

**ELECTROCHEMICAL BEHAVIOR AND ANALYSIS OF MONURON  
HERBICIDE IN WATER USING VOLTAMMETRIC METHODS AND PRE-  
ACTIVATED CARBON PASTE ELECTRODE**

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**ABSTRACT**

This research work dealt with the electrochemical behavior and voltammetric analysis of monuron, a phenyl urea herbicide. The sensitive enhancement of the monuron electrochemical signal, using a pre-activated carbon paste electrode, and the explanation of its mechanism were the main findings of this study. Unlike most used herbicides (linuron, diuron, fenuron, etc), monuron was rarely studied before by electrochemical methods. Indeed the square wave voltammetry allowed to optimize and to analyze monuron in water samples; the results showed two linear ranges of concentration: from 1.98 to 0.39  $\mu\text{g mL}^{-1}$  and from 0.35 to 0.08  $\mu\text{g mL}^{-1}$ , with detection and quantification limits of  $\text{LOD} = 0.016 \mu\text{g mL}^{-1}$  and  $\text{LOQ} = 0.054 \mu\text{g mL}^{-1}$  respectively. Besides these quantitative results, the anodic oxidation of monuron has been explained by an irreversible adsorption-controlled process, following a “one electron – one proton” mechanism.

**Keywords:** Monuron electroanalysis; carbon paste electrode; anodic activation; square wave voltammetry.

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## 1. INTRODUCTION

The steadily increasing number of pesticides produced and used in the agricultural industry and other applications have always received a great deal of interest. As emergent organic micro-pollutants, pesticides are responsible for severe public health and environmental consequences[1]. Here, the analytical chemistry plays a vital role in environmental protection, by meeting the challenge of providing a qualitative and quantitative characterization of pesticides[2].

The good effectiveness and the low toxicity of phenylureas with low concentrations made them highly produced chemicals of the herbicide industry and one of the largest group of herbicides used in agricultural and non-agricultural fields[3]. In contrast to these advantages, these herbicides present major drawbacks, their low biodegradability, and their high solubility, which made them widely distributed in the water environment[4].

The presently studied phenylurea herbicide is monuron or 1-(4-chlorophenyl)-3,3-dimethylurea CAS 150-68-5 (structure in Scheme 1), it is a selective unsystemic herbicide [5]. It was introduced in 1952 and has been mostly used for the control of grasses and weeds in non-cropland areas[6]. Monuron is not classifiable as carcinogen to humans (group III)[7], although it was found to produce kidney and liver tumors in male rats, and some of its possible metabolite products (phenol derivatives, aniline) are known for their carcinogenic risk [8].

In soil and plants, monuron is transformed to its metabolites primarily by slow biodegradation, (half-life in field soils ranges from less than 30 days to 166 days)[6]. The lack of information about; the environmental fate of monuron in water; its metabolic products; and its toxicity to humans, made the control of its levels in water, a very important step to prevent any risks that comes from it, and made it important to make sure that it does not exceed the limits put by international community's (all pesticides in drinking water should be lower than  $0.5 \mu\text{g L}^{-1}$ , and that the maximum individual pesticide concentration is  $0.1 \mu\text{g L}^{-1}$ )[9].

Classically phenylurea herbicides are analyzed by chromatographic methods, HPLC method is more successful coupled with different detection systems, such as ultraviolet UV detector[10-11-12], mass spectroscopy MS detector [13], diode array detector (DAD) and post-column photolysis (PPD) after derivatization [14]. Capillary electrophoresis also was used to separate and quantify some phenylurea including monuron in water with UV [15] and an electrochemical detector [16]. These methods permit precise and accurate detection and quantification of trace levels. However, they require time-consuming extraction and cleanup steps, highly trained personnel, expensive equipment, and are not convenient for on-site analysis [17].

Recently, more accurate, simple, inexpensive, rapid and sufficiently sensitive voltammetric methods have been used for monitoring of electrochemical active pollutants [18]. Electrochemical determination of phenyl urea herbicides has been performed with different configurations of working electrodes including carbon pastes [19-20-21], carbon fiber [22], carbon nanotube [23] and electrochemical immunosensors [24].

