

Detection of Lead (II) on a Boron-doped Diamond Electrode by Differential Pulse Anodic Stripping Voltammetry

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Authors' contributions

This work was carried out in collaboration among all authors. All authors read and approved the final manuscript.

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ABSTRACT

Lead, even in low concentrations, can be dangerous and toxic to humans and their environment. Due to the toxicity of this metal, an electroanalytical method has been developed for the direct quantitative determination of Pb²⁺. The Pb²⁺ detection was performed using Differential Pulse Anodic Stripping Voltammetry. The quantification of Pb²⁺ by these electrochemical methods was carried out on a boron-doped diamond micro electrode in HNO₃ medium (0.01 M). This work made it possible to efficiently detect lead with a detection limit equal to 0.052 μM and a quantification limit equal to 0.173 μM. This method made it possible to selectively detect and quantify the Pb²⁺ in the presence of other metals such as Cd²⁺ and Cu²⁺. In the presence of other metals, a recovery rate of 94.53% was observed. This value is close to the recovery rate obtained (98.6%) when the Pb²⁺ is alone in electrolyte.

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1. INTRODUCTION

Concentrations of heavy metals in the environment are increasingly high near road traffic areas, urban and industrial sites or mining activities. Heavy metal pollution is the source of the most serious environmental problems [1]. Even at low concentrations, they can constitute a serious public health problem due to their toxicity and their bioaccumulative character [2]. Certain heavy metals such as Cu or Zn are an integral part of biogeochemical cycles (trace elements) and only become toxic at high levels, while others, such as Cd or Pb, are not necessary and are toxic for the trophic chain whatever their contents [3]. Lead has been widely used for centuries around the world for its ease of extraction, malleability, low melting point. It is currently the 5th most commonly used metal in the world. However, lead has detrimental effects on the central nervous system of the developing infant [4,5]. Although neurotoxicity has the most sensitive endpoint, lead can also cause blood pressure, affect kidney function, cause mutagenesis and have reproductive effects [6]. Due to its toxicity, lead must be quantified in aqueous media with a view to controlling its use in several areas of human activity. However, lead detection methods remain expensive for poor countries. This is why a simple, fast and less expensive method of detecting lead must be implemented.

In the literature, the quantification of metallic trace elements (example: lead); researchers use several very expensive analytical methods such as Atomic Absorption Spectrometry [7,8] and Inductive Plasma Mass Spectrometry. In recent years, electrochemical detection has become a promising technique [9,10] because the cost of manufacturing electrochemical sensors is low, it is more sensitive and inexpensive. Several electrochemical methods have made it possible to quantify Pb^{2+} , we have adsorption voltammetry [11], square wave anodic stripping voltammetry [12], differential pulse anodic stripping voltammetry (DPASV) [13]. These methods owe their sensitivity to the use of electrodes based on mercury [14], glassy carbon [15], iridium [16], platinum electrode [17] and silver carbon electrode [18].

A boron-doped diamond electrode has been widely studied due to its interesting electrochemical properties, including: high thermal conductivity, high hardness and chemical inertia, a

wide window of electrochemical potential in aqueous and non-aqueous media, very low capacity and very high electrochemical stability [19-23]. This material is very resistant to corrosion, which has allowed its use in more extreme environments, such as in extremely corrosive environments [24]. Its robustness is strongly recommended, and very well suited to voltammetric analysis.

In this article, we will determine the conditions for the use of an anodically pre-treated boron-doped diamond (μ BDD) micro electrode (1.4 V / ESM) for the selective determination and quantification of Pb^{2+} by DPASV.

2. MATERIALS AND METHODS

2.1 Experimental Methods

Cyclic voltammetry (CV) and Differential Pulse Anodic Stripping Voltammetry (DPASV) were used for the electrochemical measurements. These measurements were carried out using an ECHOCHEMIE Autolab Potentiostat (PGSTAT 20) controlled by a computer (GPES software). The cell used to perform the electrochemical measurements is a cell with three electrodes: the working electrode, the counter electrode and the reference electrode. A boron-doped diamond (μ BDD) micro electrode was used as the working electrode. The geometric contact area between the working electrode and the electrolyte is 14.28 mm². A mercurous sulfate electrode (ESM) was used as a reference electrode. To decrease the contribution of ohmic losses, the reference electrode was mounted in a luggin capillary and placed close to the working electrode by a distance of 1 mm. The counter electrode (CE) was a platinum wire. The pH of the electrolytes was measured using a HI2211 pH meter probe with a glass electrode. All measurements were performed at 26 ° C.

2.2 Chemicals

Lead nitrate ($(Pb(NO_3)_2$, 99.5%) was obtained from Merck, Darmstadt and solutions of perchloric acid ($HClO_4$, 60%) and nitric acid (HNO_3 , 69%) were obtained from Panreac, Barcelona (Spain). Cadmium nitrate tetrahydrate ($CdN_2O_6.4H_2O$, 98%) and anhydrous copper sulfate ($CuSO_4$,) were obtained from Sigma-Aldrich (India).

The chemicals used are analytical types and stored at room temperature and protected from light, all solutions were prepared from distilled water. The pH was adjusted by adding appropriate amounts of sodium hydroxide solution (2M) and nitric acid (2M).

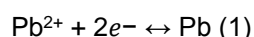
3. RESULTS AND DISCUSSION

3.1 Electrolytic Medium

In order to study the influence of Pb^{2+} on the electrochemical behavior of the μ BDD electrode, voltammetric measurements were carried out in the absence and in the presence of lead (II) with the μ BDD electrode. The results obtained are presented in Fig. 1.

