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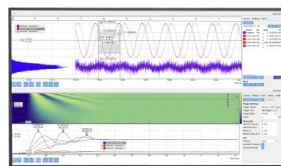
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Platinum Nanoparticle Electrode Electro-Chemical Lead (II) Determination with Square-Wave Voltammetry Modified with Iodine

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Abstract. Platinum nanoparticle electrode modified by iodine monolayer (PtNPs/I) has been designed to simultaneously determination of Pb²⁺ using square wave voltammetry (SWV), the analyzed the anodic peak current and anodic maximum potential is done. The scanning electron microscopy (SEM) and Energy-dispersive X-ray spectroscopy (EDX) demonstrate some fascinating characteristics of the uniform particle distribution, growth and self-assembly of the iodine-modified platinum nanoparticle. The also examined the effect of the concentration of lead and the effect of the scan rate on anodic top parameters. The results show that the anodic current peak increases and that the anodic peak potentials increase in comparison to the clean electrode to a negative value. The have noticed that lead and scan rate increases, anodic peak current increases. As the lead concentration and scan rate increase, the anodic peak potential changes to more positive values. The easily reproducible RSD 5% (n=5) removal peak current for heavy metal ion was shown under ideal conditions. For a pre-concentration time of 120 s, the linear ranges are between 1 to 100 µg. L⁻¹. For lead ions, the concentration limit was measured as 0.408 µg. L⁻¹ using the improved analytical process. The transmitter was a lead ion with a 0.215 µA/µM-cm² range, with a measurement maximum of 0.01 µM.

INTRODUCTION

Square Wave Voltammetry (SWV) is one of the most effective analytical tools to map the heavy metal particle. Voltammetry is an extremely sensitive method for the determination of heavy metals in different matrices [1-2]. As a result of its high reproducibility and sensitivity, mercury-setting electrodes are typically used to assess lead and cadmium [3-4]. The mercury free work electrode for the SWV, including gold, silver, platinum, bismuth and modified electrodes, was created due to the high environmental toxicity of mercury [5-7]. Platinum is one of the most inert elements, but is extremely active in some electro-chemical conditions. In electrochemical analysis, it has many advantages such as reliability, and low background current, high conductivity, reproducibility, and simple processing. [8]. The earlier studies showed that the surface area and conductivity of platinum nanoparticles could increase with good catalytic properties to electron transfer and the high catalytic effectiveness of platinum nanoparticles has demonstrated a great appeal [9]. Another metal is needed to provide oxygenated species (source) which cause inactivity of Platinum. Some techniques have been proposed to clarify this thought: bifunctional mechanism, electronic properties alteration of the base metal and the third body effect [10]. The Pt alloy with a second metal (M = Sn, Ru, Au, Bi, Mo, Pb, Pd, Bi and Os) is an efficient way to enhance the electrocatalytic and electroanalytical properties (stability and activity) and decrease the use of costly Pt in electrocatalysis [11-13]. The exemplary alloys showed better catalytic efficiency than the monometallic platinum catalysts. Recently, Bi-metallic materials (Pt-Metal), such as core/shell structures, plates, aggregates, sheets, and spheres were of great interest, especially as electro-analysis were theirs [14-16]. For electrochemical analysis and selective determination of lead, cyclic, linear and square-wave voltammetry in pharmaceutical formulations has been investigated [17]. Interesting work was found in the new era in the design and development of nanoparticles sensors [18]. In particular, the highly interesting structure of the surface, good electrical and mechanical characteristics, solid stability and a low aggregation have been found to be the perfect supporting materials for the electrocatalytic [19]. The aim of this work is to studied detection of Pb²⁺

at trace levels by electrochemical voltammetry method using platinum nanoelectrode modified by Iodine (PtNPs/I) in aqueous solution employing electrochemical.

