

Electrochemical Determination of Diuron in Soil Using a Nanocrystalline Cellulose Modified Carbon Paste Electrode

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An electrochemical sensor based on nanocrystalline cellulose (NC) modified carbon paste electrode (CPE) was developed for the electrochemical detection of diuron in soil samples. Energy dispersive X-ray spectroscopy (EDX) and Scanning electron microscopy (SEM) were used for the elemental and morphological characterization of elaborated CPEs, respectively. Electrochemical studies of diuron using the elaborated CPE were performed by cyclic voltammetry (CV) and square wave voltammetry (SWV). The NC-CPE showed an enhanced electrochemical response, with sensitivity 2.3-fold higher than the signal obtained at the unmodified CPE. Key analytical parameters such as pH, scan rate, and the amount of NC in the carbon paste were investigated and optimized. Excellent results were obtained with a linear range from 4.2 to 47 $\mu\text{mol/L}$ with a detection limit (LOD) of 0.35 μM (S/B = 3). An application of the proposed method to enriched soil samples resulted in a recovery of 96%, showing good stability, reproducibility and reliability of tested sensors.

Keywords: Diuron, carbon paste electrode, cellulose, voltammetry, detection.

1. INTRODUCTION

Diuron [3-(3,4-dichlorophenyl)-1,1-dimethylurea] (Fig. 1) is an herbicide, which belongs to the class of phenylurea. It is widely used to control weeds in a variety of crops, such as sugarcane, coffee, rice, soybean, potato and cotton [1,2]. The persistence and stability of diuron in soil and water were

clearly reported by *Sharma et al.*[3]. Diuron is known to be highly toxic, teratogenic, mutagenic, carcinogenic, and genotoxic after exposition [4-6], [5,6]. Because of its persistence in the environment and its high mobility, diuron can easily contaminate both surface and ground waters. In Burkina Faso, diuron is largely used by cotton farmers due to its accessibility in term of cost and efficacy in weeds control. Meanwhile, because of the lack of wastewater treatment systems, diuron regularly contaminates soil and water. Thus, there is a need to continuously monitor the level of this substance in the environment, notably in waters and soils. Traditional analytical methods used for the determination of diuron include spectrometric [7], chromatographic [8], fluorimetric [9], capillary electrophoretic [10], and particularly electrochemical techniques [11] demonstrated more interest these last years due to their simplicity, selectivity and good reproducibility.. Indeed, electrochemical sensors have been developed for the determination of diuron, using different materials such as graphene, gold nanoparticles and organometallic complexes [13-15]. Likewise, carbon paste electrodes (CPEs) chemically modified have been shown to be useful tools for diuron detection [16]. This work describes the development of an electrochemical sensor based on carbon paste modified with nanocrystalline cellulose (NC) for the detection of diuron. Several factors affecting the electrochemical performances of the proposed sensor were also optimized, including the amount of nanocellulose used as modifier, the scan rate and the pH of supporting electrolyte.

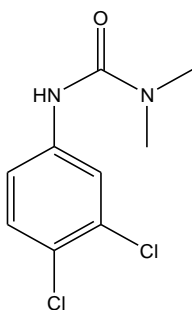


Figure1.Chemical structure of Diuron

2. EXPERIMENTAL

2.1. Reagents and solutions

Nanocrystalline cellulose (NC) was acquired from Sigma-Aldrich. Diuron (80%) was purchased from Louis Dreyfus Commodities agrochemical company, Burkina Faso and used as received. A stock solution of diuron 1 g/L was prepared by dissolving 50 mg of the compound in 500 μ L of methanol and diluting to 50 mL with distilled water. The prepared solution was then diluted to obtain others using solutions. (0.1 M, pH 7.0) Phosphate buffer solution (PBS) was used as supporting electrolyte since diuron bears an amine functional group in its structure and it is expected that the acidity of the supporting electrolyte could affect its redox behaviour. All other aqueous solutions were prepared from analytical grade chemicals, using distilled water.

2.2. Apparatus

Electrochemical measurements were carried out on a DY 2300 electrochemical analyser (Digi-IVY Instruments, USA) running with the DY 2300 EN software and connected to a computer. A three-electrode cell configuration was used, consisting of modified CPEs used as working electrodes, a saturated calomel electrode (SCE) as reference electrode, and a platinum wire as counter electrode. Scanning electron microscopy (SEM) of CPE surfaces was achieved by Field Emission Gun Scanning Electron Microscopy (FEGSEM) on a JSM-6301F apparatus from JEOL (SCIAM, University of Angers, France). Modified and unmodified carbon pastes were immobilized on a SEM sample holder using adhesive carbon tape. Images obtained were from secondary electrons of 3 keV, with magnifications between $\times 25$ and $\times 20,000$. For energy dispersive X-ray spectroscopy (EDX) experiments conducted on the same equipment, the beam energy was 20 keV. Electrochemical impedance spectroscopy (EIS) measurements were carried out performed on a PalmSens BV (Utrecht, Netherlands) running with the PSTrace software. Experiments were done at the frequency range of 0.10 Hz to 104 kHz with the voltage amplitude of 0.18 V.