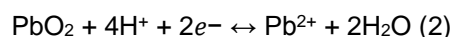
In the presence of 0.156 mM Pb^{2+} , two oxidation peaks in the forward direction and two reductions peaks in the backward potential scan are observed. The first oxidation peak observed at -0.848 V / ESM is associated with the reduction peak observed at -1.014 V / ESM. The anode peak (-0.848 V / ESM) attributed to the reoxidation of the lead metal previously deposited in Pb^{2+} then the cathodic peak (-1.014 V / ESM) is due to the reduction of Pb^{2+} in metallic lead [25].

The reaction involved here is:



The second oxidation peak is observed at 1.124 V / ESM which is also associated with a

reduction peak located at 0.646 V / ESM. The presence of this second anodic peak would be due relatively to the oxidation of Pb^{2+} to Pb^{4+} in the form of PbO_2 and the associated reduction peak would be the reduction peak of Pb^{4+} previously formed in Pb^{2+} . The redox couple involved here is Pb^{2+} / Pb^{4+} [26] represented by the following equation:



The choice of the electrolytic medium is one of the key parameters which govern the efficiency of the detection of a metal (Pb, Cd, Cu ...) in an electrochemical process. Thus the influence of the Pb^{2+} concentration on the cyclic voltammogram was studied in 0.01M HNO_3 and 0.01M $HClO_4$. Figs. 2A and 2B show the results obtained. These curves show that the intensities of the lead reoxidation peaks increase with the Pb^{2+} concentration in the two media studied. These growths are due to the increasing accumulation of Pb^{2+} on the surface of μ BDD. We also note that the intensities of the second oxidation peaks increase with the concentration of Pb^{2+} . On these curves, we note the presence of two oxidation peaks and two reduction peaks regardless of the concentration of Pb^{2+} . It is noted that the intensities of these peaks vary with the concentration of Pb^{2+} . Fig. 3 shows the intensity of the first oxidation peak as a function of the Pb^{2+} concentration. This figure shows the evolution of the peak reoxidation currents of metallic lead as a function of the Pb^{2+} concentration.

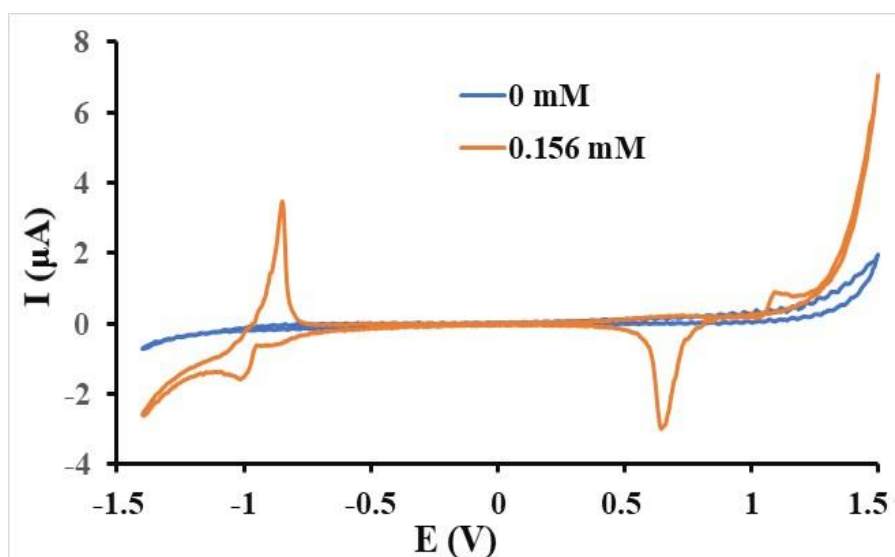


Fig. 1. Cyclic voltammogram in 0.01M HNO_3 containing 0 mM and 0.156 mM of Pb^{2+} ; scan rate = 50 mV / s

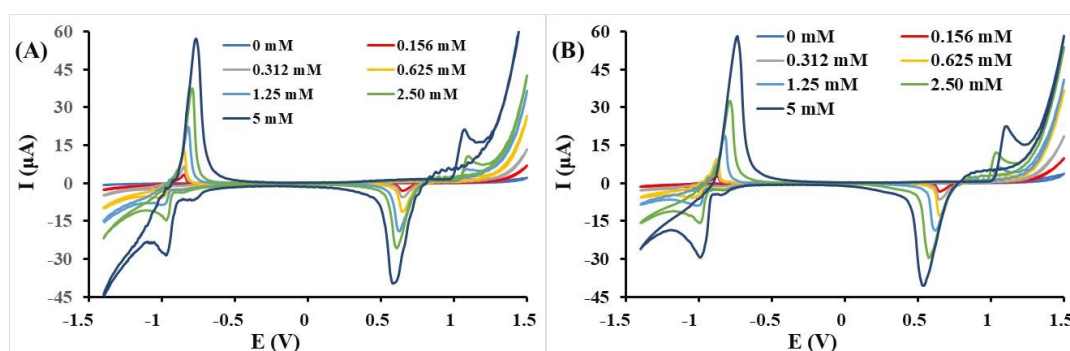


Fig. 2. Cyclic voltammogram in (A) 0.01 M HNO₃ and (B) 0.01 M HClO₄ medium containing Pb²⁺ (0 mM to 5 mM) at 50 mV / s

