \text{ cm}^2$ . The reference electrode is a mercurial sulfate electrode (MSE) placed in a capillary luggin close to the working electrode in order to avoid the ohmic drop. A coiled platinum wire is used as a counter electrode.

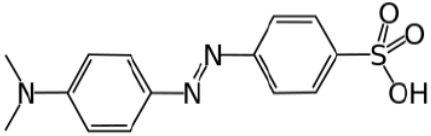
### **2.2 Chemicals**

The electrolytes used in this study were prepared with sulfuric acid ( $\text{H}_2\text{SO}_4$ ) and sodium hydroxide ( $\text{NaOH}$ ). The reagents and solids used were stored in the dark and at room temperature. All solutions were prepared with distilled water.

Methyl orange (C.I Acid Orange 52) otherwise known as Helianthine or Orange III is a mainly acidic anionic dye. It is a color indicator used mainly for printing textile dyeing, but also in chemistry.

Table I summarizes the methyl orange properties. The reagents and solids used are stored in the dark and at room temperature. All solutions were prepared with distilled water.

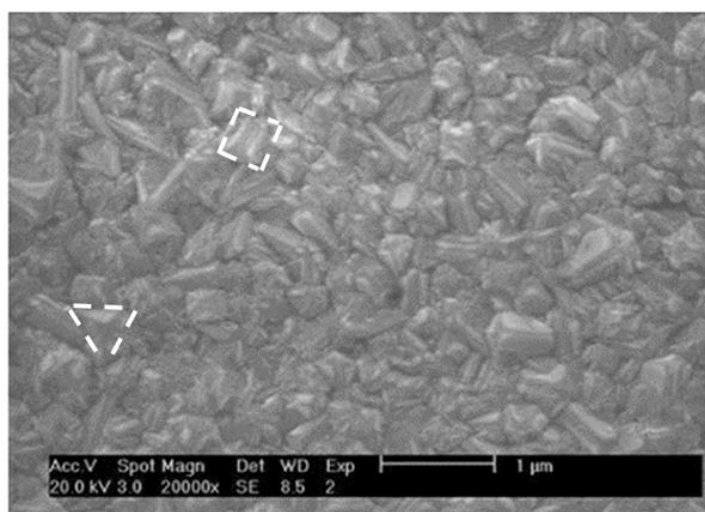
**Table I.** Methyl orange characteristics

<b>Name</b>	Methyl orange (C.I. 13025) PA-ACS
<b>Family</b>	Acid dye
<b>Formula</b>	C <sub>14</sub> H <sub>14</sub> N <sub>4</sub> NaO <sub>3</sub> S
<b>Structure</b>	
<b>Molar mass</b>	327.336
$\lambda_{\max}$	465 nm
<b>Solubility</b>	5.2 g.L <sup>-1</sup> in water at 20°C; 1.0 g.L <sup>-1</sup> in ethanol at 20°C

### 3. Results and Discussion

#### 3.1. Physical characterization of the electrode

Figure 1 shows the scanning electron microscope (SEM) image of the boron-doped diamond electrode. Randomly oriented crystals of a few micrometers in size with predominantly cubic and triangular faces can be distinguished. This image indicates that DDB has a polycrystalline structure [34]. The crystals have sizes between 0.3 and 0.6  $\mu\text{m}$ . The grains are strongly bonded to each other. In sum, polycrystalline diamond consists of crystallites mixture, of micrometer size and of two distinct geometric shapes, separated by grain boundaries.



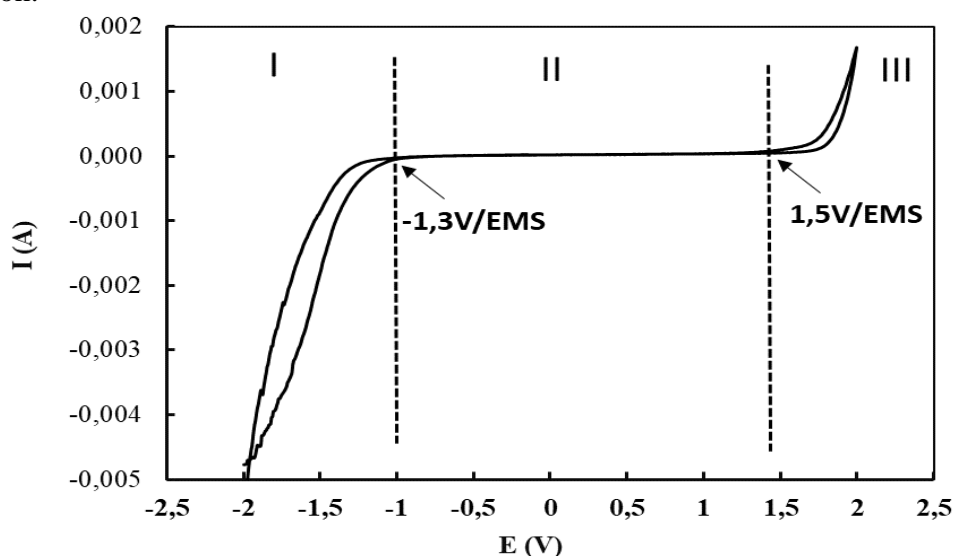
**Figure 1:** SEM image of BDD anode surface

The crystallites have square or triangular faces. However, these faces are not present in equal quantity, the triangular faces predominate. This effect would come from the conditions of the electrode preparation [35]. Also, the triangular faces are darker than the square faces. This contrast would be related to a more important incorporation of boron [36].

