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Article *in* Asian Journal of Research in Chemistry · June 2022

DOI: 10.52711/0974-4150.2022.00037

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ISSN 0974-4169(Print)
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RESEARCH ARTICLE

Kinetic of the Oxygen and Chlorine Evolution Reaction on Platinum Electrodes at Neutral pH

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ABSTRACT:

The platinum anode modified by metal oxides electrodes degrades Abidjan wastewater which contains a high concentration of Cl^-. During this degradation process, the organic polluants are oxidized, O_2 and Cl_2 are produced. The purpose of this study is to contribute to the understanding of these reaction mechanisms by studying the kinetics of O_2 and Cl_2 evolution at neutral pH on Pt. The study was performed by interpreting the voltammograms and Tafel slopes obtained. The voltammetric measurements were carried out using an Autolab Potentiostat from ECHOCHEMIE (PGSTAT 20) connected by interface to a computer. Pt electrode was prepared on titanium (Ti) substrate by thermal decomposition techniques at 400°C. The characterization of the surface of the prepared electrode by scanning electron microscopy and X-ray photoelectron spectrometry showed the presence of platinum on its surface. The results obtained show that the OH· are adsorbed on the active sites of Pt. Then they react to form PtO. Then by reaction between the surface oxygen and PtO, O_2 is produced and the active sites are regenerated. In the presence of low Cl^- concentration, there is a competition between the Cl_2 and O_2 evolution reactions. However, Cl_2 only is produced for high Cl^- concentrations. The kinetics of the evolution reaction of chlorine increases with the concentration of Cl^- and remains constant for concentrations greater than 0.5 M. This study also showed that the chlorine reduction reaction produced in solution is a diffusion-controlled reaction for low scan rates.

KEYWORDS: Oxidation, Platinum, Oxygen, Chlorine, Kinetic.

1. INTRODUCTION:

Electrochemistry is a multidisciplinary science. It is applied in various sectors such as: energy (production and storage), electrosynthesis, corrosion phenomena study, the preparation of reactive metals or non-metals, analysis, or even the world of life[1-8]. It gives rise to a great diversity of industrial applications.

It is continuously exploring new application paths in various fields, including the treatment of urban wastewater, industrial wastewater, agricultural wastewater, hospital wastewater or more recently wastewater from the petrochemical industry[9-11], since biological treatments are becoming impotent in the face of refractory or recalcitrant molecules[12-14]. Activated carbon is widely used to treat organic and inorganic pollutants and also, to immobilize metal ions such as copper, zinc, cadmium or chromium[15-21]. The main limitation of such treatment lies in the fact that in any case, pollutants are not degraded, but concentrated on the activated carbon wich must be subsequently treated in order to regenerate it. Thus, electrochemistry has been

Received on 12.12.2021 Modified on 15.03.2022
Accepted on 19.05.2022
Asian J. Research Chem. 2022; 15(3):213-219.
DOI: 10.52711/0974-4150.2022.00037

shown to be effective in the treatment of wastewater from Abidjan which contains a high concentration of chloride ions during our previous work[11]. However, it was found that during the degradation of organic matter in these waters, there is competition between the oxidation reaction of organic compound and the reactions of oxygen and chlorine production[11,22,23]. This shows that the study of the oxidation mechanisms of organic compound implies the control of the kinetics of dioxygen (O2) and chlorine (Cl2) evolution reactions.
In the literature, several studies have been carried out on the kinetics of these gases evolvement and several different reaction mechanisms have been elaborated showing the complexity of these reactions[24,25]. Faced with this complexity, it is still necessary to carry out investigations to elucidate kinetics of O2 and Cl2 evolution reactions.

However, it should be noted that using this technique implies the electrode choice that will serve as anode. Thus, the platinum (Pt) electrode is the ideal candidate because of its good electrical conductivity and also its resistance to corrosion. Moreover, it is chemically and electrochemically inert and has intrinsic electrocatalytic properties[26,27]. Previous investigations[11] have shown that the coupling of platinum with metal oxides allows to obtain anodes that efficiently degrade wastewater. This work has also shown that the preparation of anodes by thermal decomposition at 400°C from precursor solutions results in electrodes with good electrocatalytic properties for organic compound oxidation.