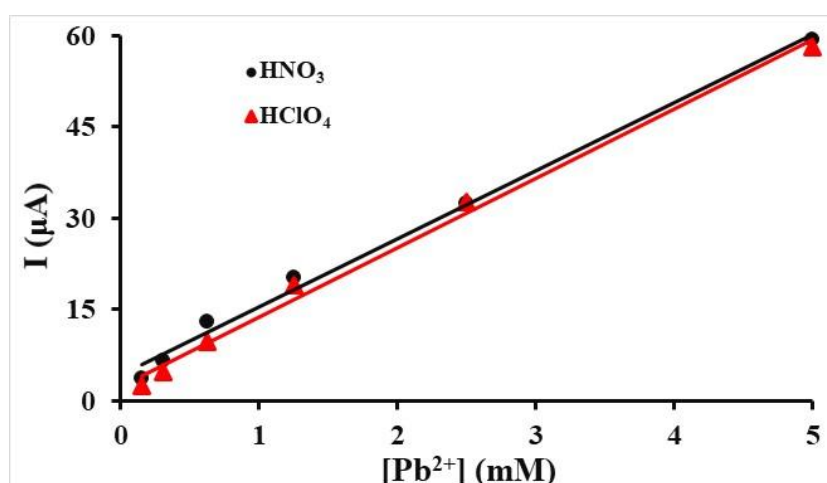


Fig. 3. Evolution of the intensity of the reoxidation peak of metallic lead as a function of Pb²⁺ concentration in 0.01M HNO₃ and 0.01M HClO₄ medium at $v = 50$ mV / s

We note in the two studied media, the curves obtained are straight lines. In 0.01 M HNO₃ medium, the curve obtained is a straight line with a coefficient of determination of 0.993. The proportionality between the current intensity of the oxidation peak and the Pb²⁺ concentration confirms that this peak is related to the oxidation of lead. In 0.01 M HClO₄ medium, the curve obtained is a straight line with $R^2 = 0.994$. This result shows that the increase in the oxidation wave is directly related to the reoxidation of metallic lead.

It is noted that the two curves obtained are almost stackable. This would mean that the amount of lead (II) detected is approximately the same in the two electrolytes. This result was attested by the work of Fernando Barbosa Jr et al who used several supporting electrolytes with a concentration varying from 0.001 M to 0.1 M for the detection of lead [8]. However, we note that the straight line obtained with HNO₃ is slightly

above that obtained in HClO₄ medium. This shows that HNO₃ is the best carrier electrolyte of the two acids. It will be retained for the rest of our work.

3.2 Influence of the pH

The influence of pH on the lead oxidation peaks was investigated. The results obtained are presented in Fig. 4A. This figure shows the voltammetric measurements carried out on the BDD micro electrode, in 0.01 M HNO₃ containing 2.5 mM of Pb²⁺, by varying the pH from 0.43 to 11.23. The pH of the electrolyte was modified by adding NaOH (2M) or HNO₃ (2M). Examination of the voltammograms reveals that the position and intensity of the Pb (II) reoxidation peaks vary with pH.

As shown by the curve in Fig. 4B, the intensity of the Pb²⁺ reoxidation peak is remarkably influenced by the pH values in the range studied.

Indeed, in basic solution (pH = 7.45 and pH = 11.23) a plateau is observed which proves that at this pH there is no cation exchange since the intensities of the reoxidation peak of recorded metallic lead is practically non-existent, then the signal increases with pH from 0.43 to 1.98. From 1.98, a decrease in the peak is observed until a plateau at basic pH is obtained. The disappearance of the reoxidation peak of metallic lead (II) in the basic medium is due to the fact that Pb^{2+} are more dominant and mobile for pH below 6 [27]. Some heavy metals (Cd and Pb) have been shown to form larger inorganic complexes in alkaline media ($Pb_6(OH)_8^{4+}$), which cannot be transported as easily across biological membranes as the divalent metal ion M^{2+} [28].

3.3 Lead Detection by Differential Pulse Anodic Stripping Voltammetry

DPASV is an electrochemical method for the detection of trace or ultra-trace chemical

elements. For better lead detection it is important to optimize the various analysis parameters.

3.3.1 Optimization parameters

3.3.1.1 Best deposit time

The deposition time is an important parameter for the detection of metals (lead) on a substrate (μ BDD). The deposition time effect was achieved for times between 10s and 900s. The deposition took place in the potential range -1.4 V / ESM to -0.4 V / ESM and the chosen deposition potential is -1.4 V / ESM. In Fig. 5, it can be seen that the metal detection peak intensity of (lead) with the deposition time. This increase in peak could be due to the surface of the working electrode. In order to reduce the analysis time and be more energy efficient, we will use as optimized deposition time: $Dt = 300s$.

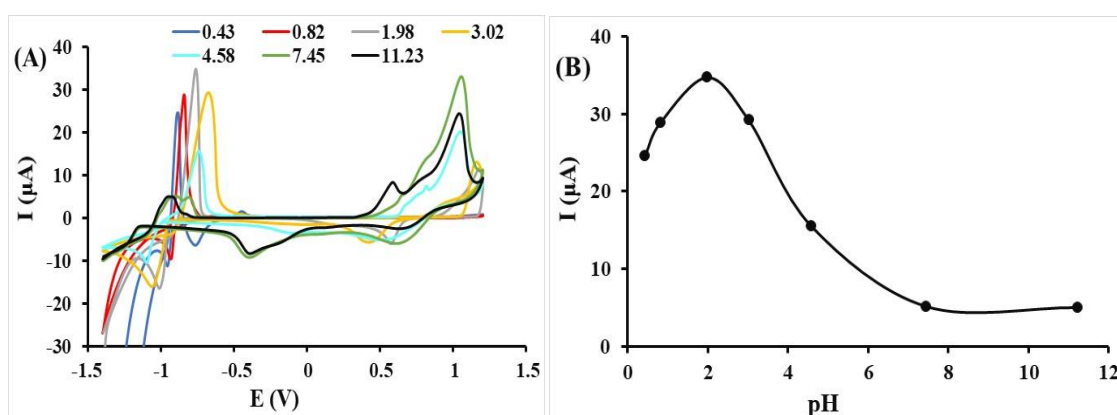


Fig. 4. (A) Cyclic voltammograms on a μ BDD in 0.01M HNO_3 containing Pb^{2+} (2.5 mM) at different pH; (B) I curve as a function of the electrolyte pH; scan rates = 50 mV / s