### 3.2 Electrochemical characterization of the electrode

#### 3.2.1 Electrode characterization in $H_2SO_4$ (0.1 M)

The electrochemical behavior of the BDD electrode was studied in sulfuric acid medium (0.1 M), in a potential range from -2 V/MSE to 2 V/MSE. The results obtained are shown in Figure 2. The measurements were performed under a potential sweep rate of 20 mV/s. The voltammogram obtained shows three regions. A first region (I) from -2 to -1.3 V/MSE, characterized by a rapid decrease in current, corresponds to the dihydrogen release domain. Then, a second region (II), from -1.3 to 1.5 V/MSE, where we note an absence of electrochemical reaction translated by a quasi-null current. Indeed, there was no modification of the electrode surface in the electrolytic solution. This area is called the electroactivity domain of the supporting electrolyte with a potential window  $\Delta E = 2.8$  V translating the stability of the BDD electrode towards acid corrosion. Finally, the last region (III) for potentials higher than 1.5 V/MSE, characterized by a rapid increase of the current reflecting the oxygen evolution reaction.



**Figure 2:** Voltammetric curve of BDD in  $H_2SO_4$  (0.1M) at 20 mV/s

#### 3.2.2 Behavior of the electrode in $NaOH$ (0.1 M)

In  $NaOH$  0.1 M and keeping the same experimental conditions as in the acidic medium, the voltammogram of the DDB electrode was recorded. The measurements were performed at 20 mV/s. The voltammogram obtained is shown in Figure 3. In the basic medium, three regions are also noted, this time with the existence of an electrochemical reaction in region II. In this medium, the oxygen evolution reaction starts a little earlier than in the acidic medium, at 1.3 V/MSE. The hydrogen evolution reaction is a little more delayed than in the acidic medium. It starts at a potential close to -1.8 V/MSE. In region II, we note the presence of an oxidation wave around 0.8 V/MSE. We can therefore assume that this wave is due to the water decomposition. These results are in agreement with those of Yano *et al* [37]. The stability range of the BDD electrode in this medium ( $NaOH$ ) extends to 2.6 V/MSE.

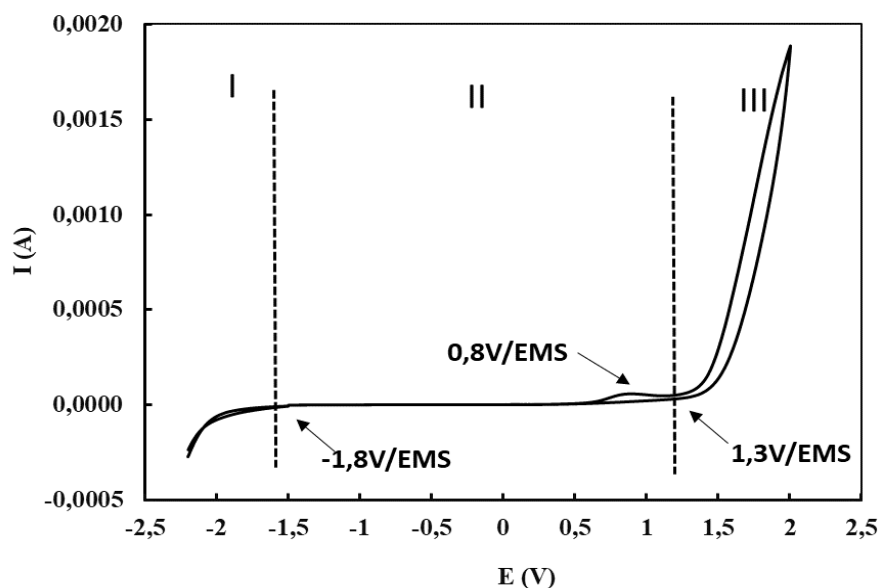
### 3.3 Voltammetric study of methyl orange oxidation