Carbon paste electrodes became popular for mechanism characterization and quantification of pesticides [25]. A simple mixture of carbon powder (graphite) and a binder (pasting liquid); unmodified carbon paste electrode CPE [19-26]; or chemically ( or biologically) modified carbon paste electrode CMCPE [27-28] are used as working electrodes for voltammetric determination of phenylurea herbicides in environmental matrices. This method is fast; less sophisticated and was used successfully with some phenylurea herbicides. To our knowledge, there is no publication dealing with the electrochemical behavior or the determination of monuron using carbon paste electrode.

Thus, the main goals of this work were the voltammetric characterization of monuron, understand the way it oxidizes on carbon paste electrode by voltammetric techniques (cyclic and square wave voltammetry) and subsequently the elaboration of a simple and direct electroanalytical method for its determination in water by square wave voltammetry (SWV).

## 2. EQUIPMENT AND METHODS

### 2.1. Equipment and reagent

All electrochemical measurements were performed with an Autolab PGSTAT30 potentiostat/galvanostat (Ecochemie, The Netherlands). The experiments were carried out in a three-electrode cell at room temperature ( $25 \pm 1$  C°), using a platinum wire as a counter electrode, Ag/AgCl/KCl (3M) as a reference electrode, and unmodified CPE as a working electrode. Cyclic voltammetry (CV) and square wave voltammetry (SWV) were applied to investigate the electrochemical behavior and analyze monuron.

A  $5 \times 10^{-3}$  M Stock solution of monuron (Sigma-Aldrich; 99.7%) was prepared daily by dissolving this herbicide in an acetonitrile: water (50:50, v: v) mixture. Phosphate buffer solutions (0.2 M) ranging from pH 5 to pH 8 were used as supporting electrolytes. These buffer solutions were prepared by mixing different rates of Na<sub>2</sub>HPO<sub>4</sub> and NaH<sub>2</sub>PO<sub>4</sub>. All other reagents were analytical grade reagent.

### 2.2. Procedures

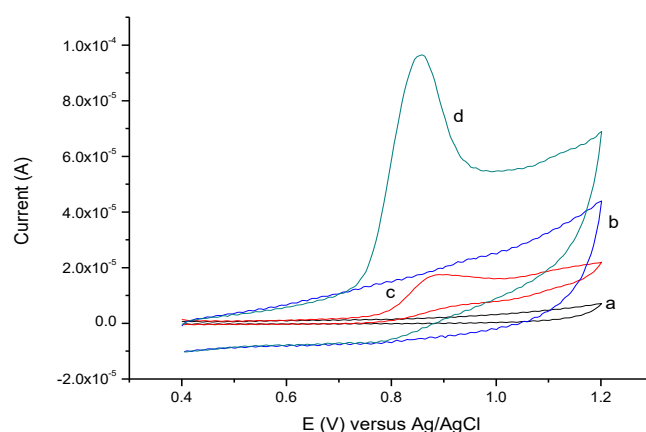
Carbon paste was prepared by mixing spectroscopic-grade graphite (particle size < 50 μm) and Nujol oil (Sigma-Aldrich) at 80%: 20% (w: w). The mixture was manually homogenized in a

mortar for 15 min, and inserted into a plastic tube provided with an electrical contact. The end of the carbon paste (2 mm in diameter) was renewed by pasting out some of it, then smoothed with a moist piece of filter paper and finally rinsed with distilled water. The working electrode was placed in a measuring cell filled with 20mL of phosphate buffer at pH (6.55). Before each measurement, the buffer-immersed working electrode was activated by applying an anodic potential (1.7V) for 30 s. After electrochemical activation, a predetermined amount of monuron was added to the cell containing the buffer solution under stirring. The stirring continues for the selected accumulation time at open circuit conditions, and then the resolution step started after 7 second rest period.