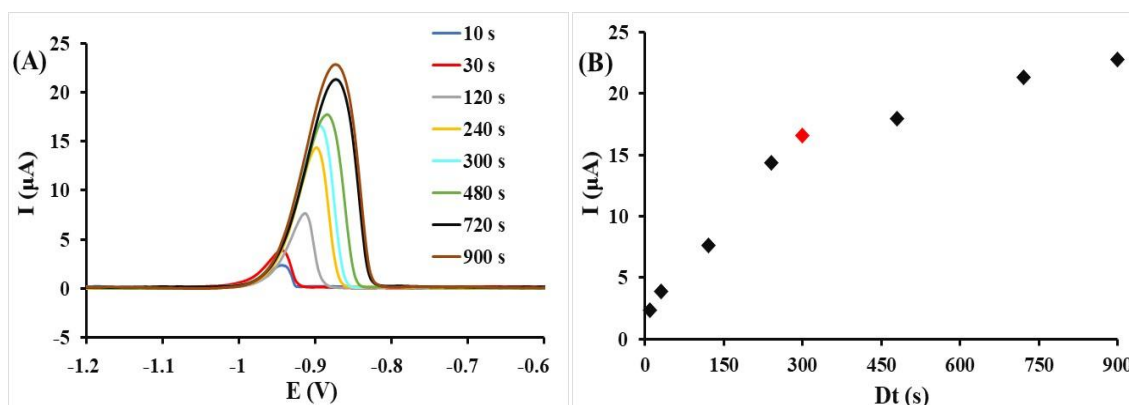


Fig. 5. (A) DPASV of 10 $\mu M Pb^{2+}$ as a function of the lead deposition time in HNO_3 ; (B) Variation of lead oxidation peak currents as a function of deposition time (Dt), pH = 2

3.3.1.2 Best modulation time

Fig. 6A represents the variations of the lead detection peak by the DPASV method as a function of the modulation time. It should be noted that the reduction of Pb^{2+} occurred at the potential $-1.4\text{ V} / \text{ESM}$ and in the range $-1.4\text{ V} / \text{ESM}$ to $-0.4\text{ V} / \text{ESM}$. This deposit occurred at 300s. The various peaks obtained made it possible to plot the curve of the Fig. 6B representing the current density as a function of the modulation time. We observe a decrease in the current peak during the evolution of the modulation time. However, the distorted shape of the voltammograms and the reading potential of these voltammograms we have chosen as modulation time (Mt) of 30 ms.

3.3.1.3 Best potential step

To obtain the optimum value of the potential step, measurements were carried out for a deposition time $Dt = 300\text{ s}$ and a modulation time $Mt = 30\text{ ms}$, then the various voltammograms obtained were recorded in Fig. 7A. These measurements were made in the range of potentials from $-1.4\text{ V} / \text{ESM}$ to $-0.4\text{ V} / \text{ESM}$. Fig. 7B is the curve showing the evolution of the reoxidation peaks of the Pb as a function of the potential step.

In this figure we observe an increase in the current peak up to the potential of 4 mV, from this potential value, the current peak is almost constant. This constancy could be due to the saturation of the surface of the μBDD electrode. For this reason, the value 4 mV will be chosen as the optimal value of the potential step for our method.

3.3.1.4 Better modulation amplitude

The effect of the modulation amplitude of the Pb^{2+} response on μBDD was studied in the potential range of $-1.4\text{ V} / \text{ESM}$ to $-0.4\text{ V} / \text{ESM}$.

The optimum amplitude was obtained using the following values: $Dt = 300\text{ s}$, $Mt = 30\text{ ms}$ and $Ps = 4\text{ mV}$. For modulation amplitude values between 5 mV and 200 mV, several voltammograms were recorded and shown in Fig. 8A. Fig. 8B indicates the variation of the lead oxidation peak currents as a function of the modulation amplitude then it is observed that the variation of the current peak increases exponentially with the amplitude, this increase can be at the amplitude applied for the detection of lead. We observe that the higher the amplitude, the more the peak moves to the left and one moves away from the detection potential of the lead peak.

In order to reduce the analysis time in the rest of our work, the value of the modulation amplitude (Ma) equal to 30 mV was taken as the optimum value for detecting lead on μDDB .

3.3.1.5 Better deposit potential

One of the valuable parameters for the detection of lead by our method is the effect of the deposition potential. This parameter has an influence on the selectivity of the oxidation peak of Pb in nitric acid (0.01M). Fig. 9A shows the different voltammograms recorded while varying the deposit potential from $-1.7\text{ V} / \text{ESM}$ to $-1.2\text{ V} / \text{ESM}$ by setting the following optimal values: $Dt = 300\text{ s}$, $Mt = 30\text{ ms}$, $Ps = 4\text{ mV}$ and $Ma = 30\text{ mV}$. These recordings were made in the potential range -1.4 V to -0.4 V . In Fig. 9B, we observe that the lead deposition potential increases with its oxidation peak. This increase in the oxidation peak is due to the fact that the oxidation potential of the Pb^{2+} has not yet been reached. For potentials greater than -1.4 V , the oxidation peak of the metal decreases with the increase in the deposition potential. In the remainder of our study, $Dp = -1.4\text{ V}$ was taken as the optimum value of the detection potential of Pb. Because at this potential the current generated is the maximum.