#### 3.3.1 Study of methyl orange oxidation in sulfuric acid medium.

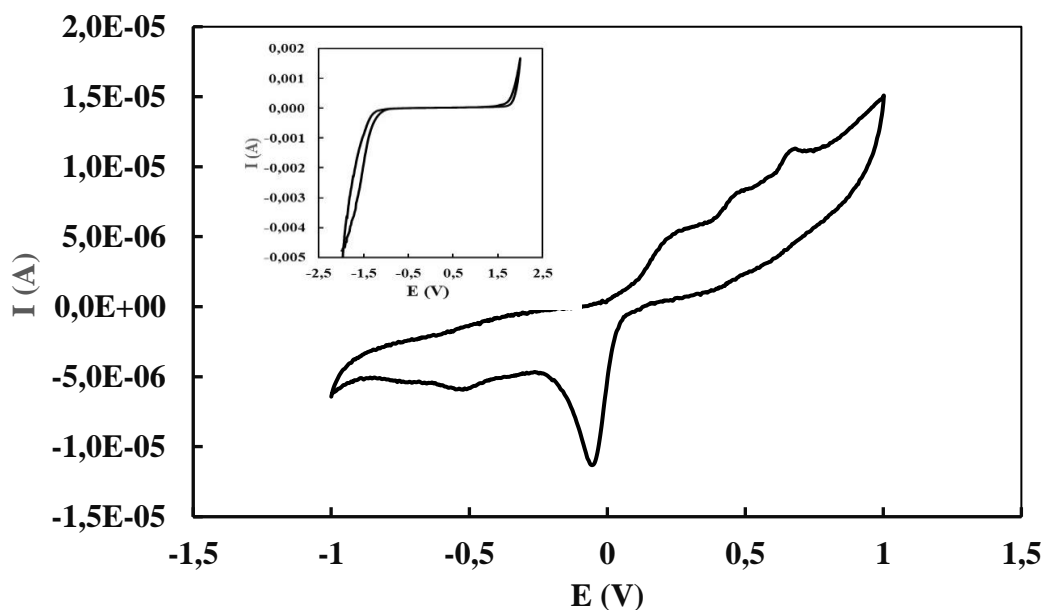
##### 3.3.1.1 Cyclic voltammogram of methyl orange in sulfuric acid medium.

The voltammogram curve presented in Figure 4 shows the behavior of the BDD electrode in the presence of methyl orange (50 mg/L) in  $H_2SO_4$  medium (0.1 M) at 50 mV/s, with the cyclic

voltammogram in the absence of methyl orange in the inset. In the presence of the orange methyl, we observe an oxidation peak at 0.22 V/MSE followed by two oxidation waves at 0.46 and 0.68 V/MSE respectively, in the forward potential scan.



**Figure 3:** Voltammetric curve of BDD in NaOH (0.1 M) at 20 mV/s



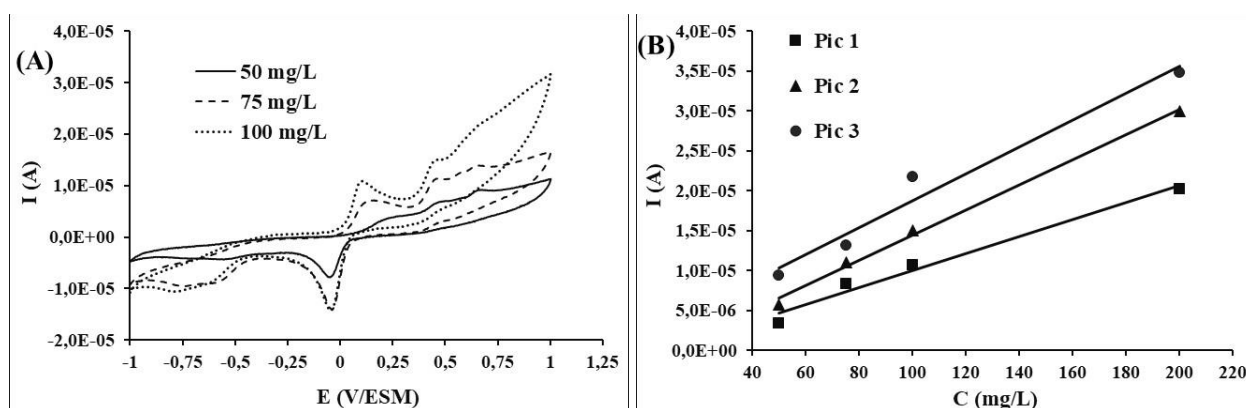
**Figure 4:** Cyclic voltammogram of BDD in H<sub>2</sub>SO<sub>4</sub> medium (0.1 M) in the presence of MO (50 mg/L) at 50 mV/s

In the backward potential scan, we observe a peak and a reduction wave at -0.053 V/MSE and -0.521 V/MSE, respectively. These different peaks are located in the electroactivity range of the BDD electrode in sulfuric acid medium. This could indicate a direct oxidation of methyl orange promoted by an electronic exchange between the BDD surface and methyl orange. This oxidation could correspond to that of the azo group and would take place in two steps. A first mono-electronic step

quasi-reversible in the less positive potential range (0.22 V/MSE) and a second irreversible step at more positive potentials, almost simultaneously with the oxygen evolution reaction.

### 3.3.1.2 Influence of concentration

Figure 5A shows the effect of MO concentration on its oxidation. An increase in the anodic and cathodic peak current intensity is observed with methyl orange concentration. The curves of the oxidation peak current versus the methyl orange concentration are shown in 5B. The curves obtained are linear lines of equations 1, 2, and 3. The determination coefficients of ( $R^2$ ) of these lines are all close to 1. This linearity shows that the different anodic peaks observed are related to the methyl orange oxidation. This linearity also shows that the BDD electrode can be used for a quantitative determination of methyl orange.