2.3. Preparation of CPEs

The unmodified CPE was prepared by hand mixing 30 mg of silicone oil with 70 mg of graphite powder (analytical grade, ultra F, <325 mesh, from Alfa) in a mortar. A portion of the composite mixture was packed into the cylindrical hole of a Teflon tube equipped with a copper wire serving as electrical contact with the rest of the circuit. The surface exposed to the solution was polished on a weighing paper to give a smooth aspect before use. Nanocellulose modified carbon paste electrodes (NC-CPEs) were prepared as described for the bare CPE, by using 67 mg of graphite powder, 30 mg of silicone oil, and 3 mg of nanocellulose powder. The surface obtained was similarly polished. When not used, CPEs were removed from supporting electrolyte and stored at room temperature.

2.4. Soil samples study

Soil samples used were collected from Bobo Dioulasso, (West part of Burkina Faso) on a cultivated soil between 0 to 20 cm layer depths. 10 g of soil was weighed and introduced into 100 mL flask containing 10 mL of methanol and was enriched with a known amount of diuron. Flasks were then hermetically closed, placed on an agitator, and agitated for 60 min. The mixture was left to rest for a few minutes and the aliquot was obtained by centrifugation at $4000 \times g$ for 15 min at 25°C, then evaporated with a rotary vapor at 40°C. After evaporation, dried suspension was regenerated with 400 μ L of methanol, transferred into 10 mL of PBS (pH 7.0) and the concentration of residual diuron was determined using SWV. A control that consisted of a flask containing the starting soil solution without diuron was also stirred under the same conditions.

3. RESULTS AND DISCUSSION

3.1. EDX and SEM characterization of various electrodes

EDX spectra of the bare CPE and NC-CPE are presented respectively in Fig. 2A and B. In general, one can see the presence of elements such as carbon and silicon, attributed respectively to carbon powder and silicone oil used for the preparation of carbon paste. The unmodified electrode (Fig. 2A) shows a little amount of oxygen attributed to silicone oil which increased significantly when the electrode is modified by nanocellulose (Fig. 2B). The increase in oxygen observed with NC-CPE shows the presence of nanocellulose in the carbon paste due to the composition of cellulose, which is known to be rich in hydroxyl groups. However, the reduction of silicon in the modified electrode (Fig. 2B) can be due to the probable absorption of silicone oil by cellulose before the sample analysis.. In addition, one can note on Fig. 2C and D representing the SEM 2D images of the unmodified and NC modified CPE, a significant difference in the surface structure of these electrodes: the unmodified CPE was compact and homogenous, whereas NC-CPE surface was predominated by irregular shape, which shows the presence of nanocellulose that induced a certain microporosity in the carbon paste made of carbon powder and silicone oil. As expected, the created microporosity could further enable an easy spreading of diuron in the bulk of NC-CPE.

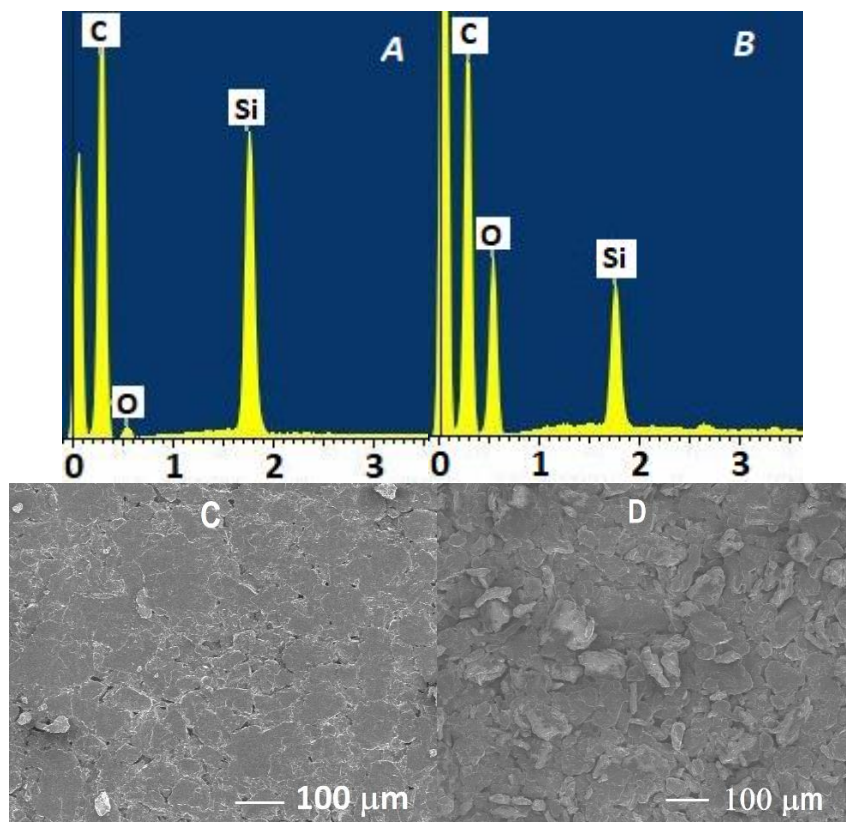


Figure 2.EDX spectra of (A) bare CPE and (B) NC-CPE. 2D-SEM images of (C) bare CPE and (D) NC-CPE.

3.2. Electrochemical Impedance Spectroscopy (EIS)

In order to confirm the modification of CPEs, EIS is used to evaluate the charge transfer rate for both unmodified and NC modified CPE. Fig. 3 shows the Nyquist diagrams of 5 mM $[\text{Fe}(\text{CN})_6]^{3-/4-}$ recorded in 0.1 M PBS (pH 7.0).