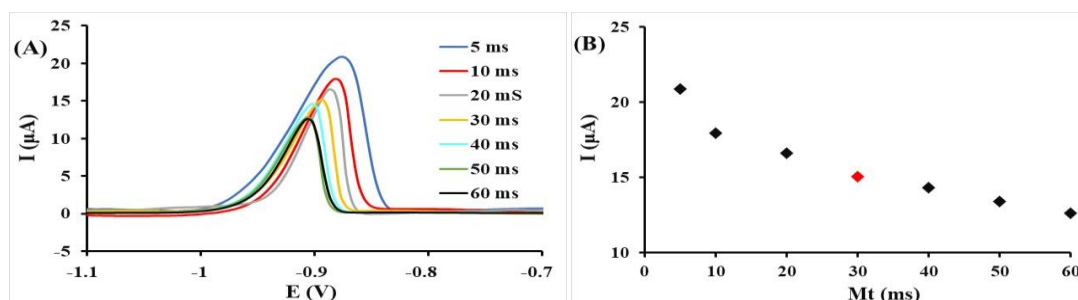


Fig. 6. (A) DPASV of $10\text{ }\mu\text{M}$ Pb^{2+} as a function of lead modulation time in HNO_3 ; (B) Lead oxidation peak currents as a function of modulation time (Mt), $Dt = 300\text{ s}$, $\text{pH} = 2$

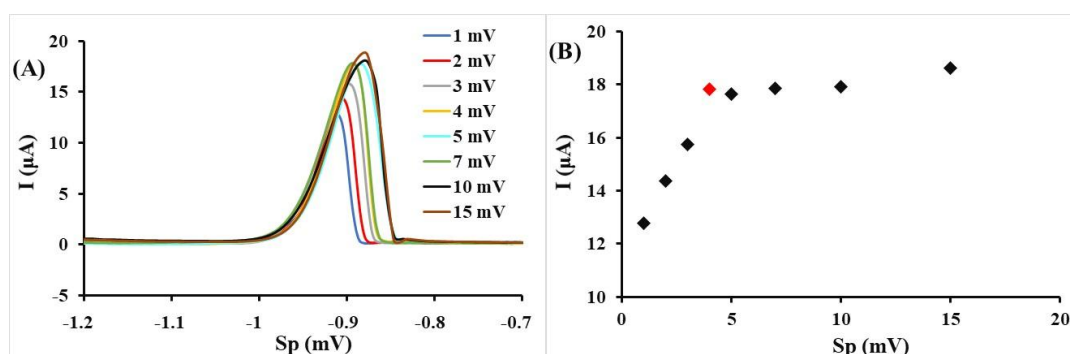


Fig. 7. (A) DPASV of 10 μM Pb²⁺ as a function of the potential step of lead in 0.01M HNO₃; (B) Variation of lead oxidation peak currents as a function of the potential step (Ps), Dt = 300s, Mt = 30 ms, pH = 2

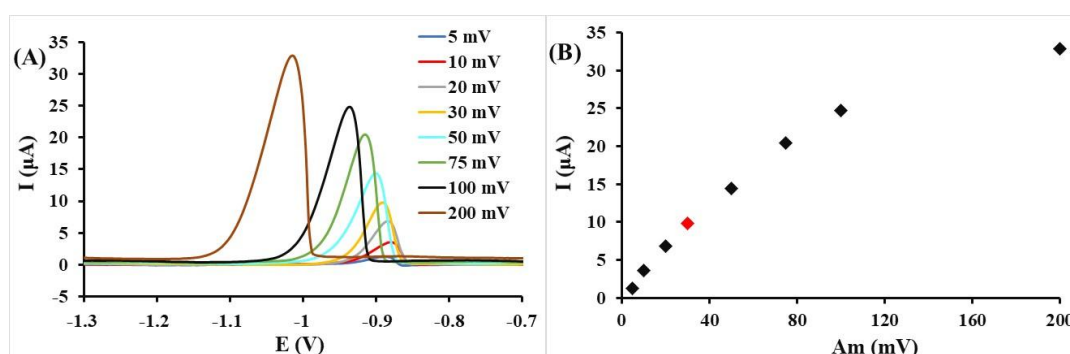


Fig. 8. (A) DPASV of 10 μM Pb²⁺ as a function of the detection modulation amplitude of lead in 0.01M HNO₃; (B) Variation of lead oxidation peak currents as a function of the modulation amplitude (Ma), Dt = 300s, Mt = 30 ms, Ps = 4 mV, pH = 2

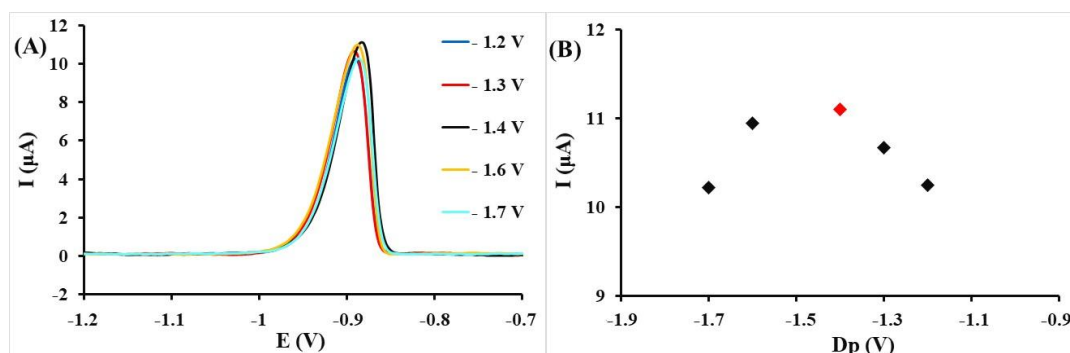


Fig. 9. (A) DPASV of 10 μM Pb²⁺ as a function of the deposition potential for the detection of lead in 0.01M HNO₃; (B) Variation of lead oxidation peak currents as a function of the deposition potential (Dp), Dt = 300s, Mt = 30 ms, Ps = 4 mV, Ma = 30 mV, pH = 2