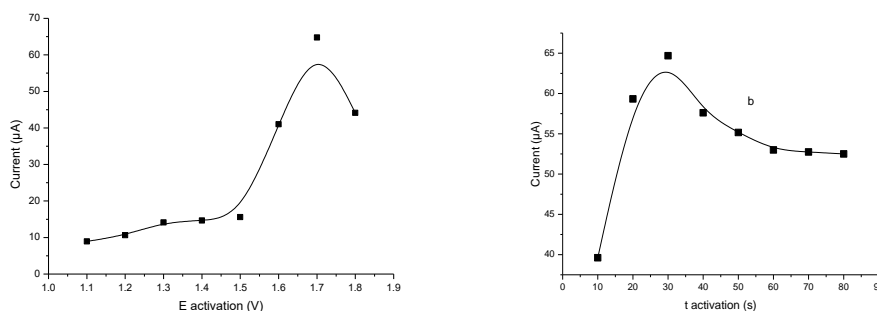
### 3. RESULTS AND DISCUSSION

#### 3.1. Electrochemical behavior of monuron at carbon paste electrode and activated carbon paste electrode

Figure 1 shows the obtained cyclic voltammograms (CVs) for non activated and anodically activated carbon paste electrode in the absence (a and b) and the presence of monuron (c and d) in the potential range of +0.4 to +1.2 V. For the inactivated CPE monuron exhibits one irreversible anodic peak at +0.90V (curve c), while after the anodic activation the peak was better defined and greatly enhanced ( $E_p = +0.85$  V) (curve e).



**Fig.1.** CVs using inactivated CPE (a, c) and anodic activated CPE (b, d);  $E_{\text{activation}}=1.7\text{V}$ ,  $t_{\text{activation}}=30\text{s}$ , in the absence (curves a, b) and presence (c, d) of monuron.  $[\text{monuron}] = 19.66 \mu\text{g mL}^{-1}$ ; phosphate buffer 0.2 M, pH 6.55;  $\nu = 0.8 \text{ V s}^{-1}$



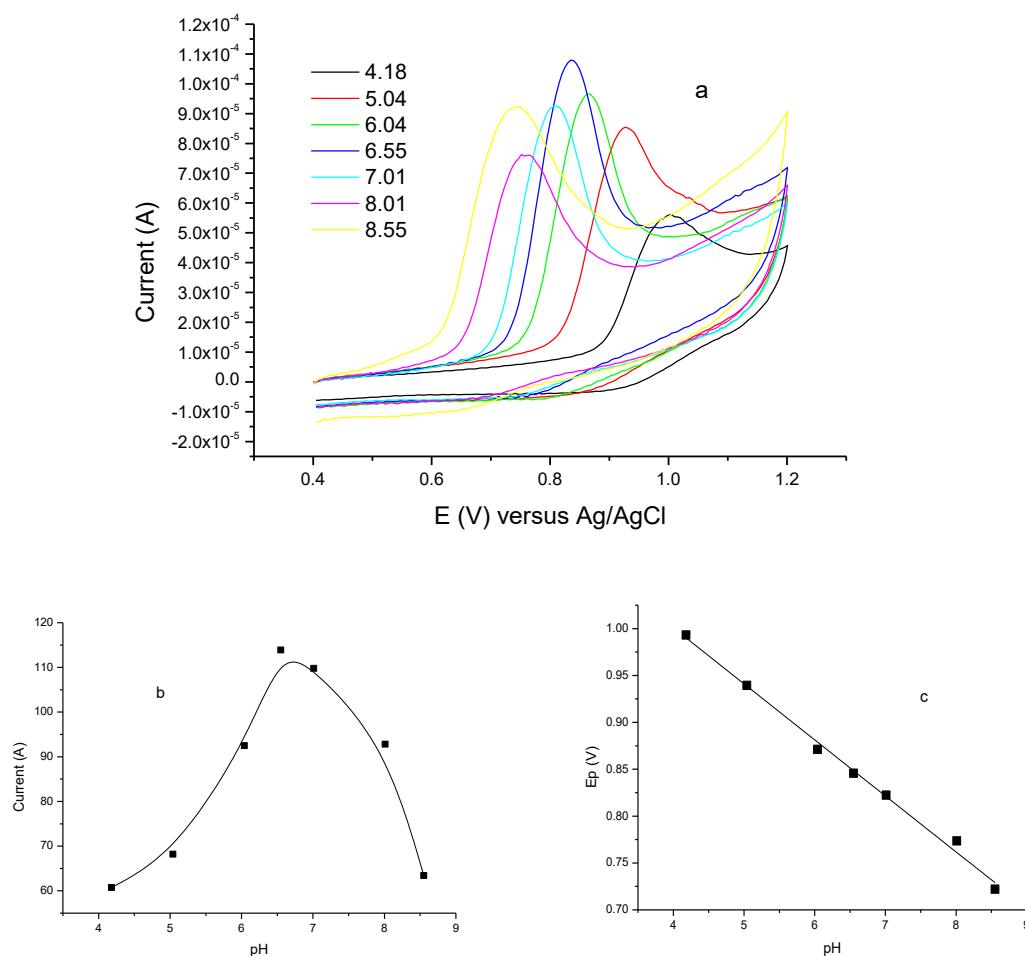
**Fig.2.** Influence of (a)  $E_{\text{activation}}$  and (b)  $t_{\text{activation}}$  on the peak current. [monuron]=  $19.66\mu\text{g mL}^{-1}$ , phosphate buffer 0.2M, pH 6.55