In order to contribute to the understanding of the kinetics of O_2 and Cl_2 evolution reaction on these coupled electrodes, it appeared necessary to elucidate the kinetics of these gases evolution reactions on the platinum anode. The aim of our work is to study the kinetics of oxygen and chlorine evolution reactions on a platinum electrode prepared by thermal way at 400°C. This study will be carried out at neutral pH because the pH of the wastewater in Abidjan is close to 7[28,29].

2. MATERIAL AND METHODS:

2.1. Material:

The electrochemical measurements were carried out with an Autolab Potentiostat of ECHOCHEMIE (PGSTAT 20) connected by interface to a computer. These measurements were carried out with an electrochemical cell comprising 3 electrodes. The electrodes used consist of the working electrode (prepared platinum anode), a counter electrode (platinum wire) and a saturated calomel reference electrode (SCE).

The chemicals used for the electrochemical study are: Na_2SO_4 (Suprapur Merck) and NaCl (Fluka). Hexachloro-platinum-Hexahydrate ($H_2PtCl_6.6H_2O$) was used as a precursor solution for the preparation of electrodes. All solutions were prepared with distilled water and the experiments were performed at room temperature at 25°C.

2.2. Methods:

A titanium substrate (plate) was cut and roughened by sandblasting to facilitate the adhesion of the precursor solution. Then the platinum (Pt) anode was prepared thermally at 400 °C, after application of the precursor solution on the treated surface of the titanium substrate. In detail, the preparation of the electrodes is as follows:

- Application of the solution by brush, on the treated surface;
- Baking at 80°C for 10 minutes to evaporate the solvent;
- Thermolysis in the oven at 400°C for 15 minutes;
- Removal from the oven and cooling.

The succession of operations constitutes the deposition protocol for a layer. Generally speaking, a certain number of layers are deposited in order to obtain the desired thickness. More than three sequential layers are always deposited to ensure good substrate coverage. The final step consists of a calcination at 400°C for one hour in the ambient atmosphere of the oven.

The reference electrode is placed in a capillary luggin whose end is placed very close to the working electrode to avoid the ohmic drop. The area of the counter-electrode is at least 100 times larger than that of the working electrode whose surface is 1 cm^2 in order not to modify the molar concentration of the electroactive species during the electrochemical study. The ohmic drop correction of the polarization curves was performed by assuming that the experimentally observed overpotential η (V) is given by the following equation:

$$\eta = a + b \ln j + jR \quad (1)$$

Where: a (V) is the Tafel constant; b (V. dec^{-1}) is the Tafel slope;

j ($A.cm^{-2}$) is the current density and R ($\Omega.cm^{-2}$) is the specific resistance of the total uncompensated area of the system assumed to be constant and independent of current.

The derivative of equation (1) with respect to the current density gives equation (2) from which b and R can be easily obtained by plotting dη / dj as a function of 1 / j.

$$\frac{d\eta}{dj} = \frac{b}{j} + R \quad (2)$$

The knowledge of R allows the correction of the ohmic drop of the experimental overpotential by subtraction of jR according to the following equation:

$$\eta_{corr} = \eta - jR \quad (3)$$

In the numerical calculation, the derivative dη/dj was replaced by Δη/Δj calculated from each pair of consecutive experimental points.

3. RESULTS AND DISCUSSION:

3.1. Physical characterization of the prepared electrode:

The surface morphology of the titanium support and the surface of the prepared electrode was characterized by scanning electron microscopy. The results obtained are shown in Figure 1. After sandblasting, the titanium surface becomes rough as shown in Figure 1A. The roughened titanium surface can facilitate the attachment of deposits as reported in the literature[23,30,31].

In Figure 1B, is presented the scanning electron microscopy results of the Pt electrode. In this image, the surface of the titanium plate is not visible; this shows that the entire surface of the titanium is completely covered by the different deposits. The platinum electrode, presents a smooth, compact and almost homogeneous surface[25,26].