3.3.1.6 Best cleaning time

The regeneration of the surface of μBDD is just as important as the previous parameters. The repeatability of the different voltammograms was used to achieve the best cleaning time for the working electrode. The chosen deposit time is five (5) minutes; the cleaning times applied are

between 100s and 600s. For each cleaning time a deposit was first applied to the optimum values chosen previously. Then, we carried out a pretreatment of the μBDD by imposing the reverse potential of Pb deposition (1.4 V / ESM) during the chosen pretreatment time (100s to 600s). Then we measured the cyclic voltammetry in the blank (0.01 M nitric acid) for the potential

range [-1.4 V / ESM; -0.4 V / ESM]. This step is important because it certifies that the cleaning has been done well. Finally, we applied another deposit in order to verify the repeatability thanks to the different voltammograms obtained.

Fig. 10A defines the recordings obtained thanks to the DPASV applied to a solution of lead nitrate (10 μM) contained in HNO₃ (0.01M) for cleaning time values between 100s and 600s. In order to see the difference between the current peaks obtained; Fig. 10B has been carried out.

In Fig. 10B, the different recovery rates for each cleaning time, repeated twice are entered therein. A cleaning time of 400s was chosen, allowing total cleaning of the μBDD surface, even after reoxidation of the Pb carried out with a concentration of 10 μM and ensuring a good coverage rate, ie 98.76%.

3.3.2 Determination of method validation parameters

3.3.2.1 Current intensity calibration curve as a function of Pb²⁺ concentration

Fig. 11A shows the different voltammograms linked to the concentrations of Pb²⁺ ranging from 0.799 μM to 6.392 μM. Analysis of this figure

indicates that the intensity of the lead oxidation peak current varies with the Pb²⁺ concentration. These quantitative analytical measurements were carried out taking into account the optimal values grouped in Table 1.

To obtain a method of quantitative analysis, one must be able to relate the intensity of the current peak to Pb²⁺ concentration. This part is devoted to the plotting of the calibration curve (Fig. 11B) in a Pb²⁺ concentration range of between 0.799 μM to 6.392 μM. The curve obtained is a straight line of equation $I_{pic} (A) = 1.836 C (M) \cdot 10^{-6}$ with a coefficient of determination $R^2 = 0.996$ and which is very close to 1. This reflects good linearity of the method for the chosen concentration range.

The detection limit (LOD) and quantification limit (LOQ) for the proposed method were determined from equations 3 and 4 [21,29,30].

$$LOD = 3 \cdot \sigma / S \quad (3)$$

$$LOQ = 10 \cdot \sigma / S \quad (4)$$

Here, σ is standard deviation of blank current signals and S is a slope of the calibration curve. For this study, the limits of detection and quantification are respectively $LOD = 0.052 \mu M$ and $LOQ = 0.173 \mu M$.

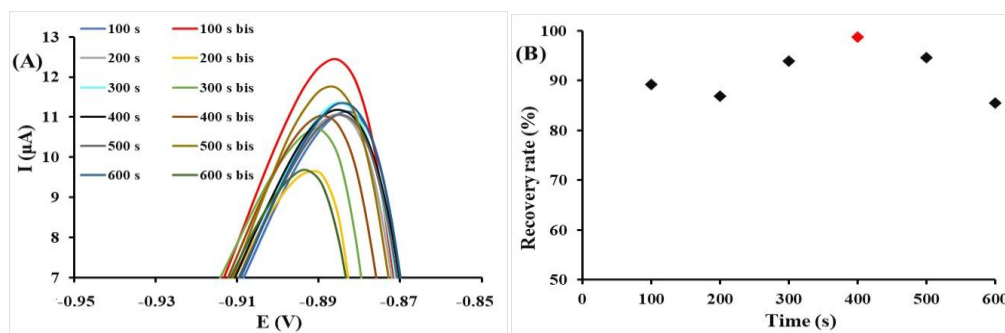


Fig. 10. (A) DPASV of 10 μM Pb²⁺ as a function of electrode cleaning time and (B) Lead recovery rate as a function of electrode cleaning time. electrode. Dp = -1.4 V / ESM Dt = 300s, pH = 2, Mt = 30 ms, Ps = 4 mV, Ma = 30 mV

Table 1. Method optimization parameters

Paramètres	Plomb
Deposit potential (V)	-1.4
Deposit time (s)	300
Cleaning potential (V)	1.4
Cleaning time (s)	400
Modulation amplitude (mV)	30
Modulation time (ms)	30
Potential step (mV)	4
Time interval (s)	0.1

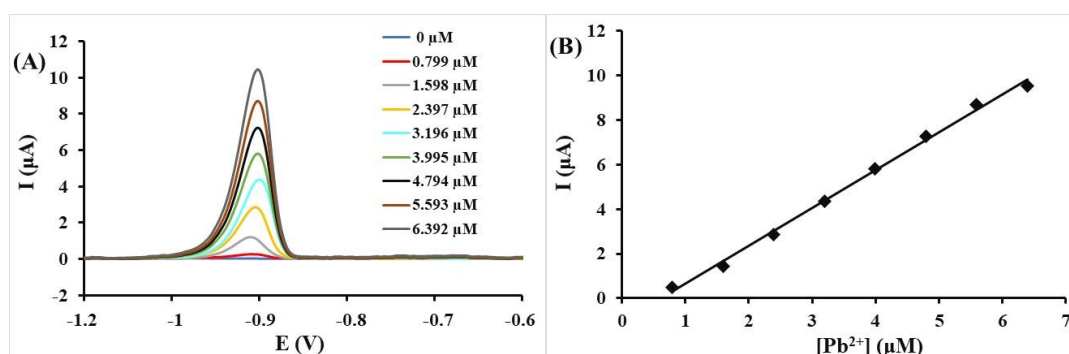


Fig. 11. DPASV of different Pb^{2+} concentrations; (B): Method calibration curve

3.3.2.2 Recovery and reliability rate

For the determination of the recovery rate we used six (6) samples of Pb^{2+} concentration. Three (3) independent experiments for each of the samples were performed. The results obtained were recorded in the form of recovery rate \pm Standard Deviation (SD) as shown in the following Table 2.