**Figure 5:** (A) BDD cyclic voltammogram of different MO concentrations at 50 mV/s, (B) Plot of oxidation peak currents as a function of MO concentration

$$I_{P1} = 3.10 \cdot 10^{-7}[\text{MO}] - 5.10 \cdot 10^{-6} \quad (R^2 = 0,9558) \quad (1)$$

$$I_{P2} = 2.10 \cdot 10^{-7}[\text{MO}] - 3.10 \cdot 10^{-6} \quad (R^2 = 0,9946) \quad (2)$$

$$I_{P3} = 2.10 \cdot 10^{-7}[\text{MO}] - 5.10 \cdot 10^{-6} \quad (R^2 = 0,9868) \quad (3)$$

### 3.3.1.3 Influence of scan rate

Figure 6 shows the influence of the potential scan rate on the current intensity of the methyl orange oxidation peaks. The voltammetric curves obtained were recorded in the presence of 50 mg/L methyl orange in  $\text{H}_2\text{SO}_4$  (0.1 M) medium. An increase in current intensity is observed with increasing potential scan rate. There is also a shift in the potentials of the anodic peaks to positive values. This shows that the potential scan rate influences the MO oxidation.

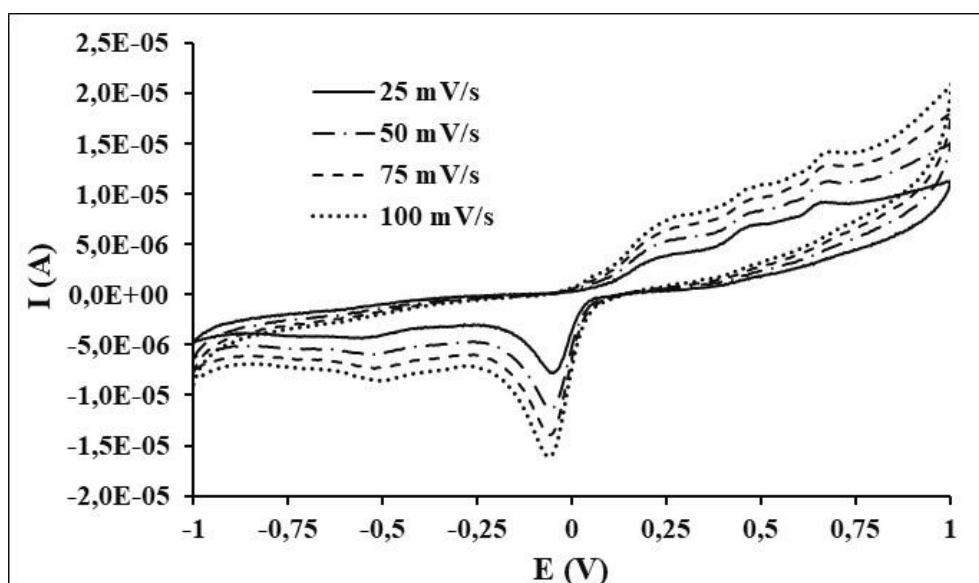
The reversibility and kinetic regime (adsorption or diffusion) can be studied by analyzing the dependencies of peak current intensity ( $I_p$ ) on the square root of the scan rate ( $v^{1/2}$ ) [38] and the logarithm of peak current intensity ( $\ln I_p$ ) on the logarithm of the scan rate ( $\ln v$ ) [39].

For the first approach, curves of  $I_p$  versus  $v^{1/2}$  (Figure III.7) were plotted. The curves obtained are straight lines of equations 4, 5 and 6 for peaks 1, 2 and 3 respectively:

$$I_{Pa1} = 3.10 \cdot 10^{-6} v^{1/2} - 4.10 \cdot 10^{-5} \quad ; (R^2 = 0,9289) \quad (4)$$

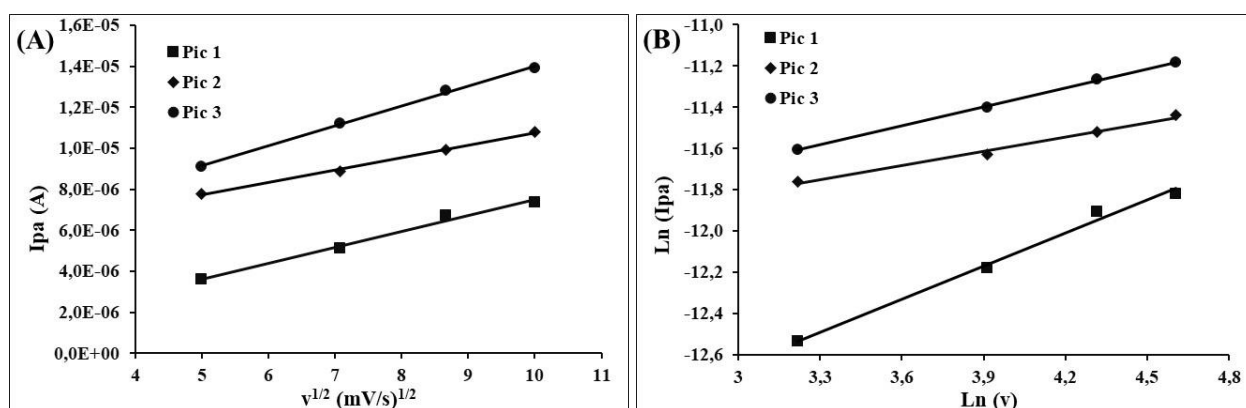
$$I_{Pa2} = 7.10 \cdot 10^{-6} v^{1/2} - 8.10 \cdot 10^{-5} \quad ; (R^2 = 0,861) \quad (5)$$

$$I_{Pa3} = 9.10 \cdot 10^{-7} v^{1/2} - 6.10 \cdot 10^{-5} \quad ; (R^2 = 0,9846) \quad (6)$$