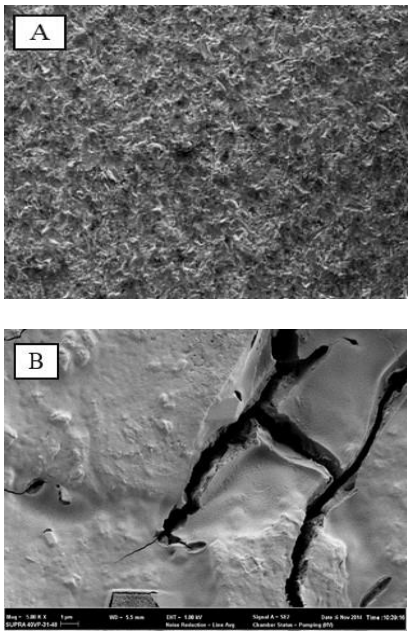


Figure 1. Surface appearance of the sandblasted titanium (A) and the platinum electrode (B)

In order to determine the chemical composition of the electrode surface, X-ray photoelectron spectrometry (XPS) measurements were performed. Figure 2 shows the results obtained. On the XPS spectrum, we note the presence of three intense peaks at 70, 312 and 335 eV, which characterize the presence of platinum on titanium. Carbon is also present with a peak observed at 286 eV. It could come either from a poisoning of the measuring apparatus, or from the isopropanol used in the preparation of the precursors, or from a contamination in the furnaces used to prepare the electrodes. Two very close peaks are observed at 218 and 224 eV. These two peaks would characterize the presence of platinum oxide.

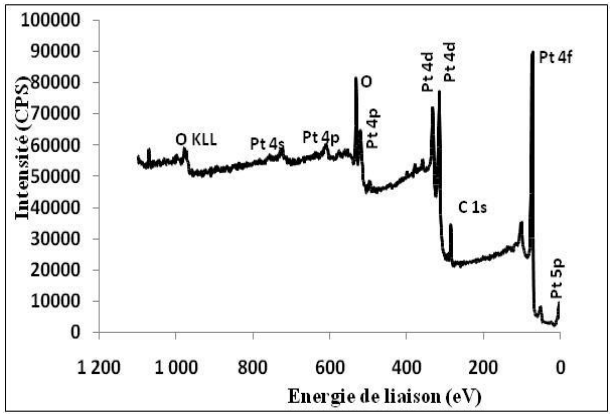


Figure 2. XPS spectrum of the platinum electrode

3.2. Electrochemical characterization of the electrode:

In Figure 3, the cyclic voltammogram obtained with the prepared electrode (Pt) in the potential range from - 0.25 to 1.5 V is presented. This cyclic voltammogram shows several regions. From - 0.25 to - 0.1 V, we have a reduction current which corresponds to the dihydrogen evolution reaction. Then from - 0.1 to 0.45 V, no peak is observed. This area is characterized by an almost zero current and corresponds to the double layer domain. Then from 0.45 to 1.08 V, we observe an increase of the current density in the forward potential scan on the voltammogram (inset of Figure 3). This wave corresponds to the formation of an oxide layer on the electrode surface. A reduction peak is present in the backward potential scan of the voltammogram around 0.3 V. From 1.08 V, a rapid evolution of the current characterizing the oxygen evolution reaction is observed.

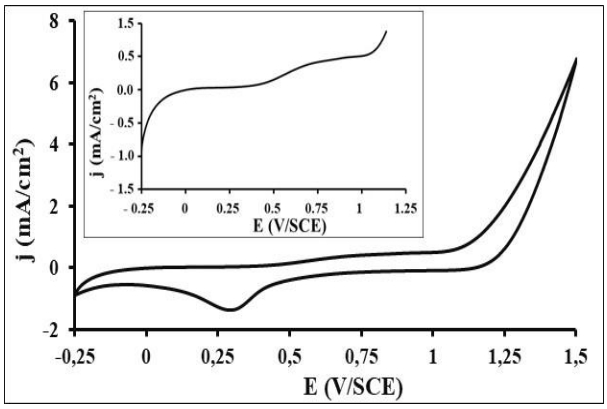


Figure 3. Cyclic voltammogram of platinum in 0.1 M Na_2SO_4 at 10 mV/s.