The recovery rate varies between 95.194 ± 0.242 to 102.034 ± 0.018 (Table 2).

The peaks of intensities of the six (6) concentrations of Pb^{2+} introduced ($[\text{Pb}^{2+}]_{\text{theo}}$) made it possible to calculate new concentrations of Pb^{2+} ($[\text{Pb}^{2+}]_{\text{exp}}$) with our method (the equation of the calibration curve). The curve (Fig. 12) obtained by plotting the theoretical concentration as a function of the concentration found is a straight line whose correlation coefficient is 0.991 very close to 1 and the equation is: $[\text{Pb}^{2+}]_{\text{exp}} = 0,9757 [\text{Pb}^{2+}]_{\text{theo}} - 0.0264$. The coefficient R^2 indicates perfect proportionality between these two types of concentration.

3.3.2.3 selectivity and interference phenomenon

Figs 13A and 13B show the voltammograms measured respectively in the presence of 6 μM Cd^{2+} and 6 μM Cu^{2+} in nitric acid medium (0.01M). In this figure, an oxidation occurs at 1.158 V / ESM in the presence of Cd^{2+} and an oxidation peak in the presence of Cu^{2+} at -0.319 V / ESM. These peaks correspond to the oxidation of metallic cadmium and the oxidation of Cu^+ to Cu^{2+} [31,32]. These voltammograms were measured by applying the various optimized values contained in Table 1.

The influence of some heavy metals on the detection of lead has been studied to demonstrate the effectiveness of our method. It

should be noted that the concentrations of Cd^{2+} and Cu^{2+} are approximately ten (10) times higher than the concentration of Pb^{2+} . The result obtained is shown in Fig. 14.

In this figure we observe that although the concentration of other ions (Cd^{2+} and Cu^{2+}) is much higher, we manage to detect lead at the same potential (-0.88 V / ESM). This indicates that this method of DPASV is selective with regard to the probabilities of interference that could occur when they are detected simultaneously.

In Table 3, it can be seen that in the presence of the other ions (Cu^{2+} , Cd^{2+}), the recovery rate of lead is almost the same as the recovery rate obtained with lead alone. This finding shows that the interference phenomenon is almost negligible on the surface of our electrode.

Up to now, Pb^{2+} was determined by various electrochemical methods using BDD electrode. A comparison of our findings with some literature data [33-36] is presented in Table 4.

Data in Table 4 clearly show that the detection limit of 0.052 μM (52 nM) determined in this work. This value is lower than those obtained by certain authors [33-35] who determined the detection limit for lead using a BDD electrode. This shows that our method can make it possible to make a good detection of lead. However, it should be noted that the detection limit of lead obtained in this work is greater than the value obtained by the HPLC method. Some authors have shown that the detection limit for lead by the HPLC method is less than 5 nM [37,38]. However, it should be noted that our detection limit is lower than the standards of the World Organization of health (WHO). Our method can therefore be used for lead detection in agricultural products because according to the

chemical information sheets of the World Health Organization (WHO), the allowable level for lead is 0.01 mg. L^{-1} and the maximum limit of Pb (II) in vegetables, adopted by FAO-WHO, is 0.3 mg.kg^{-1} (0.3 mg.L^{-1}) [39,40]. In addition, the detection limit obtained in this work is clearly lower than

the lead quantity obtained in food by several authors [41,42]. This detection limit (0.052 nM) is also lower than the lead quantity contained in human blood obtained by certain authors [43,44]. Thus our method can be effectively used for lead detection in food and human blood.

Table 2. Recovery rate of the method

Samples	Concentration introduced (μM)	Concentration found (μM)	Recovery rate (%)	$\pm \text{SD}$
1	0.799	0.815	102.034	0.018
2	1.598	1.532	95.889	0.076
4	2.397	2.315	96.579	0.073
4	3.196	3.246	101.557	0.051
5	3.995	3.803	95.194	0.063
6	4.794	4.666	97.338	0.024

Table 3. interference phenomenon on the BDD

Species	Concentration of introduced species	Concentration of Pb found	Recovery rate (%)	$\pm\text{SD}$
Pb^{2+}	$1.5 \mu\text{M}$	$1.479 \mu\text{M}$	98.6	0.064
Pb^{2+} , Cd^{2+} et Cu^{2+}	$1.5\mu\text{M} + 15 \mu\text{M} + 15 \mu\text{M}$	$1.418 \mu\text{M}$	94.53	0.072

Table 4. Comparison of the efficiency of certain voltammetric methods in the determination of lead (II)

Working electrode	Detection medium	Technique	LOD	Reference
BDD	0.2 M acetate pH 5	DPASV	$0.25 \mu\text{M}$	[33]
BDD	0.1 M HNO_3	SWASV	$0.10 \mu\text{M}$	[34]
BDD	0.01 M HNO_3	DPASV	$2.65 \mu\text{M}$	[35]
BDD	0.2 M KCl pH 1	LSASV	$1.99 \mu\text{M}$	[36]
BDD	0.01 HNO_3	DPSAV	$0.052 \mu\text{M}$	Present work