The anodic activation of the carbon paste electrode significantly enhanced the anodic peak and decreases the peak potential by 50 mV, indicating that the activation of the carbon paste have a catalytic effect on the oxidation of monuron. The anodic activation made the surface of the carbon paste more hydrophilic as result of the removal of lipophilic layer from the paste and the formation of surface oxygen-containing functional groups[27].

The effect of time and potential of activation was also estimated, according to figure 2 (a) and (b), the optimal activation conditions were found to be 1.7V for the activation potential, and 30s for the activation time.

### 3.2. Effect of pH

The effect of pH on the electro-oxidation of monuron was investigated over the pH range of 4.00-8.55 using 0.2M Phosphate buffer, after the activation of the working electrode as previously described, knowing that monuron is uncharged compound, and its saturated solution gives a pH of 6.25. Figure 3 shows the cyclic voltammograms of monuron at different pH values (a), peak currents versus pH (b), and peak potentials versus pH (c). The intensity of the oxidation peak increased with pH until it reaches maximum at pH= 6.55, and then begun to decrease (Fig. 3c), pH 6.55 shows the best catalytic effect of the activated carbon past on the oxidation process, and also has a sharper response which means better sensitivity, therefore, it was selected for further experiments.



**Fig.3.** (a) CVs of monuron at different pH values, (b)  $I_p$  as a function of pH, (c)  $E_p$  as a function of pH, [monuron] =  $19.66 \mu\text{g mL}^{-1}$ ;  $[\text{H}_2\text{PO}_4^-/\text{HPO}_4^{2-}] = 0.2 \text{ M}$ ;  $\nu = 0.8 \text{ V s}^{-1}$

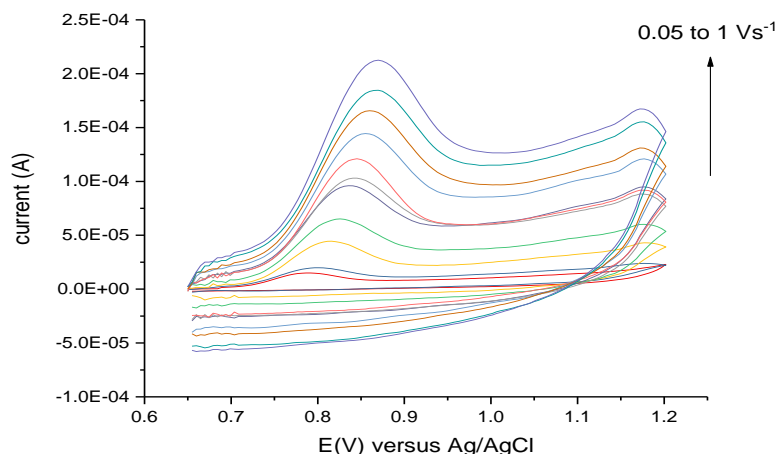
The peak potentials were found to shift linearly towards more negative values with the increase in pH, indicating the intervention of protons in the electrochemical process of monuron (Figure 4a). The slope value in equation (1) is 0.0583, almost matching the theoretical Nernst equation, suggesting that the same numbers of protons and electrons are involved in the electrochemical oxidation of monuron.

$$E_p(\text{V}) = -0.059 \text{ pH} + 1.24 \quad R^2 = 0.996 \quad (1)$$

### 3.3. Effect of potential scan rate

The influence of potential scan rate on the peak current ( $I_p$ ) and potential ( $E_p$ ) was studied for  $29.8 \mu\text{g mL}^{-1}$  monuron in  $0.2 \text{ mol L}^{-1}$  phosphate buffer solution at pH 6.55 over the 0.05 to  $1.00 \text{ V s}^{-1}$ . The figure 4 represents cyclic voltammograms of monuron recorded at different potential scan rates after activation of the working electrode for 30s at 1.7V.