**Figure 6:** Cyclic voltammogram of BDD in the presence of 50 mg/L OM at different scan rates in sulfuric acid medium (0.1 M)

These curves show a linear dependence between the current intensity of the oxidation peaks and the square root of the scan rate. However, the lines obtained do not intercept the origin of the axes which indicates that the electrooxidation process of MO could be controlled by adsorption and preceded or followed by a homogeneous chemical reaction [38].



**Figure 7:** (A) Curves of oxidation peak currents as a function of the square root of the scan rate; (B) Curves of the logarithm of oxidation peak currents as a function of the logarithm of the scan rate in potential.

The second approach is summarized by plotting the curve  $\ln I_p = f(\ln v)$ . The resulting curves presented by Figure 7 are straight lines described by equations 7, 8 and 9 for peaks 1, 2 and 3 respectively.

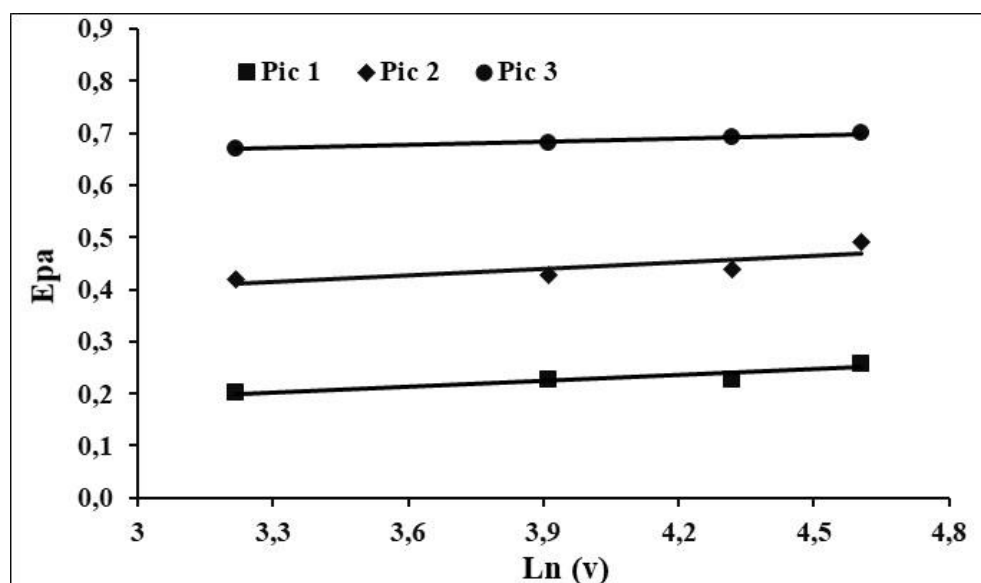
$$\ln(I_{Pa1}) = 0,4646 \ln v - 13,862 \quad ; \quad R^2 = 0,9911 \quad (7)$$

$$\ln(I_{Pa2}) = 0,2540 \ln v - 12,607 \quad ; \quad R^2 = 0,9932 \quad (8)$$

$$\ln(I_{Pa3}) = 0,2686 \ln v - 12,380 \quad ; \quad R^2 = 0,9882 \quad (9)$$

The slopes of the  $\ln I_p = f(\log v)$  curves are equal to 0.464; 0.254 and 0.268 for peaks 1, 2 and 3 respectively. These slope values are all close to 0.5, so the MO electrooxidation process is diffusion-limited. The results of both approaches indicate that an adsorption-diffusion phenomenon occurs.

Figure 8 shows the oxidation peak potential ( $E_{Pa}$ ) as a function of the potential sweep rate ( $v$ ).



**Figure 8:** Oxidation ( $E_{Pa}$ ) and reduction ( $E_{Pc}$ ) peak potentials as a function of potential scan rate ( $v$ ) for the MO electrooxidation in  $H_2SO_4$  medium.

The curves of the oxidation peak potentials  $E_{Pa1}$ ,  $E_{Pa2}$  and  $E_{Pa3}$  as a function of the logarithm of the velocity are linear lines with slopes 0.07; 0.05 and 0.02 respectively with a coefficient of determination close to 1. This result allows us to say that the process related to the anodic peaks is slow and controlled by diffusion.