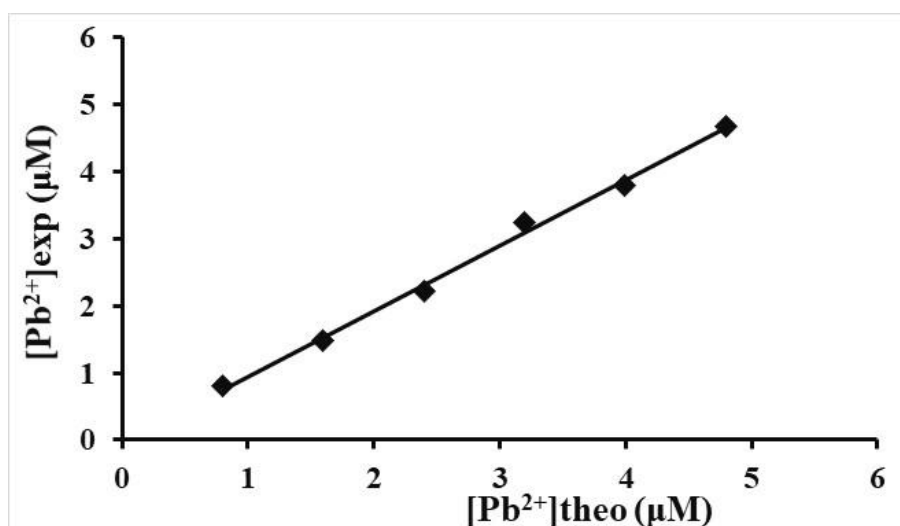


Fig. 12. Curve of the experimental concentration as a function of the theoretical concentration

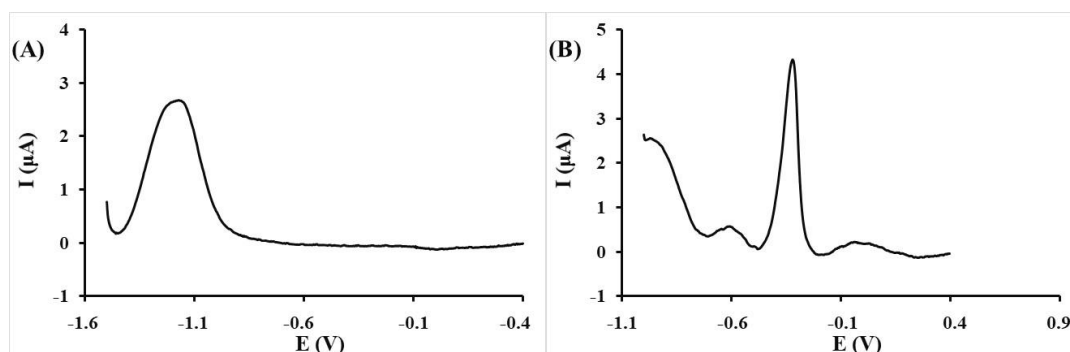


Fig. 13. (A) DPASV of a HNO_3 (0.01M) containing $6 \mu\text{M}$ in Cd^{2+} and (B) DPASV of a HNO_3 (0.01M) containing $6 \mu\text{M}$ in Cu^{2+}

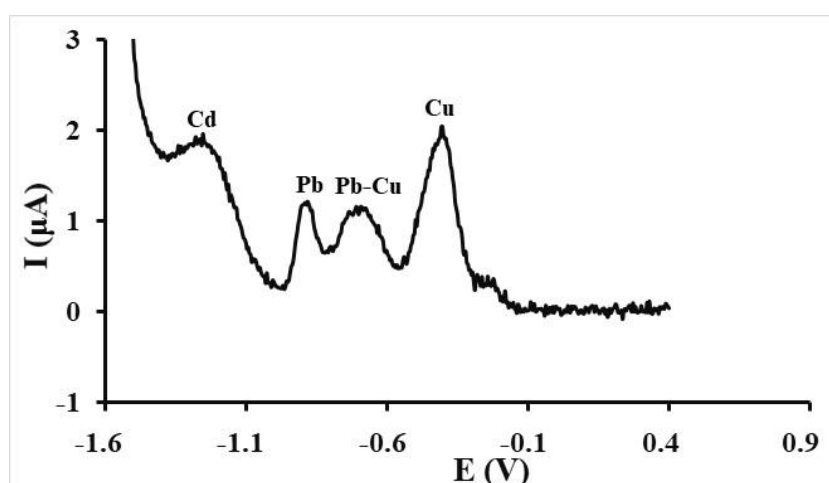


Fig. 14. DPASV of HNO_3 (0.01M) containing $15 \mu\text{M}$ Cu^{2+} , $15 \mu\text{M}$ Cd^{2+} and $1.5 \mu\text{M}$ Pb^{2+}

4. CONCLUSION

It appears in this work that the electrochemical method DPASV is a simple, sensitive and selective technique allowed us to detect lead (II) in nitric acid medium (0.01M). The results showed that the detection limit of Pb^{2+} is equal to $0.052 \mu\text{M}$ and the quantification limit equal to $0.173 \mu\text{M}$. This method made it possible to selectively detect and quantify the metal Pb (II) in the presence of other metals, Cd^{2+} and Cu^{2+} . In the presence of other metals, a recovery rate (94.53%) was observed which is close to the recovery rate obtained when Pb^{2+} (98.6%) was alone in solution. This technique can be used directly for the analysis of heavy metals.

DISCLAIMER

The products used for this research are commonly and predominantly use products in our area of research and country. There is absolutely no conflict of interest between the authors and

producers of the products because we do not intend to use these products as an avenue for any litigation but for the advancement of knowledge. Also, the research was not funded by the producing company rather it was funded by personal efforts of the authors.

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COMPETING INTERESTS

Authors have declared that no competing interests exist.

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