As shown in figure 4, the oxidation peak of monuron is observed in the potential range of 0.78 to 0.88 shifting more towards the positive potential with increasing scan rate and the oxidation peak current steadily increase with an increase in scan rate with a correlation coefficient of 0.9985.



**Fig.4.** CVs of activated carbon paste electrode at scan rates 0.05, 0.08, 0.10, 0.20, 0.30, 0.40, 0.50, 0.60, 0.70, 0.80, 1.00 Vs<sup>-1</sup> in phosphate buffer (pH 6.55) containing 29.8 μg mL<sup>-1</sup> of monuron

The plot of oxidation current versus the scan rate (figure 5a) shows better linearity compared to its dependence on the square root of scan rate (figure 5b), indicating that the electrochemical oxidation of monuron at the activated carbon paste electrode is a surface-adsorption controlled process [28]. And the fact that the plot of  $I_p$  on  $v^{1/2}$  does not cross the origin of the axes indicates that the anodic oxidation is followed by a homogeneous chemical reaction [29].

A plot between the logarithms of scan rate versus logarithms of  $I_p$  (Figure 5c) exhibited a linear relationship, and the respective linear regression can be expressed as Equation. 2, the slope value of 0.85 indicated that the oxidation of monuron occurring at activated carbon paste electrode is an adsorption-controlled process [29-30]. Furthermore the peak potential shifts to higher positive potentials with increasing scan rate (Figure 5d) confirming the irreversibility of the oxidation reaction [23].

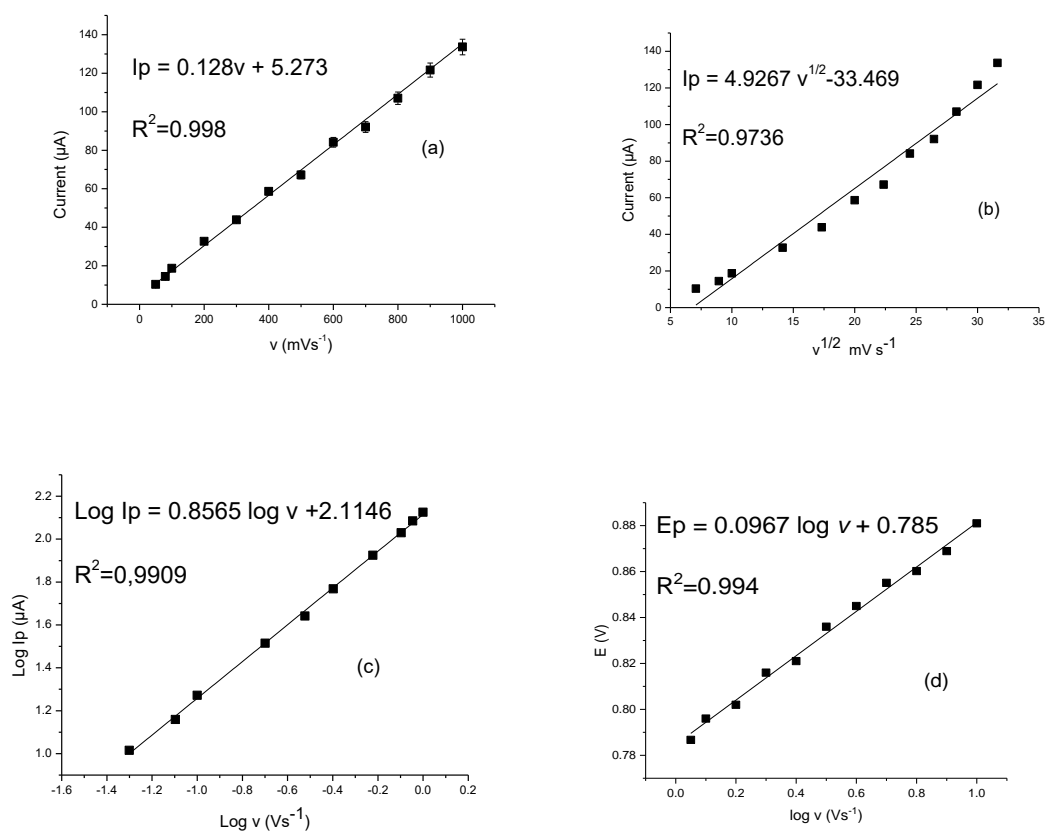
$$\log I_p (\mu A) = 0.856 \log v + 2.115 \quad R^2 = 0.991 \quad (2)$$