### 3.3.2 Study of MO oxidation in basic medium (NaOH)

#### 3.3.2.1 Cyclic voltammogram of methyl orange in sodium hydroxide medium.

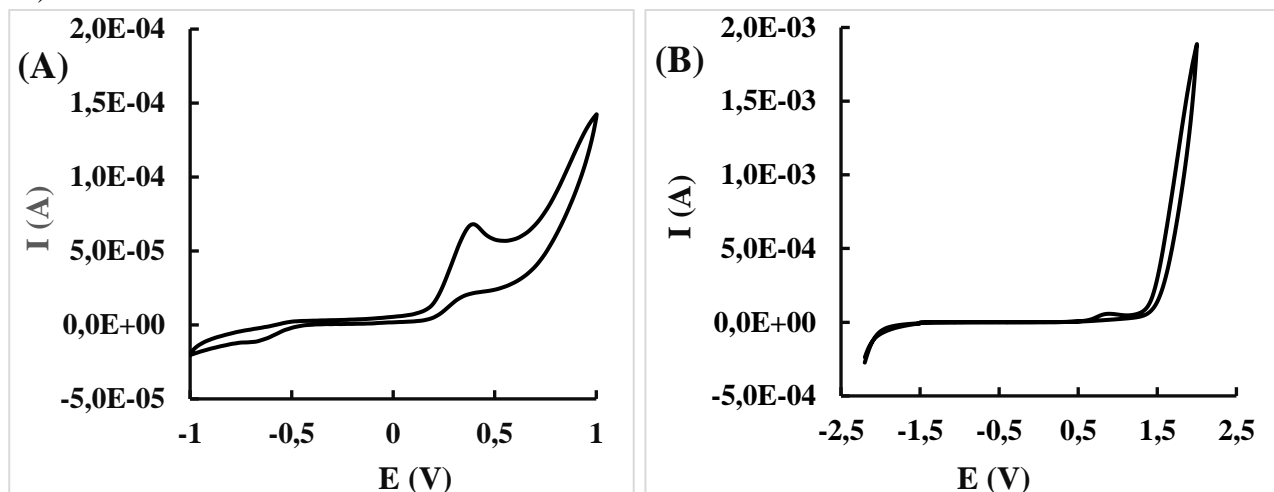
The electrochemical characterization of BDD electrode was performed in NaOH medium (0.1 M) in the presence of methyl orange (75 mg/L) at 50 mV/s in a potential range from -1 to 1 V/MSE. The results obtained are shown in Figure 9A. Figure 9B shows the cyclic voltammogram of BDD in NaOH medium in the absence of methyl orange. In the presence of MO, we observe in the potential range from -0.5 to 0.15 V/MSE an almost zero current reflecting an absence of reaction at the electrode surface. Then, in the forward potential scan, we note a rapid increase of the current from 0.15 V/MSE followed by an oxidation peak at 0.41 V/MSE. This peak is characteristic of MO oxidation. The oxidation peak of MO is located in the electroactivity range of DDB. This could indicate direct oxidation of methyl orange promoted by direct electronic exchange between the BDD surface and methyl orange. This result is consistent with the results obtained in acidic media. The presence of a single peak under the same conditions as the acidic medium could suggest that MO oxidation would be less favored in the basic medium.

#### 3.3.2.2 Influence of the concentration

Concentration variation is a frequently used kinetic parameter in the electrooxidation of organic compounds. The study of the concentration variation of methyl orange on DDB electrode was performed for a range of MO concentration from 50 to 100 mg/L. Voltammeter curves were recorded

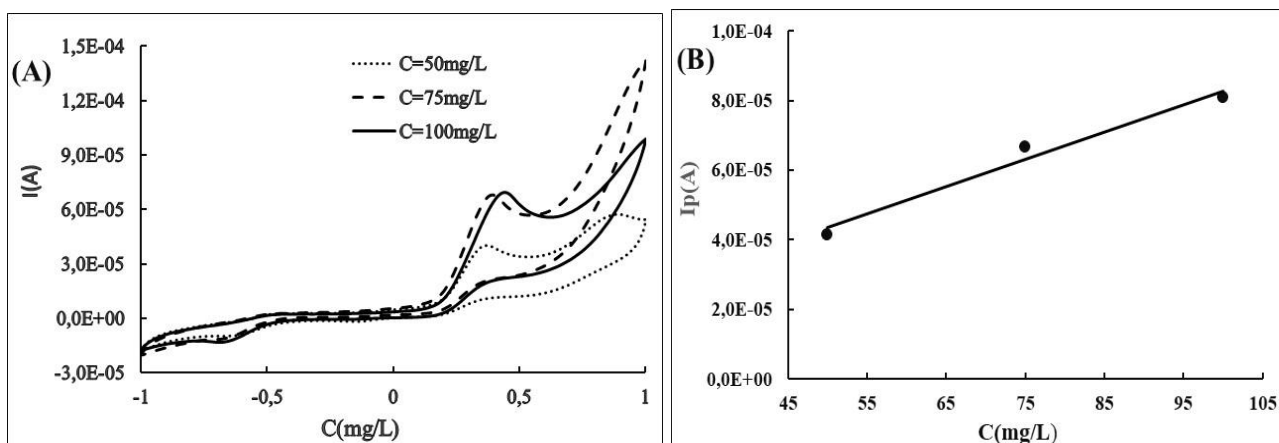


under a potential scan rate of 50 mV/s in a potential range from -1 V/MSE to 1 V/MSE in NaOH (0.1 M) medium.