EXPERIMENT

CPA-HC5 auto-label electrochemistry analyze (Hanoi, Vietnam) using a three-electrode configuration platinum wire as counter-electrode, Pt nanoparticle in tantalum surface as working electrode and Ag/AgCl as reference. Electrochemical experiments were carried out using potentialostatic (273A, Princeton Applied Research). Analytical efficiency or the best commercially available purity of reagents is used. All the analytical grade chemicals were made by Merck (Germany). $\text{H}_2\text{PtCl}_6 \cdot 6\text{H}_2\text{O}$, H_2SO_4 and Lead stock solution (1000 ppm) were purchased from Merck. Both treatments were formulated using distilled water.

The platinum nanoparticles were electrodeposited to a bare tantalum electrode in H_2SO_4 0.5 M solution, with a constant potential of 1.0 mM H_2PtCl_6 of -0.2 V and 120s deposition time of [20]. The electrode Pt/Ta was then carefully washed before use with purified water. The platinum surface was modified by dipping the superficial electrode into a solution containing iodine cations in open-circuit conditions. The cation concentration in such cases corresponds to the solubility of 0.5 M sulfuric acid of the salt unit. Electrochemical tests of the proposed sensor were conducted with square wave voltammetry at room temperatures (25 ± 1 °C).

The SWV tests were performed in 0.5 M sulphuric acid in 0.050 s; 0.03 s phase time; 0.25 V s^{-1} sweep rate. In the range of scans -0.2 to 1.2 V the Pb^{2+} ions were eliminated and the peak currents measured. Using normal addition procedure (3 additions), electrochemical quantitative identification of metal ions was performed. After through normal introduction per analytical approach and approach and an average of three runs has been measured. The PtNPs/I electrode surface was electrochemically cleaned after every measurement. The water delivery system (Province of Binh Dinh, Jordan) gathered all the local water samples. This was sampled by HNO_3 (pH=2) and purified by a glass fiber filter of 0.45 μm . The samples were then digested for 90 minutes by UV digesters to decompose organic compounds to simple ones, after adding 30 per cent H_2O_2 to each sample. Before the SWV analysis.

RESULTS AND DISCUSSION

Characterization of Pt nanoparticle electrode modified by iodine

The synthesized Pt nanoparticle electrode modified by iodine was characterized for functional groups using (SEM, scanning electron microscopy), (CV, cyclic voltammetry) and (EDX, energy-dispersive X-ray spectroscopy).

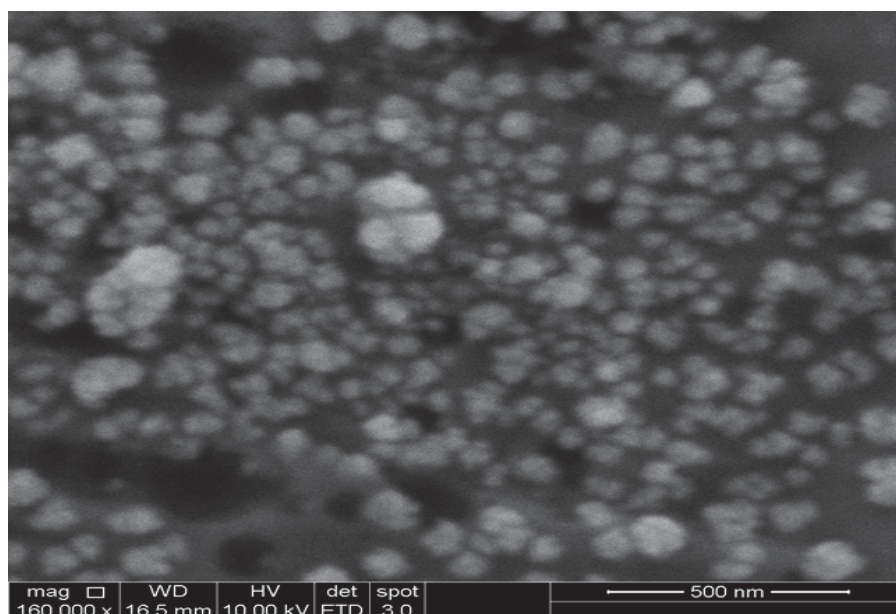


FIGURE 1. The SEM micrographs of Pt nanoparticle electrode modified by iodine chart.