$$E_p (V) = 0.097 \log v + 0.785 \quad R^2 = 0.994 \quad (3)$$

For an adsorption -controlled irreversible electrochemical oxidation according to Laviron the number of electrons ( $n$ ) involved in the electrochemical reaction and  $\alpha$  the anodic transfer

coefficient can be calculated experimentally using the slope of the peak potential versus the logarithm of the scan rate,  $k_0$  the standard heterogeneous rate constant of the reaction,  $n$  the number of transferred electrons,  $v$  the scan rate, and is the formal redox potential. Thus the value of  $\alpha n$  can be easily calculated from the slope of  $E_p$  vs.  $\log v$ , the slope is 0.097 (equation 3), with  $T = 298$  K,  $R = 8.314$  J/K mole, and  $F = 96480$  C/mole,  $\alpha n$  was found to be 0.608. For an irreversible reaction  $\alpha$  the anodic transfer coefficient, can be from 0.4 to 0.6[32], so  $n$  and  $\alpha$  were equal to 1 and 0.608 respectively.

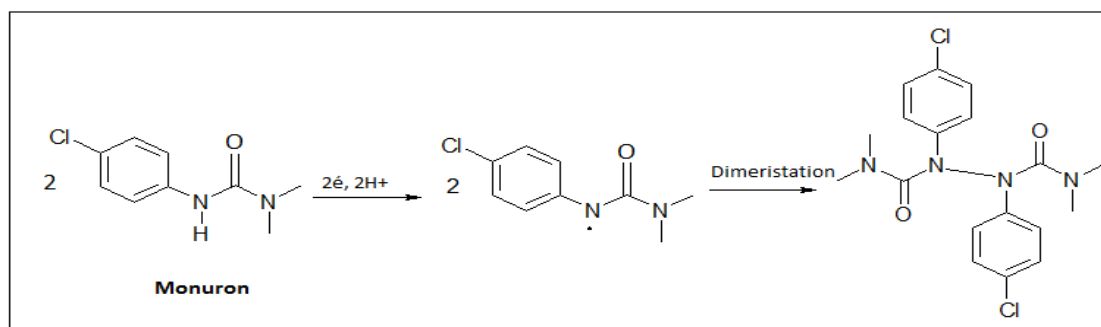
$$E_p = E^{0'} + \left(\frac{2.303RT}{anF}\right) \log\left(\frac{RTk^0}{anF}\right) + \left(\frac{2.303RT}{anF}\right) \log v \quad (4)$$



**Fig.5.** (a)  $I_p$  vs.  $v$ . (b)  $I_p$  vs.  $v^{1/2}$ ; (c)  $E_p$  vs.  $\log v$ ; (d)  $\log I_p$  vs.  $\log v$ ,  $[\text{monuron}] = 29.80 \mu\text{g mL}^{-1}$ ,  $\text{pH} = 6.55$

This result is consistent with the proposed oxidation mechanism for similar molecules; diuron and fenuron [23-32-33]. Scheme 1 shows the proposed mechanism for the oxidation of monuron, it thought to involve one electron and one proton removal pathway to produce a free cationic radical intermediate which in turn dimerises.



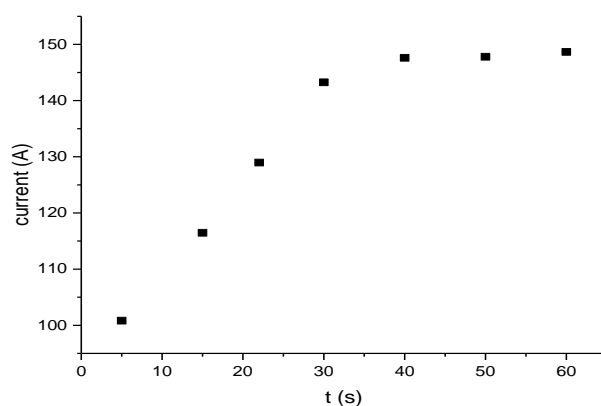


**Scheme 2.** Mechanism of the oxidation of monuron

The proposed mechanism can also explain the catalytic role of the anodic activation of the working electrode. The aggressive oxidation of the carbon surface leads to the profilation of carboxyl functionalities, that generates a variety of surface oxides and oxygen-containing functional groups [35-36], these functionalities can be responsible for the proton removal [37].