In Figure 4A, the current density of the reduction peak is plotted against the current density of the average oxidation wave (platinum oxide formation) from 0.45 to 1.08 V. The curve obtained is a straight line with a determination coefficient of 0.996 which passes through the origin of the reference frame. This means that the oxidation wave is proportional to the current of the reduction peak. The reduction peak therefore corresponds to the reduction of the oxide layer formed.

Figure 4B shows the influence of the scan rate on the current density related to the reduction peak. It can be seen that the current density related to the reduction peak evolves linearly with the scan rate in potential. This indicates that the observed peak is characteristic of platinum at neutral pH and that the process is related to the adsorption of chemical species (OH) on the platinum surface.

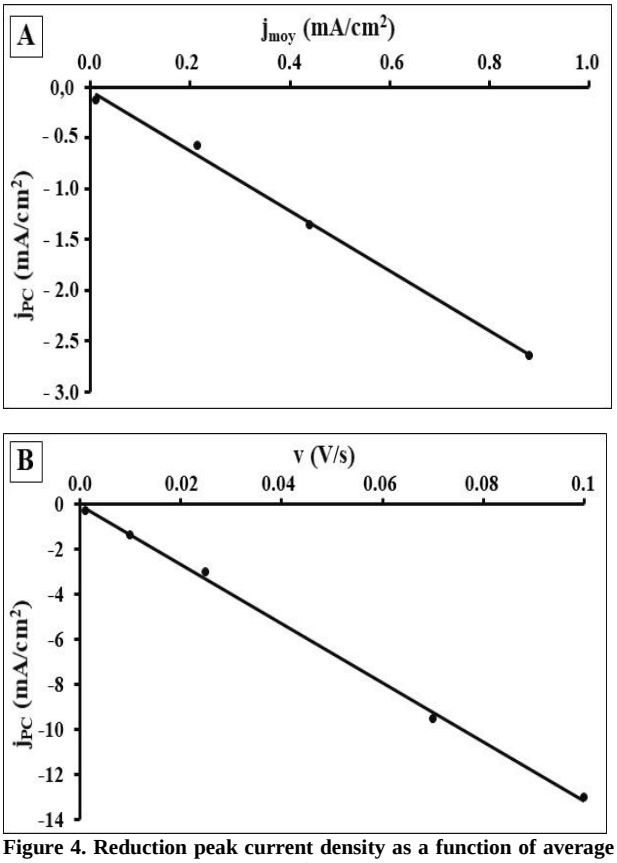


Figure 4. Reduction peak current density as a function of average oxidation wave current density from 0.45 to 1.08 V (A) and as a function of scan rate (B) in 0.1 M Na_2SO_4 medium at 10 mV/s.

The Tafel slope for O_2 evolution reaction was determined from the recorded voltammograms and a value of 173 mV/dec was obtained. This value shows that the kinetics of O_2 evolution reaction is slower in neutral media than in acidic and basic media. According to our previous work, a value of Tafel slope equal to 120 mV/dec was obtained in acidic and basic media[32].

This study showed that in neutral medium, platinum oxide is formed before the oxygen evolvement. These results indicate that the mechanism of oxygen evolution reaction in neutral medium on the platinum electrode is the same as that observed in acidic medium[32]. Thus the mechanism of oxygen evolution reaction in neutral medium on a platinum anode is:

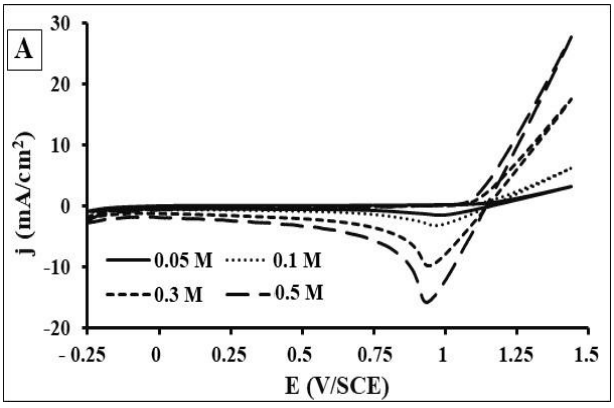
$$Pt + H_2O \longrightarrow Pt(OH) + H^+ + e^- \quad (4)$$

$$Pt(OH) \longrightarrow PtO + H^+ + e^- \quad (5)$$

$$2\,PtO \longrightarrow 2Pt + O_2 \quad (6)$$

With:
Pt: active sites of platinum; OH and O: adsorbed reaction intermediates.