**Figure 9:** Cyclic voltammogram of DDB in NaOH medium (0.1 M) in the presence of 75 mg/L MO (A) and in the absence of MO (B) at 50 mV/S

Figure 10A shows the results obtained. An increase in the intensity of the oxidation peak is observed with increasing concentration of methyl orange. The curve of the oxidation current intensity versus MO concentration (Figure 10B) is a straight line with determination coefficient  $R^2 = 0.9747$  close to 1. This result indicates a good linearity between the oxidation peak current and MO concentration. This shows that the observed oxidation peak is related to methyl orange oxidation. These results also show that BDD can be used for quantitative determination of methyl orange in basic media.

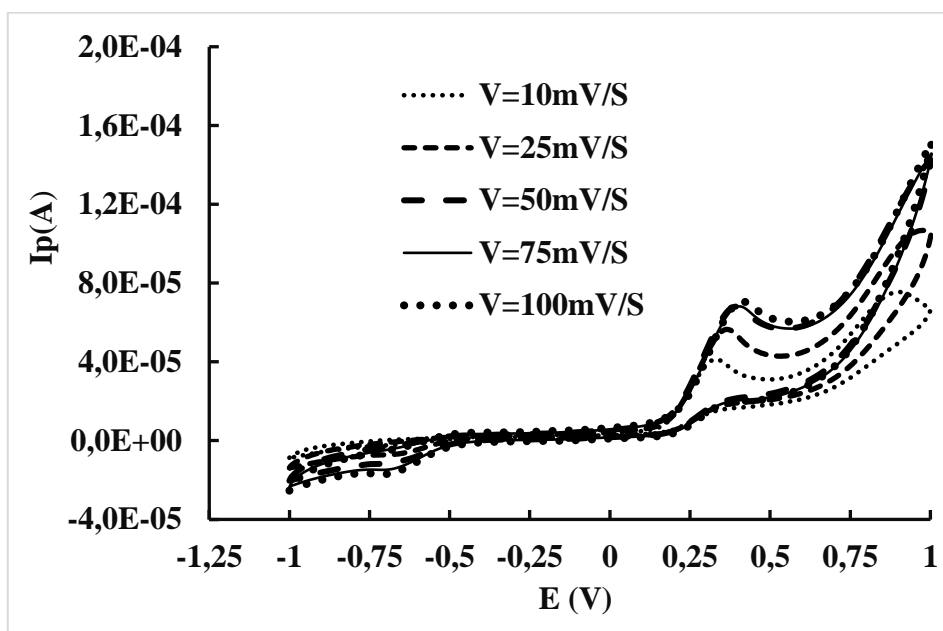


**Figure 10:** (A) Cyclic voltammogram of DDB in NaOH medium (0.1 M) in the presence of different MO concentrations at 50 mV/s, (B) Peak current intensity as a function of MO concentration

### 3.3.2.3 Influence of scan rate

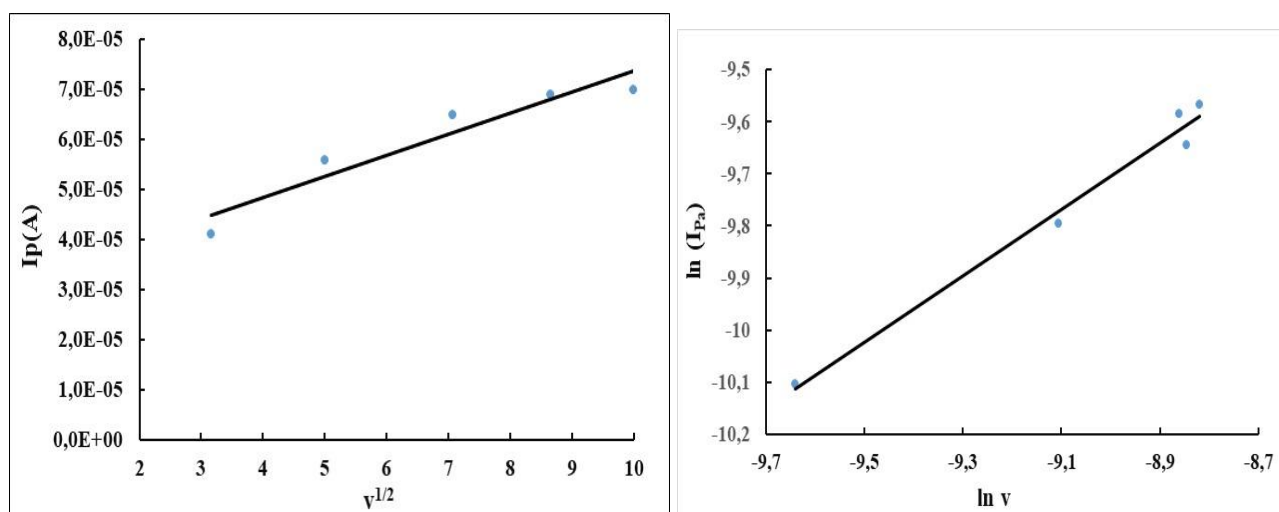
Figure 11 shows the voltammograms obtained for different scan rates in NaOH medium (0.1 M) containing 75 mg/L MO in the potential range between -1 and 1 V/MSE. This figure shows that the current density increases with the scan rate in potential. To determine the kinetic regime of MO oxidation in basic medium, the curve  $I_p = f(v^{1/2})$  was plotted in Figure 12A. The curve obtained is a straight line with determination coefficient  $R^2 = 0.9105$  that does not pass through the origin. This result indicates that the electrooxidation process of OM could be controlled by adsorption and preceded