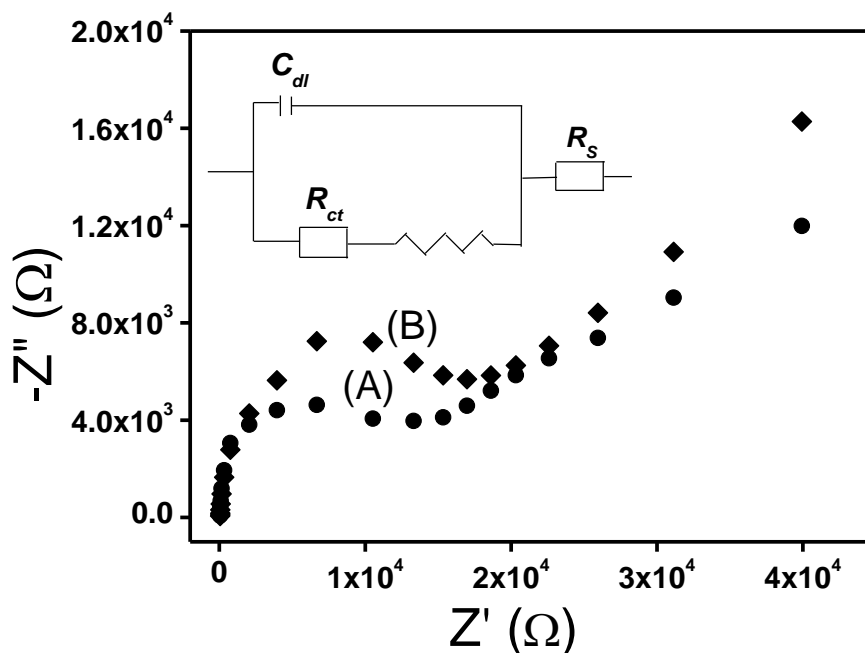


Figure 3. Nyquist plots of (A) CPE and (B) NC-CPE obtained in 0.1 M PBS (pH 7.0) containing 5 mM $[\text{Fe}(\text{CN})_6]^{3-/4-}$ (1 : 1). The frequency range was from 0.1 to 104 Hz at the formal potential of 0.18 V

It appears clearly a difference between the bare CPE (Fig. 3B) and NC-CPE. The interface electron transfer resistance decreases a nearly 2-fold in ($R_{ct} = 18$ and 10 k Ω , respectively) and could be attributed to the presence of nanocellulose in the bulk of CPE that reduced the charge-transfer resistance at the electrode/solution interface, leading thereby to an enhancement of the electron transfer rate for the $[\text{Fe}(\text{CN})_6]^{3-/4-}$ redox system. In agreement with the previous works [17], this result confirms also the modification of CPE surface by NC.

3.3. Electrochemical behaviour of diuron in different electrodes

The electrochemical behaviour of 15 mg/L diuron in different elaborated electrodes was studied by cyclic voltammetry in 0.1 M PBS at pH 7.0 and illustrating on Fig. 4. As it is appears clearly on the figure, diuron undergoes following one irreversible electron transfer in anodic domain as previously reported in the literature [16, 18].

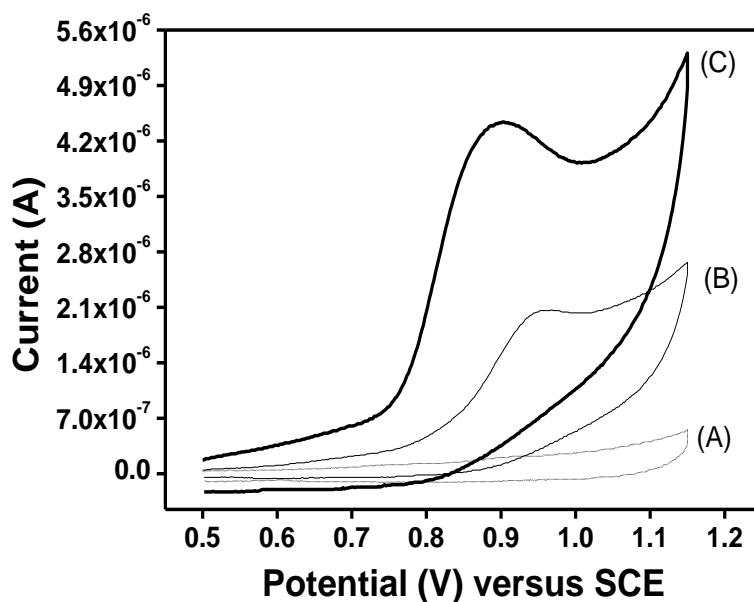
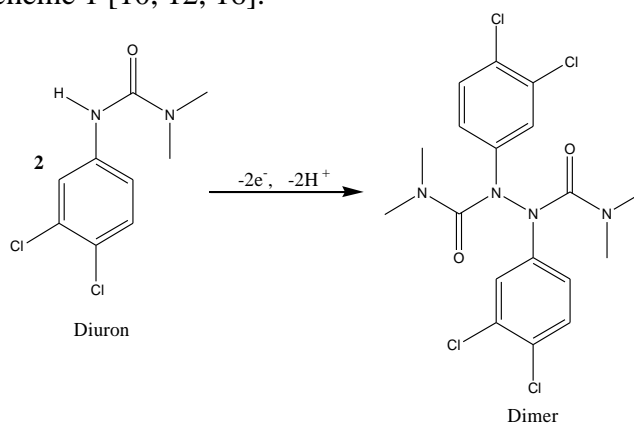


Figure 4. Cyclic voltammograms recorded in 0.1 M PBS containing 15 mg/L of diuron on (B) bare CPE, and (C) NC-CPE. Curve (A) corresponds to the CV behaviour of NC-CPE in blank solution. Potential scan rate: 100 mV/s.