Figure 1 shows the typical SEM image obtained for the electrodeposited Pt nanoparticle electrode modified by iodine. Herein the SEM image has been obtained for 500 nm surface areas. The SEM image shows that the Pt nanoparticles are uniformly dispersed on the electrode surface. The identity of the Pt nanoparticle electrode modified by iodine is further confirmed by the EDX spectrum of the deposited particles (Fig. 2).

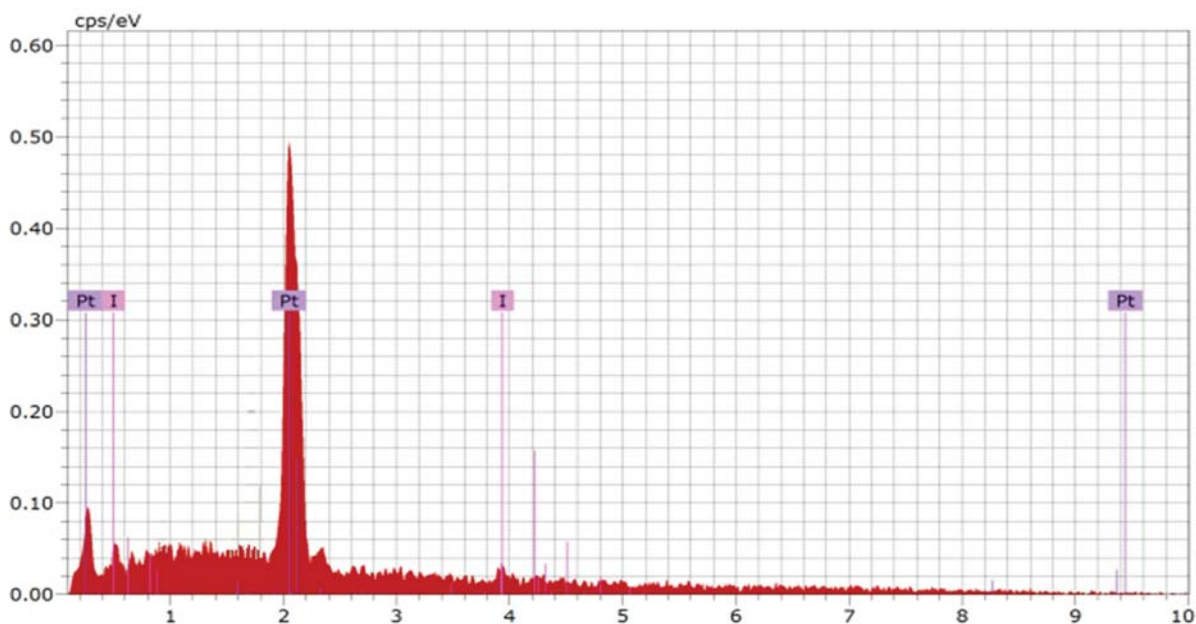


FIGURE 2. The EDX spectra of Pt nanoparticle electrode modified by iodine.

TABLE 1. Spectrum Acquisition.

Element	AN	Series	Unn. C [Wt. %]	Norm. C [Wt. %]	Atom. C [at. %]	Error [%]
I	53	L- Series	9.49	11.82	17.08	0.7
Pt	78	M- Series	70.80	88.18	82.92	2.9
Total	-----	-----	80.29	100.0	100.0	-----

The electrochemical behavior of Pt nanoparticle electrode modified by iodine was studied using cyclic voltammetry at a scan rate of 100 mV/s. The Figure 3 shows the iodine coated platinum nanoparticles electrode representative voltammograms recorded in 0.5 M H₂SO₄ (dashed line) superposed to the process voltammograms of the bare platinum electrode nanoparticles (solid line). The absence of redox (oxidation/reduction) activity and in particular oxygen and hydrogen adsorption indicates a near total surface suppression between the hydrogen production limits (around -0.2 V) and surface iodine suppression levels (around 0.95 V). The iodine is partially deorbited when the potential of the electrode with iodine-coated scans is above 0.95 V. The restoration, between its hydrogen and the oxygen production limits, of cleanness in the surface that involves the daily washing of the work electrode and the compartment requires a number of possible cycles. A 1.18 V based peak displays a platinum nanoparticle electrode with iodine electrooxidation. Nevertheless, the iodine is stable if the potential electrode does not exceed 0.95 V. Therefore, the practical analytical potential range usually varies from -0.1 to 0.95 V.