or followed by a homogeneous chemical reaction. This result is consistent with the results obtained in acidic media.

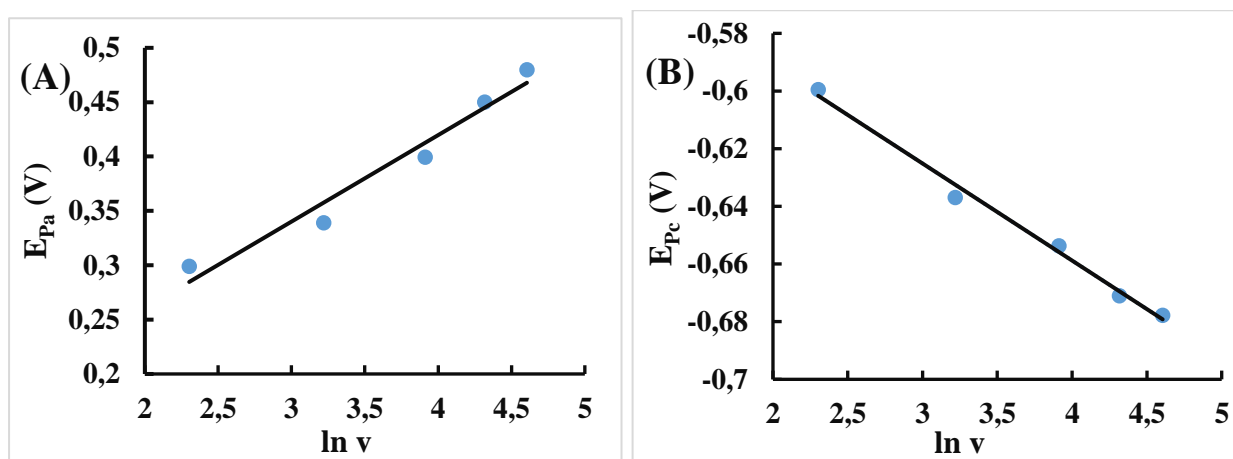


**Figure 11:** Cyclic voltammogram of BDD electrode in NaOH (0.1 M) containing 75 mg/L MO at different scan rates

Figure 12B represents the curves of  $\ln I_p = f(\ln v)$ . This curve is a straight line with slope 0.6 close to 0.5 and determination coefficient  $R^2 = 0.9827$ . This confirms the MO oxidation is governed by a diffusion-limited electrode process [40]. This result is consistent with the results obtained in acidic media. Figure 13 shows the curves  $E_{Pa} = f(\ln v)$  and  $E_{Pc} = f(\ln v)$ . These curves are straight lines with correlation coefficients 0.9101 and 0.9628 for  $E_{Pa} = f(\ln v)$  and  $E_{Pc} = f(\ln v)$  respectively. Moreover, these curves have non-zero slopes, which could justify that MO electrooxidation is slow or irreversible.



**Figure 12:** (A) Evolution of the current intensity of the MO oxidation peak as a function of the square root of the scan rate in NaOH (0.1 M), (B) Evolution of the logarithm of the current intensity of the MO oxidation as a function of the Neperian logarithm of the rate in NaOH (0.1 M)



**Figure 13:** Oxidation ( $E_{pa}$ ) (A) and reduction ( $E_{pc}$ ) (B) peak potential as a function of the logarithm of  $v$  for the electrooxidation of MO in NaOH medium (0.1 M).

## Conclusion

The physical characterization of the DDB electrode carried out by scanning electron microscopy (SEM) revealed a polycrystalline structure, crystals oriented in random ways and stuck together. The electrochemical characterization of the electrode carried out in acid and basic medium showed that it presents a stability against acid and basic corrosion and allows the oxidation of the organic matter. The voltammetric study showed that MO oxidation takes place in the stability range of the supporting electrolyte. This shows that MO oxidation occurs directly through electronic exchange at the electrode-solution interface. This oxidation is controlled by a mixed adsorption-diffusion process and preceded or followed by a homogeneous chemical reaction in acidic and basic media. In the acidic medium, three oxidation peaks are observed in the stability zone. On the other hand, in the basic medium, only one exploitable oxidation peak is observed. This study also showed that the BDD anode can be used for a quantitative determination of methyl orange in acidic and basic media. Thus, the electrochemical method appears to be an adequate means to oxidize methyl orange and thus treat wastewater containing this product.

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