The peaks observed at 0.85 V and at 0.95 V versus SCE respectively with NC-CPE and the bare CPE are attributed to an oxidation of diuron, following by chemical reaction leading to the formation of a dimer as illustrated in Scheme 1 [10, 12, 16].



Scheme 1. Electrochemical oxidation of diuron

In addition, one can observe a cathodic shift of potential and an increasing of the peak intensity. Indeed, the oxidation peaks observed at 0.95 V when CPE working electrode is used, decreases to 0.85 V with NC-CPE. This result indicates an electrocatalytic ability of cellulose that was clearly demonstrated previously by EIS. This increase of the peak current is due to the increase of the real electrode area provoked by NC as recently reported by Mbokou *et al.* [19].

3.4. Effect of nanocellulose amount in the carbon paste

In order to obtain a better signal on the determination of diuron with NC-CPE, the amount of nanocellulose in the carbon paste, some instrumental parameters in SWV have been studied as reported by Pontié et al. [20].

Fig. 5 shows the evolution of peak current according to nanocellulose mass. It can be seen that, when the NC amount in the carbon paste increases from 2 to 3 mg, the anodic peak current of diuron increases significantly whereas the peak current declines drastic for NC amount more than 5 mg. Indeed, a higher amount of NC leads to an increase of the thickness in the carbon paste, which limits considerably electrons transfer.

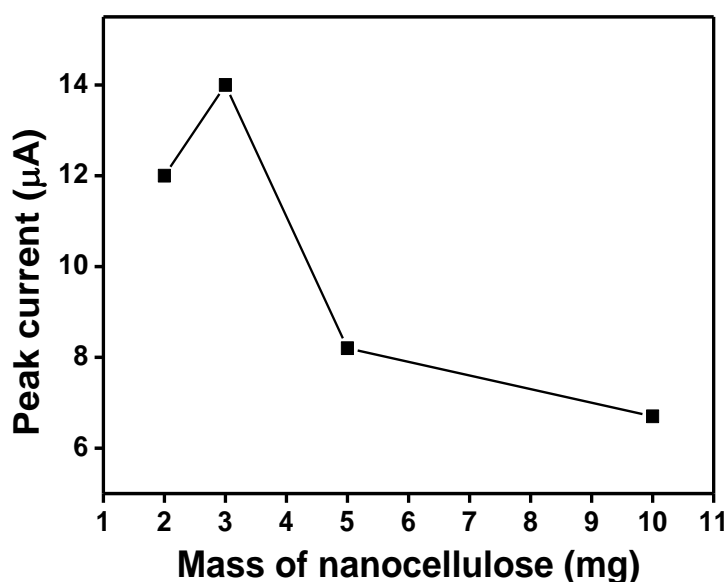


Figure 5. Effect of the nanocellulose amount (mg) in NC-CPE on the SWV peak current of 10 mg/L diuron. Experimental conditions: detection in 0.1 M PBS (pH 7.0), SWV parameters were frequency 50 Hz, pulse height 60 mV, and scan increment 20 mV.

The effect of SWV parameters are been studied (data not shown) and the following optimum parameters were: frequency of 50 Hz, pulse height of 60 mV and scan increment of 20 mV. Fig. 6 illustrates the SWV voltammogram recorded using optimized parameters on unmodified and NC modified CPEs for 10 mg/L of diuron. .

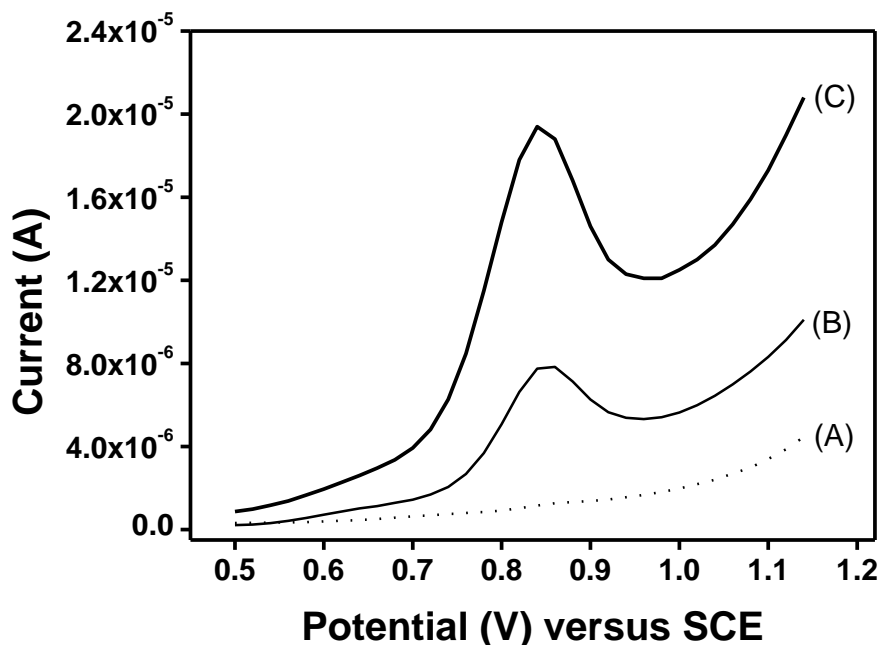


Figure 6. SWVs of 10 mg/L diuron on (B) bare CPE and (C) NC-CPE in PBS. Curve (A) corresponds to the SWV behavior of NC-CPE in blank solution.