Figure 5A shows the cyclic voltammetries of platinum at different NaCl concentrations. A rapid increase in current density is observed from 1.05 V onwards, for a concentration of 0.1 M. The intensity of this oxidation current increases with the NaCl concentration. A potential of 1.3 V was chosen and the oxidation current density (j_{ox}) was studied as a function of NaCl concentration. The results obtained are shown in Figure 5B. A straight line of determination coefficient 0.999 passing through the origin of the benchmark was obtained. Thus the oxidation current is related to the concentration of Cl^-. This confirms that the current obtained is the oxidation current of Cl^- to Cl_2. The current density of the reduction peak (j_{red}) was also studied as a function of concentration. The result obtained is shown in Figure 5B. The curve obtained is a straight line that passes through the origin of the benchmark. This shows that this reduction peak is the reduction peak of Cl_2 formed into Cl^-.



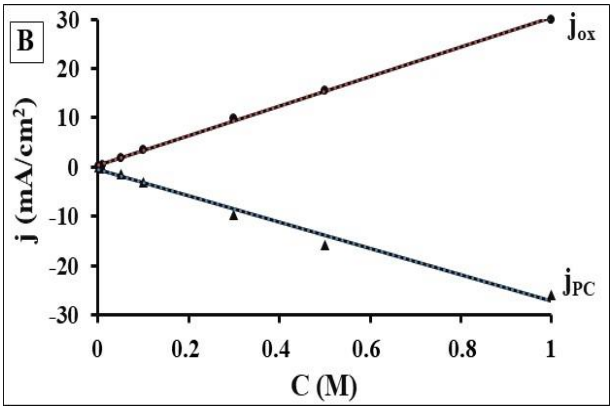


Figure 5. (A) Cyclic voltammograms of Pt in NaCl medium; (B) Reduction peak current density (Jred) and oxidation current density (Jox) at E = 1.3 V as a function of NaCl concentration; 20 mV/s scan rate.

The forward potential scan of the cyclic voltammograms in the presence of NaCl have been plotted on a reduced scale in Figure 6. This figure Indicates the presence of the oxidation wave of platinum to platinum oxide for low concentrations of NaCl (0.01 M and 0.05 M). However this wave disappears for high concentrations of NaCl. The disappearance of this wave with increasing chloride concentration could indicate that chloride influence the kinetics of the platinum oxide formation and reduction process[33]. When the concentration of chloride increases, the electrode surface tends to be completely covered by chloride which would significantly delay the platinum oxide formation/reduction process[22]. This is due to the specific adsorption of chloride ions on the surface of the platinum electrode in a competitive manner with oxygen.

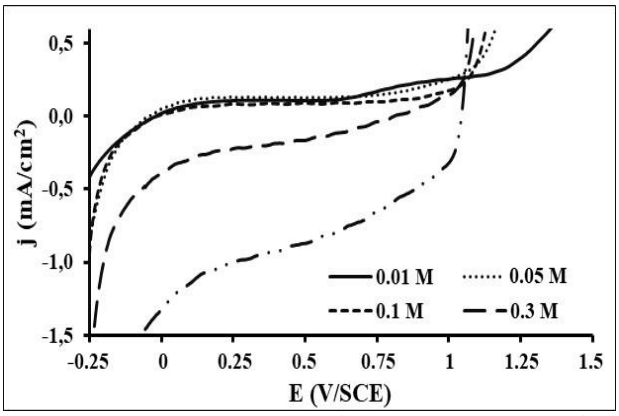
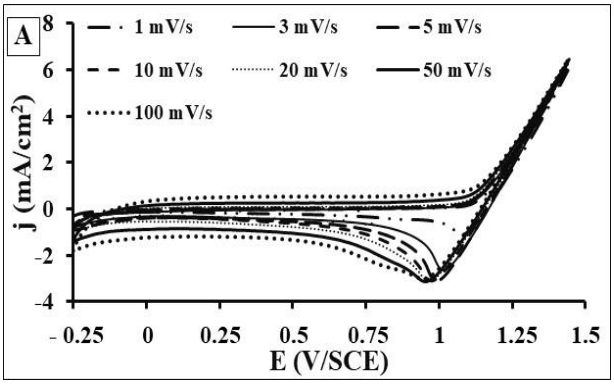