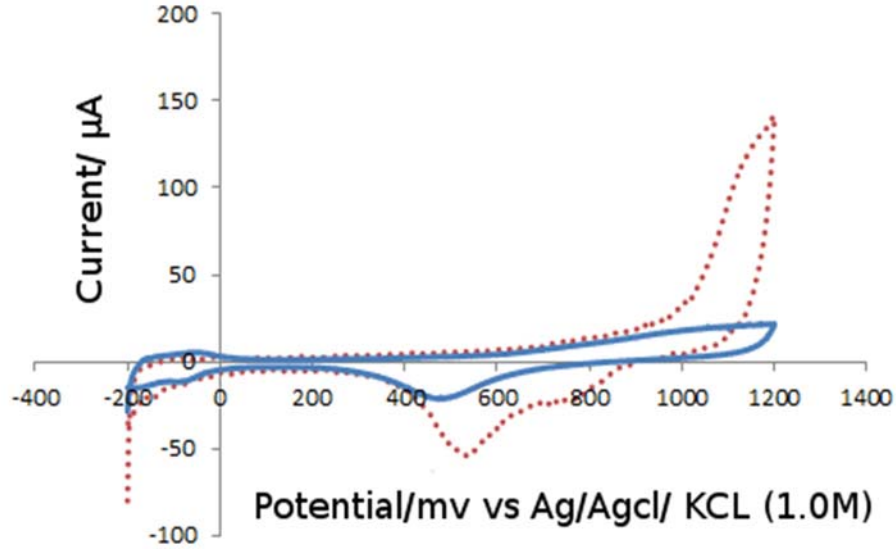


FIGURE 3. Voltammograms of platinum nanoparticle electrode (solid line) and iodine-coated platinum nanoparticle electrode (dashed line). At a scanning rate of 100 mV/s both voltammograms were registered in 0.5 M H₂SO₄.

The effect of stripping signals over a pre-concentration time was studied over a span of 60 to 300 s at 10 µg. L⁻¹. More Pb²⁺ is replaced and adsorbed on the PtNPs/I surface as the pre-concentration time increases, increasing the strip signaling rate. Figure 4 revealed a linear rise in the Pb²⁺ stripping peak currents in the pre-concentration duration observed.

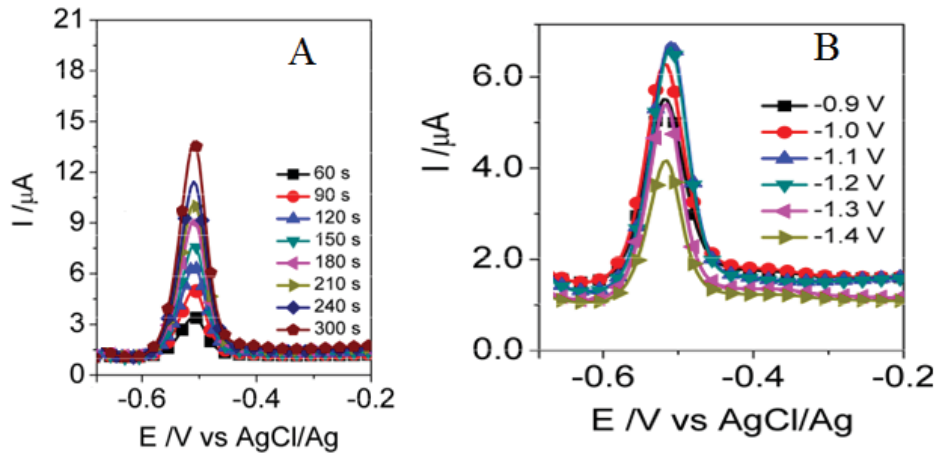


FIGURE 4. (A) Effects pre-concentration time (B) Effects pre-concentration potential on the stripping peak current of 10 µg. L⁻¹ Pb²⁺ on platinum nanoparticle electrode.