One can note through this figure that the sensitivity of NC-CPE is 2.3-fold than that of bare CPE and confirmed that the presence of nanocellulose in the carbon paste increases simultaneously the real surface area and the catalytic effect of the electrode [21].

3.5. Effect of pH

pH effect was investigated at pH ranging from 6.3 to 7.4 in PBS, for 15 mg/L diuron. Indeed, we note that the peak potentials shifts toward cathodic values with increase in pH as seen in Fig. 7. This is probably due to the deprotonation of diuron leading rise to an anion more rich in electrons and which is easily oxidable [11].

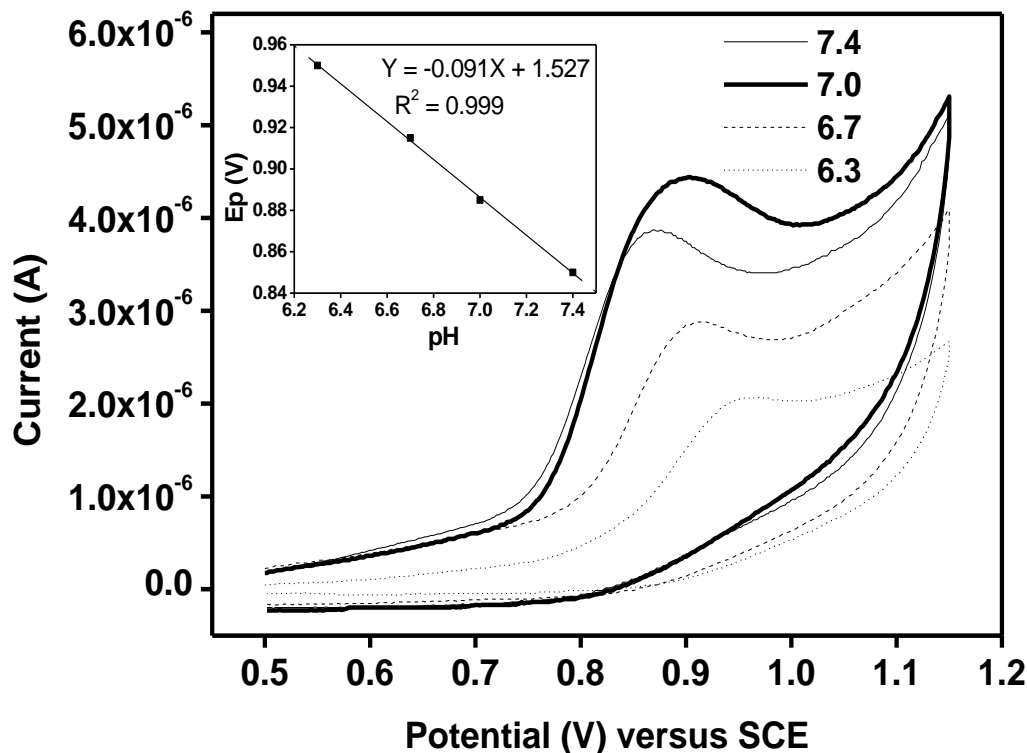


Figure 7. Cyclic voltammograms at different pH values in 15 mg/L diuron in 0.1 M PBS on NC-CPE. Inset shows peak potential against pH values.

Inset of Fig. 7 presents plots E_p (V) versus pH. The results showed a linear relation between peak potential and pH with regression equation of E_p (V) = $-0.091\text{pH} + 1.527$ ($R^2 = 0.999$). The slope obtained (0.091) of the linear regression equation is not really close to the ideal 0.059 V. Nevertheless, the highest oxidation currents were recorded at pH 7.0 (Fig. 7); hence, this pH was used for all studies.

3.6. Effect of scan rate

The cyclic voltammograms of 15 mg/L diuron on NC-CPE at different scan rates were investigated in pH 7.0 PBS, as shown in Fig. 8A. The oxidation peak current (I_p , μA) increased linearly with the square root of scan rate (ν) from 0.1 to 10 V/s with a linear regression equation of I_{pa} (μA) = $1.358 \nu^{1/2}$ (V/s) $^{1/2} - 0.252$ ($R^2 = 0.997$) (Fig. 8B) showing that a diffusion controlled process occurs on the modified electrode surface [13].