Figure 6. Linear voltammograms of Pt in NaCl; scan rate of 20 mV/s

Figure 6 also shows that the potential for the onset of Cl_2 evolution reaction decreases as the Cl^- concentration increases. The value of this potential was measured for different concentrations. The results obtained are recorded in Table 1. These values show that the more the solution is concentrated in Cl^-, the faster chlorine is produced. Thus the kinetics of chlorine production is faster in the presence of high concentration of Cl^- than in the presence of low concentration of Cl^-.

Table 1. Chlorine evolution reaction potential at j = 0.5 mA/cm² in NaCl medium

NaCl concentration (M)	0.01	0.05	0.1	0.3	0.5	1
E (V/SCE)	0.915	0.753	0.729	0.691	0.683	0.673



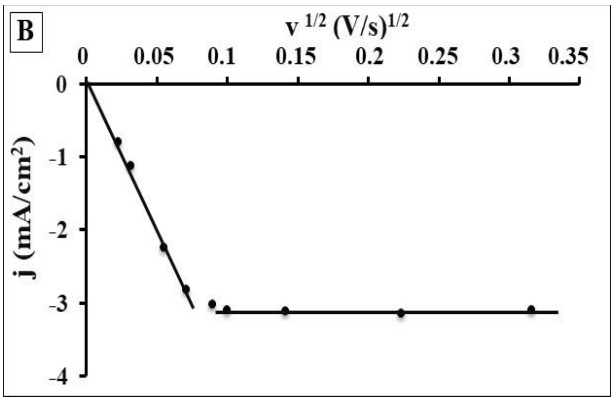


Figure 7. (A) Cyclic voltammograms of the electrode in 0.1 M NaCl; (B) Current density of the reduction peak (j_{PC}) as a function of the square root of the scan rate

The measurements in Figure 7A were performed in 0.1 M NaCl solution in the potential range from - 0.25 to 1.45 V at different scan rates. It can be seen that the current density of the reduction peaks hardly varies for high scan rates. It has a value of about -3.1 mA/cm². But for low scan rates, it decreases in absolute value with the scan rate. To better analyze the evolution of the reduction peak as a function of the scan rate in potential, its current density has been plotted as a function of the square root of the scan rate (Figure 7B). Figure 7B shows that the current density of the observed reduction peak (j_{PC}) is proportional to the square root of the scan rate in potential for low scan rates and constant for high scan rates. Thus the results obtained show that the chlorine reduction reaction is a diffusion controlled reaction for low scan rates, according to the literature[34, 35]. At high scan rates, for a Cl^- concentration of 0.1 M, the chlorine produced could saturate the electrolyte with chlorine. However, it is the dissolved chlorine that is reduced, hence the constant reduction peak current

density obtained. When the scan rate becomes low, this could lead to a decrease of dissolved chlorine by volatilization with the scan rate. Similarly, when the concentration of chloride decreases, the amount of chlorine produced and dissolved in the solution decreases, hence the decrease in the peak reduction current observed (Figure 5A).