### 3.4. Accumulation effect

Since the monuron is been adsorbed on the surface of the electrode, The accumulation influence on the oxidation peak intensity was examined in the range of 5 to 60s, after the electrode activation (30s at 1.7V), in open circuit conditions (figure 6), 40s was the best accumulation time after which the intensity of the peak became constant. With changing the accumulation potential, the peak current of monuron varied slightly, the accumulation potential had no noticeable effect on the peak current of monuron. Therefore the accumulation was carried out at open-circuit conditions.



**Fig.6.** Influence of the accumulation time on the peak intensity, [monuron]= 29.80 $\mu\text{g mL}^{-1}$ , pH=6.55,  $\nu=0.8\text{Vs}^{-1}$

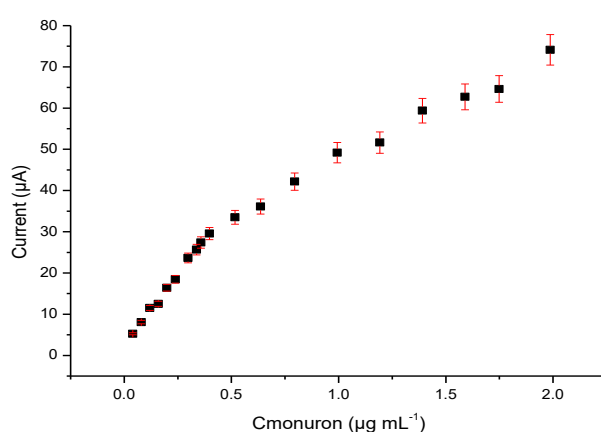
### 3.5. Optimization of square wave parameters

Adsorptive square-wave voltammetry (Ad-SW) was the voltammetric method used to determine monuron concentration in water samples. The important parameters of the SWV technique; frequency and pulse height were investigated. The dependence of peak intensity on frequency in the range of 80-250 Hz has been examined. A linear relationship was obtained between the peak current and the signal frequency between 100-230 Hz due to the increase in the effective scan rate, but at higher frequency values in the peak intensity decreased, this behavior is characteristic for an adsorption controlled irreversible process [38].

Pulse amplitude  $\Delta E_a$  was examined in the range from 10 to 100 mV. The peak heights increased upon an increase of the pulse amplitude in linear regression up to 50 mV, then, it reaches a plateau at between 60 and 90 with a maximum value of  $\Delta E_a = 70$  mV. Hence the frequency of 200 Hz and Pulse amplitude 70 mV were chosen for further experiments.

### 3.6. Reproducibility, Linearity and Detection Limits

The quantitative determination of monuron is based on the linear relationship between the peak current intensity and monuron concentration obtained by the adsorptive square wave voltammetry (Ad-SWV). Calibration graphs were constructed under the optimum conditions described above using activated carbon past electrode: for pH 6.55, scan rate of  $0.8 \text{ V s}^{-1}$ , frequency of 200 Hz, pulse height of 70mV, time preconcentration of 40s. Figure 7 shows two linear ranges of concentrations (figure 7) from  $1.986$  to  $0.39 \mu\text{g mL}^{-1}$  ( $8 \times 10^{-6} \text{ M}$ – $1.98 \times 10^{-6} \text{ M}$ ) and from  $0.357$  to  $0.079 \mu\text{g mL}^{-1}$  ( $1.9 \times 10^{-6} \text{ M}$ – $3.5 \times 10^{-7} \text{ M}$ ).



**Fig.7.**  $I_p$  vs.  $C$  monuron, pH=6.55,  $v = 0.8 \text{ V s}^{-1}$ ,  $t$  accumulation=40s,  $f=200\text{Hz}$ ,  $\Delta E_a = 70\text{mV}$

The limit of detection (LOD) and the limit of quantification (LOQ) were calculated from  $\text{LOD} = 3\text{SD}/b$ , and  $\text{LOQ} = 10\text{SD}/b$ . where (SD) is the standard deviation of intercept of the

linear calibration curve for at least, three independent curves, and (b) is the slope of the calibration curve[35]. The calculated detection and quantization limits are  $0.016 \mu\text{g mL}^{-1}$  and  $0.054 \mu\text{g mL}^{-1}$ , respectively (Table1).