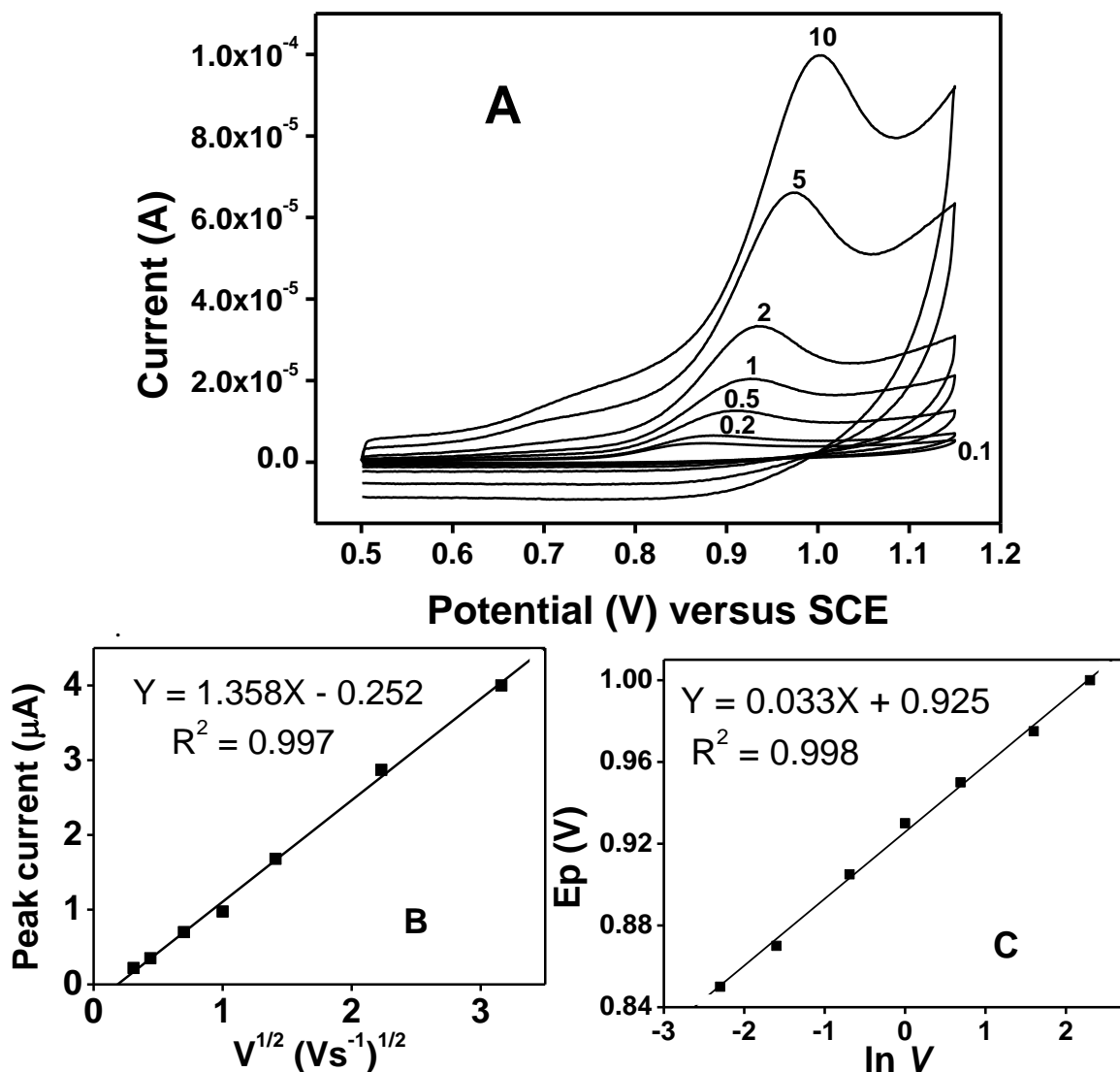


Figure 8.(A) Cyclic voltammograms of 15 mg/L diuron in PBS at NC-CPE with different scan rates: 0.1, 0.2, 0.5, 1, 2, 5 and 10 V/s; (B) linear dependence of I_p versus $v^{1/2}$ and (C) plot of peak potential versus logarithm of scan rate.

The positive shift in potential (E_p , V) of the oxidation peak was increasing scan rate (Fig. 8C) with the equation of $E_p = 0.033 \ln(v) + 0.925$ ($R^2 = 0.998$), illustrating the irreversible nature of the electrochemical oxidation of diuron. The slope of the representation close to 33 mV per decade confirms the chemical reaction following the electron transfer. In order to determine the Tafel slope, the usual equation (Equation (1)) for a totally irreversible process [22] was used:

$$E_p = \frac{b}{2} \log v + \text{constant} \quad (1)$$

where v is the scan rate and b the Tafel slope. The plot of E_p versus $\log v$ (Fig. 8c) gave a linear relationship and the Tafel slope ($b = 0.059/\alpha n$) was found to be 66 mV for diuron and was in the normal range 30-120 mV [23-25]. Using the slope of the plot of E_p versus $\log v$ (Fig. 8c), the α value for diuron was calculated to be 0.45 since the n value of transferred electrons during the oxidation process is supposed to be 2 [14].

3.7. Calibration curve

The calibration curve for determination of diuron concentration on NC-CPE using SWV and under optimized experimental conditions was obtained. Fig. 9 shows the linear range from 1 to 10 mg/L.