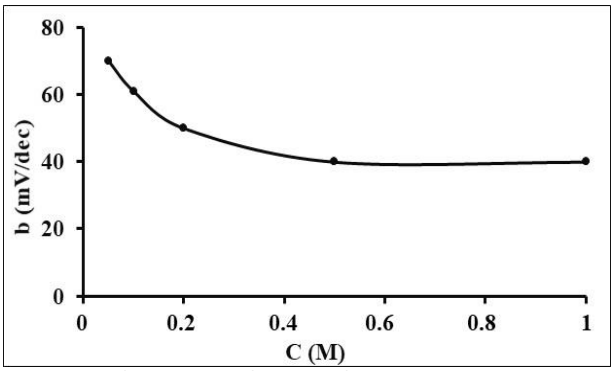


Figure 8. Tafel slope as a function of NaCl concentration; v = 0.5 mV/s

3.3. Mechanism of O_2 and Cl_2 evolution reaction:

The voltammograms obtained with a scan rate of 0.5 mV/s were used to determine the Tafel slopes for chlorine evolution on our electrode. But before any Tafel slope determinations, the ohmic drop correction was performed. The results obtained are shown in Figure 8. When the concentration of NaCl is varied from 0.05 to 0.5 M, the slope of Tafel decreases from 70 to 40 mV/dec. The observations mentioned indicate that the kinetics of the chlorine evolution reaction increases with the concentration of chloride. For electrolytes with high concentrations of Cl^- (0.5 M and 1 M) the Tafel slope remains almost constant (40 mV/dec). Indeed, for high concentrations, we have a good electrical conductivity of the solution and a good electronic transfer. According to the data obtained, this allows to increase the kinetics of chlorine evolution reaction. From 0.5 M the increase of the concentration does not have significantly more influence on the kinetic of the reaction. This could be related to the saturation of the active sites by chemisorption of chloride for high concentration electrolytes.

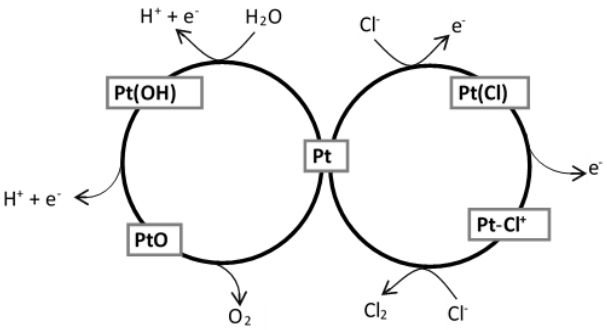


Figure 9. Mechanism of O_2 and Cl_2 evolution reaction in neutral medium with the platinum electrode

After the study of the different voltammograms and the analysis of the different Tafel slopes obtained, the mechanism of figure 9 has been proposed for the production of a mixture of oxygen and chlorine in neutral medium on Pt. Based on the electrode studied in the absence of chloride, it appears that the first step is the electrochemical discharge or decomposition of water molecules leading to the formation of adsorbed hydroxyl radicals. Then the adsorbed hydroxyl reacts by releasing a proton to form platinum oxide (PtO). The active oxygen of the platinum oxide obtained react with each other in a synergistic way to produce dioxygen and regenerate the active sites of the electrode. In the presence of low concentration of chloride, the chloride attaches themselves to the active sites of the platinum in competition with the reaction of the formation of the adsorbed hydroxyl radicals. Under these conditions there will be a simultaneous production of dioxygen and chlorine. In the presence of a high concentration of chloride, there is saturation of the active sites by chemisorption of the chloride and under these conditions it is only chlorine that is produced.

CONCLUSION:

The characterization of the surface of the prepared electrode by scanning electron microscopy and X-ray photoelectron spectrometry showed the presence of platinum on its surface. This work has shown that in the absence of chloride, platinum oxide is formed before the oxygen evolution reaction. In the presence of Cl^-, the Cl^- adsorb on the surface of platinum in competition with the reaction of the formation of platinum oxide that delays or prevents the formation of the oxide layer. In the presence of a low concentration of chloride ions, Cl_2 is produced in competition with the O_2 evolution reaction. However, in the presence of a high concentration of chloride, only chlorine is produced. We note that the kinetics of the chlorine evolution reaction increases with the chloride concentration and remains almost constant (40 mV/dec) for Cl^- concentrations higher than 0.5 M. In addition, this study showed that the chlorine reduction reaction produced in solution is a diffusion-controlled reaction for low scan rates.

CONFLICT OF INTEREST:

The authors declare that there are no conflicts of interest.

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