Figure 4-B showed the effect of pre-concentration potential on the elimination of peak current at a pre-concentration period of 120 seconds for the electrolyte with a 10 µg L⁻¹ Pb²⁺ enabling capacity range from -0.9 to -1.4 V. The Pb²⁺ peak current was improved with a potential shift from -0.9 to -1.2 V as well as a near constant rise between -1.1 and -1.2 V as a previously established possibility.

Amplitudes of 10 to 100 mV at the 6 mV phase potential vector for the purpose of pulse amplitude. After the pulse amplitude increased it was found that the peak current decreased by linear equations (Figure 6) in $I_p, Pb = 1.283 + 0,065 \Delta E, R^2 = 0,979$. This is sufficient in the case of $I_p = K.\Delta E$ However, as the pulse amplitude is raised, E_p is moved towards the negative side and the duration is extended, thereby enhancing the effect of other materials. Consequently, the amplitude for the next experiment was chosen to be 60 mV.

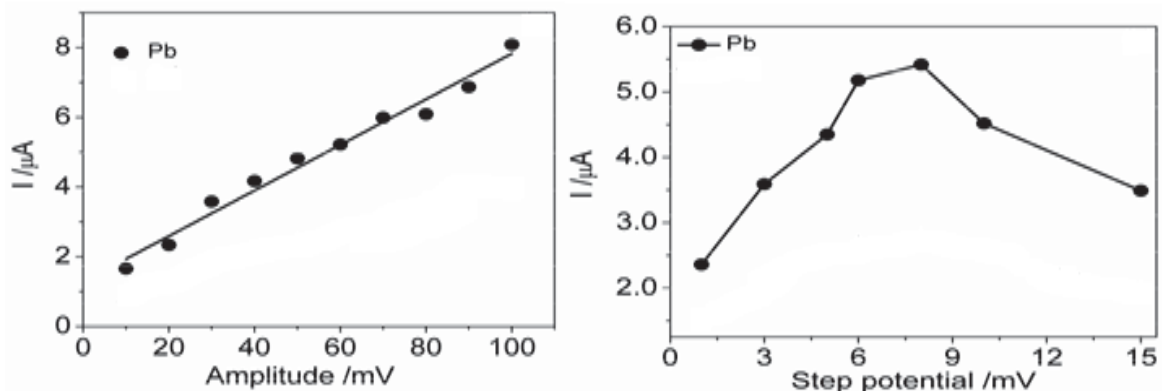


FIGURE 5. Effects of different SWV parameters on Pb^{2+} response: (a) Amplitude, (b) Step potential

Linear scale: The linear association between the Pb and I_p values for led with a Pb^{2+} correlation coefficient of 0.998 was strong in the range from 1 to 100 $\mu g. L^{-1}$. Figure 7 showed SWV and linear metal regression lines / equations. Detection limit (LOD): The detection limit was calculated at 0.408 $\mu g. L^{-1}$ of Pb^{2+} in 10 blank solution replications, respectively, under optimum conditions. For Pb^{2+} stripping peak currents, SWV with PtNPs/I achieved high reproducibility. The reproducibility RSD value was estimated to be 4.65% for Pb^{2+} , indicating a robust manufacturing process ($n = 5$).