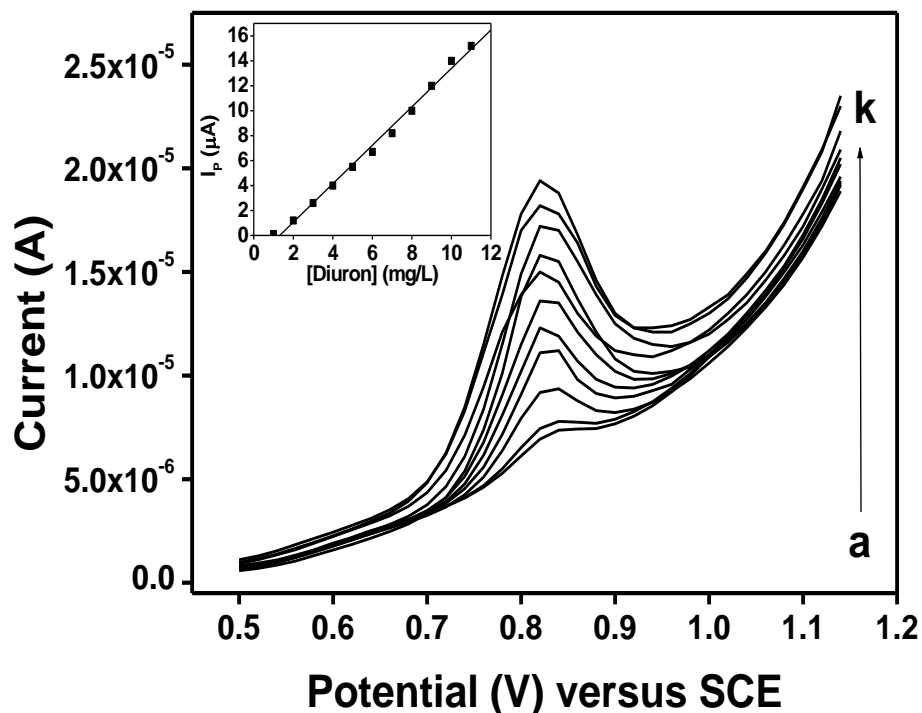


Figure 9. SWV of diuron at different concentrations in PBS on the NC-CPE. Diuron concentration (a-k): 1, 2, 3, 4, 5, 6, 7, 8, 9, 10 and 11 mg/L. Insert shows peak current versus diuron concentration.

The electrode response shows a linear dependence between the peak current (I_p) and diuron concentration (inset in Fig. 9), expressed by the equation I_p (A) = 1.54 [diuron] (mg/L) – 2.02 with a correlation coefficient (R^2) of 0.994. The limit of detection (LOD) calculated with a signal-to-noise ratio of 3 was found to be 0.35 μ M. Table 1 gives the performance of NC-CPE for the voltammetric determination of diuron in comparison to other modified CPEs.

Table 1. Comparison of detection limits (LODs) of different modified CPEs reported for the detection of diuron

Modified electrodes	pH	Linear range (μ M)	LOD (μ M)	Reference
CPE/MWCNT-COOH ^a	8.0	0.05- 1.25	0.009	[12]
CPE/Ni(II)	7.5	50 - 1000	20	[15]
CPE/NiOPc/hemine/GO ^b	8.0	9.9 - 150	6.14	[16]
NC-CPE	7.0	4.2 - 47	0.35	This work

^acarboxyl functionalized multi-walled carbon nanotubes (MWCNT-COOH)

^bnickel(II) 1,4,8,11,15,18,22,25-octabutoxy-29H,31H-phthalocyanine complex

From Table 1, the detection limit obtained in this study ($0.35 \mu\text{M}$) is better than those achieved with CPEs incorporating nickel [15] and phthalocyanine complex [16].