**Table 1.** Analytical parameters for voltammetric determination of monuron

Parameter	SWV	
Concentration interval [ $\mu\text{g mL}^{-1}$ ]	1.986-0.39	0.357-0.079
Intercept [ $\mu\text{A}$ ]	19.837	2.518
Slop [ $\mu\text{A}/\mu\text{g mL}^{-1}$ ]	27.095	69.104
Correlation coefficient $r$	0.9903	0.9949
Precision [SD%]of the intercept <sup>1</sup>	1.172	0.399
Limit of detection [ $\mu\text{g mL}^{-1}$ ]		0.0163
Limit of quantification [ $\mu\text{g mL}^{-1}$ ]		0.0549

<sup>1</sup>the standard deviation of intercept of the linear calibration curve of 4 independent curves

The repeatability and the replicability of the results were evaluated four times for a concentration of  $0.4 \mu\text{g mL}^{-1}$ , for the repeatability the relative standard deviation of current intensity was 1.85% RSD (n=4), and for the replicability the experiments were established in different days (4 days), and different standard solutions, the current measurement gives RSD =3.64%. The low RSD values show a good precision of the proposed method for the determination of monuron by SWV.

### 3.7 Influence of the interferences in the estimation of monuron

Current ( $I_p$ ) of the oxidation of monuron was evaluated, using monuron to ion/molecule ratios of 1:100. The interference of inorganic ions ( $\text{Fe}^{2+}$ ,  $\text{Zn}^{2+}$ ,  $\text{Mg}^{2+}$ ,  $\text{Cu}^{2+}$ ,  $\text{SO}_4^{2-}$ ,  $\text{NO}_3^-$ ), and phenol and hydroquinone a on the peak intensity was tested at the concentration of  $1.748 \mu\text{g mL}^{-1}$  ( $8.8 \cdot 10^{-6} \text{ M}$ ), none of these elements were found to have significant influence on the intensity of the peak.

### 3.8 Application of the proposed method to tap water and distilled water samples

Standard addition method was used in recovery experiments, samples of tap and distilled water were spiked with known amounts of monuron solution for the concentration of  $0.795 \mu\text{g mL}^{-1}$  and  $0.298 \mu\text{g mL}^{-1}$ . The recovery efficiencies (R%) range from 90.83% to 101.58%, and the

relative standard deviation (RSD) value ranges from 1.98% to 5.76% (table 2). This good result regarding the simplicity of the method indicates the efficiency of the developed method to determine monuron in water with a good precision and satisfactory results.

**Table 2.** Assay of monuron in distilled and tap water samples (n=4)

Samples	Added [ $\mu\text{g mL}^{-1}$ ]	Found [ $\mu\text{g mL}^{-1}$ ]	Recoveries %	RSD% <sup>1</sup>
Distilled water	0.7946	0.7890	99.29	1.98
	0.2979	0.2846	95.53	4.81
Tap water	0.7946	0.8072	101.58	5.76
	0.2979	0.2706	90.83	5.6

#### 4. CONCLUSION

The results of this study demonstrate for the first time the evaluation of the electrochemical behavior and the determination of monuron herbicide in water. We used an unmodified carbon paste electrode, after anodic activation of the working electrode and an interfacial accumulation at open circuit. The monuron is oxidized in an adsorption controlled process, with one electron one proton oxidation pathway. The optimum experimental variables involved in the SWV determination of monuron were investigated. The proposed method was linear in two concentration ranges 1.98 to 0.4  $\mu\text{g mL}^{-1}$  and from 0.08 to 0.35  $\mu\text{g mL}^{-1}$ . The LOD = 0.0163  $\mu\text{g mL}^{-1}$  (82.05  $\text{nmolL}^{-1}$ ) and the limit of quantification LOQ = 0.054 (272.33  $\text{nmolL}^{-1}$ ). The proposed method is easy, fast, highly sensitive and did not require any derivatization or the use of organic reagents.

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