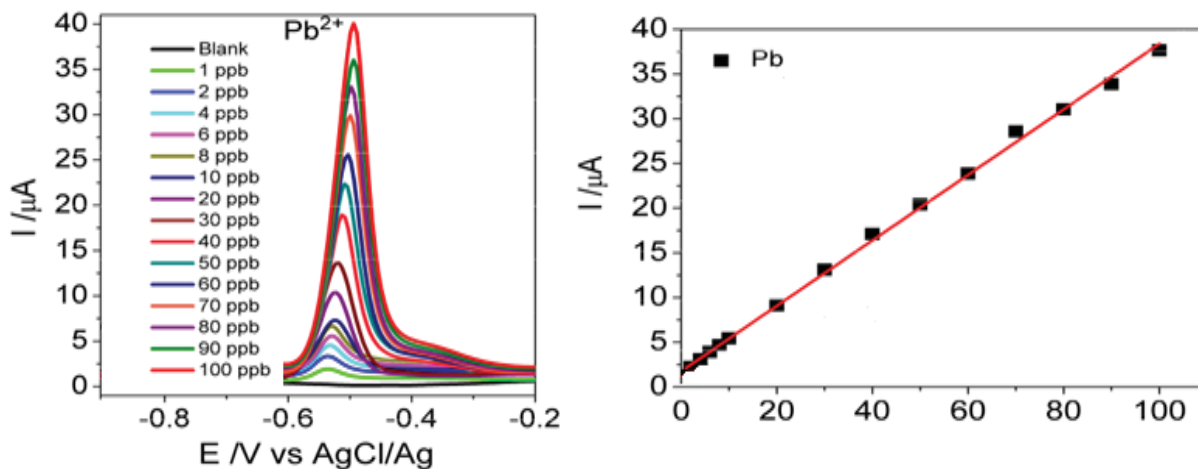


FIGURE 6. SWV of Pb^{2+} samples of 1 to 100 $\mu g. L^{-1}$ concentration and relation between concentrations of metal ions at the relevant maximum current range.

The electrocatalytic activity and current density–time responses for of lead oxidation at its fixed potential from 0.4 up to 0.8 V in Figure.7 of iodine-modified PtNPs. The Pt nanoparticle electrode modified iodine shows the highest current density (53 $mA cm^{-2}$), while in 0.3 seconds the Pt nanoparticle electrode shows a present density of 17 $mA cm^{-2}$. These data show increased operation and iodine stability. The modification of metal, which can enhance catalytic effectiveness and the bifunctional mechanism or structure of the ligand, can also explain these findings.

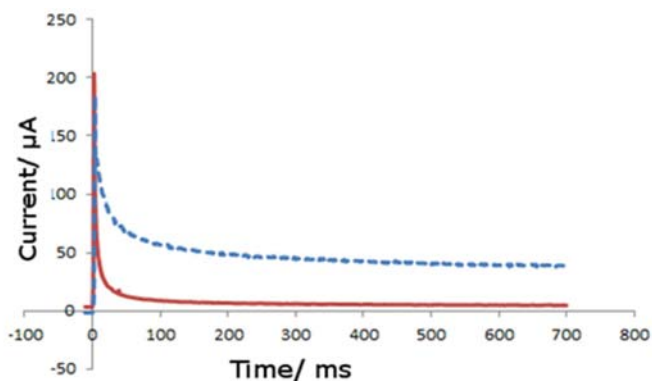


FIGURE 7. Current vs. time as potential kept at 0.4 to 0.8 V in 0.5 M H₂SO₄ on of iodine-coated platinum nanoparticle electrode (dashed line), and platinum nanoparticle electrode.

CONCLUSION

According to the results, we can conclude that the nanoparticle platinum electrode containing the iodine monolayer, modified electrode, presents electrocatalytic activity and good selectivity for iodine. This electrode can therefore be improved and used as a sensor for the identification of iodine in natural systems. Pt nanoparticle electrode, modified by iodine, showed better electrocatalytic activity against iodine oxidation and was able to react with iodine over a broad range of well stable concentrations with good sensitivity (0.215 µA/µM-cm²). In certain local waters, the platinum nanostructured electrode, modified by iodine adatoms, was used to evaluate lead with good results in line with the square wave voltammetry methods. The sensor suggested demonstrated a high electrochemical sensitivity to small amounts of ions Pb²⁺ with a weak Pb²⁺ 0.408 µg. L⁻¹ detection.

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