3.8. Interference study

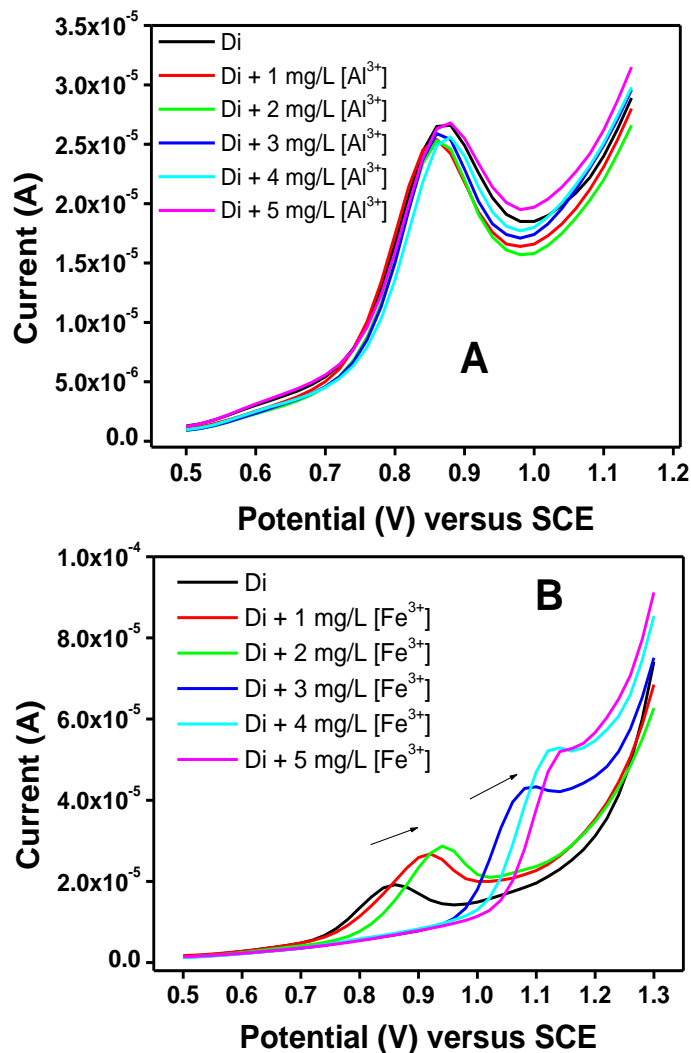


Figure 10. SWV curves recorded for 10 mg/L diuron in 0.1 PBS (pH 7.0) in the presence of A: Al³⁺ and B: Fe³⁺ ions.

The influence of two inorganic ions commonly found was also investigated under the optimal experimental conditions. Fig. 10 shows the influences of both Al³⁺ and Fe³⁺ on the determination of 10 mg/L diuron in SWV. No significant interference was found after addition of 1 to 5 mg/L Al³⁺ ions. In contrast, Fe³⁺ was found to interfere significantly on the signal of diuron by shifting the oxidation peak potential to anodic values (Fig. 10B). Moreover, it is noticed that the pH of solution decreased gradually when adding Fe³⁺ ions, reaching a value of 1.42 after addition of 5 mg/L Fe (III) ion. This result is due to a chemical reaction, which occurs between diuron and Fe (III) ion leading to the release of protons in the media. The possible interaction between diuron and Fe (III) ion may form a stable complex involving

one Fe (III) ion and two molecules of diuron, leading to a tetra dental complex [26]. This is very original and can be a way for indicating the presence of diuron in any matrices.

3.9. Application of the NC-CPE sensor in soil samples

The application of the proposed approach leads us to use the elaborated NC-CPE for the detection of diuron in enriched soil samples using standard addition method [12, 18]. Fig. 11 illustrates the linear relationship obtained between the peak intensity (observed at 0.885 V versus SCE) and concentration of added diuron. It is expressed as $I_p (\mu A) = 1.325 [\text{diuron}] (\text{mg/L}) + 0.950$ ($R^2 = 0.995$).

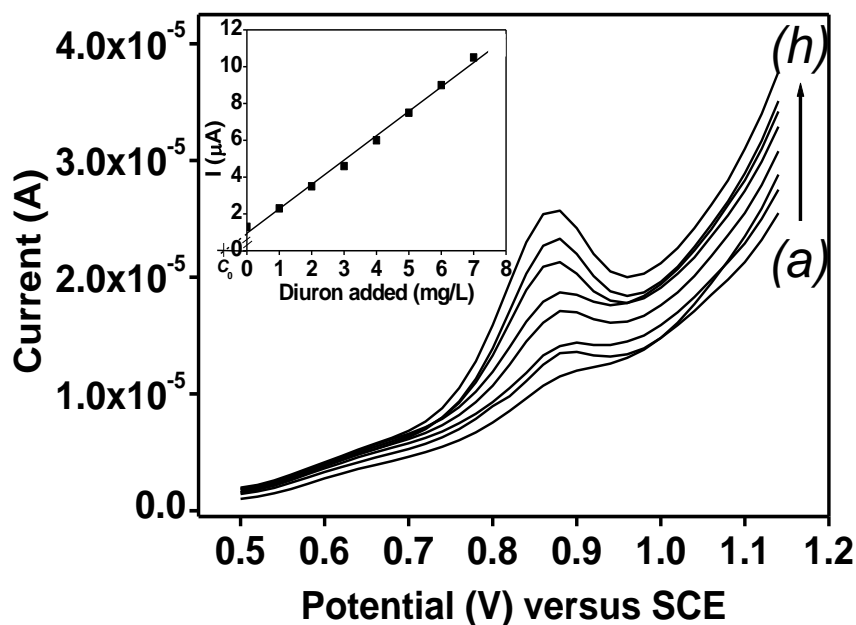


Figure 11. Application of the standard addition method for the quantification of diuron in soil samples by SWV in 0.1 M PBS (pH 7.0). Analytical conditions: [diuron] = (a) unknown, Diuron concentrations added (b-h): 1, 2, 3, 4, 5, 6 and 7 mg/L.

Diuron recovery amount in soil samples was found to be 96 % with the proposed technique (Table 2) and was in acceptable range (70 to 120 %) [27,28], proving that the proposed sensor and the standard addition method can be suitable for the determination of diuron in real matrix.

Table 2. Determination of diuron in soil samples using NC-CPE (recovery rate in % and others values in mg).

	Theoretical mass of diuron supposed to be in soil samples	3.0
Soil samples	Real theoretical mass of diuron in soil samples	2.4
	Diuron mass determined with NC-CPE	2.3
	Recovery	96

4. CONCLUSIONS

The results showed that the presence of nanocellulose incorporated into the carbon paste was essential for the increase in sensitivity observed in the electrochemical determination of diuron. The sensitivity of NC-CPE was found to be better than some existing electrochemical sensors for determination of diuron. The elaborated sensor was successfully applied to determine diuron in enriched soil samples and was demonstrated to be a suitable tool for analyses involving environmental matrices.

CONFLICTS OF INTEREST

The authors declare that they have no conflict of interest.

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