

Electrochemical Sensors and Determination for Heavy Metal by Rotating Disk Platinum Electrode and Chronoamperometric Method

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ABSTRACT

Pollution by heavy metals is one of the most severe environmental issues that threaten global sustainability. This review presents a recent advance in electrochemical sensors for heavy metal detection. Rotating Disk Platinum Electrode are discussed. This study on the production of a modern natural water electrochemical antimony (III) and copper (II) test include the use of platinum electrode. Antimony and copper were pre-concentrated on the modified electrode surface and adsorbed to the surface, oxidizing at $E = 540$ mV and $E = 85$ mV, respectively. After 20 min of accumulation, time the best-defined anodic peak was obtained of surface. The precision was tested by carrying out chronoamperometric measurements at a concentration of Sb^{+3} and Cu^{+2} 8.5×10^{-8} M and 9.5×10^{-7} M, respectively.

Keywords: Heavy Metal, Chronoamperometric, Platinum Electrode, Rotating Disk

1. Introduction

Pollution from heavy metals challenges environmentally friendly structures and poses a great global sustainability challenge. Heavy metals are the major contributors to water contamination, resulting in a severe human tragedy. As cities are expanded and industrialization is developed, waste sewage from life and industry are drained into the water environment. Heavy metals, bio-concentration, food chains, and through potable water, are severely threatening human health (Shen. et al., 2017).

Electrochemical sensors have gained attention in recent years in the area of atmosphere control and heavy metal detection. It has several advantages, online analysis, and ease of use, including simple equipment, high sensitivity and on-site testing. The TLFC towing distribution was simulated using the Finite Element Method (FEM) and electrical form and configuration were also optimized (Chmayssem, and Hauchard, 2017).

All three electrodes were manufactured on the basis of carbon nanotube (CNT) thread in an electrochemical cell. As the representative method of this analysis, Pb^{2+} , Hg^{2+} and Cu^{2+} were used. The detection limit for Pb^{2+} , Hg^{2+} and Cu^{2+} , estimated on the basis of this method, with 120 s of deposition time is 1.05, 0.53, and 0.57 nmol, L^{-1} , respectively. This study shows that a low-costs copper (Cu) electrochemical sensor is used to measure lead (Pb) in surface water samples. Our copper-based sensor has a low-cost material copper electrode that provides simple manufacturing and competitive electric chemistry detection performance (Alkhalaf, and Alkhalaf, 2020).

For both metal ions of 120 s deposition time and a potential deposition of -1.2 V in pH 4.5 acetate buffer solution, the lineal range of a Bismuth film electrode was from $\mu g L^{-1}$ to $100.0 \mu g L^{-1}$. The electrode was also applied in soil sample extracts to determine cadmium and lead. While the electrochemistry provides a simple method of metal detection on a micro scale, it is difficult or expensive to produce traditional carbon, gold (Au) or platinum (Pt) electrodes that prohibit the widespread use. Zinc detection (calculated) 140 nm (9.0 ppb), and a sensitivity above $1 \mu A \mu M^{-1}$ in the acetate buffer were shown by anodic stripping voltammetry using our new copper-based sensors (Alkhalaf et al, 2020).

Thereby, a micro-sensor (μCS) for trace heavy metal detection of highly sensitive, robust and cost-effective electro-chemical carbon-based sensors is provided. Detection limits down to $1.8 \mu g L^{-1}$ for Pb^{2+} and $1.2 \mu g L^{-1}$ for Cd^{2+} can be achieved over the μCS (Shen et al, 2017).

The chronopotentiometry of derivative stripping has been used to determine reliably, sensibly and quickly Pb, Zn, Cd, Cu, Mn, Ni, and Se in hydrochloric extracts from the CEO; detection limits of $< 1 \mu g kg^{-1}$ were obtained for all studied metal under optimal electrochemical conditions. The results showed that Mn was the largest metal in all the CEOs, followed by Zn, Se, Cu, Pb and Ni. The concentrations of Cd were often smaller than the maximum ($0.6 \mu g kg^{-1}$).

The cost-efficient technology which could make for a three dimensional combination of microfluidic colorimeter and electrochemical paper-based analytical devices (mPAD) accessible to the developing world as well as to the developed world (Hourani and Alkawaldehy, 2016).

The exfoliated ZrP is shown to be the best possible adsorption to Pb (II) between all heavy metal ions, resulting in selective detection. The nanocrystals of octahedral Fe₃O₄ exhibit better electric sensor output in comparison with cubic heavy metal ions investigated such as Zn (II), Pb (II), Cd (II), Hg (II) and Cu (II). Finally, the potential application of Fe₃O₄ nanocrystals in drinking water to electrochemical detection for As (III) is demonstrated as a test to find a disposable platform totally free of any noble metals. We have assembled a two-dimensional silica/gold spherical cavity microelectrode array using the monolayer template of self-assembly Polystyrene microspheres and the pulse electrodeposition method to test the performance of this method using the square wave pulse voltammetry method (Altweiq and Alkhawaldehy, 2019).

The array has a greater capacity to resist Cu (II) interference. Hydroxyapatite Nano-sized has been developed and used for the detection of Pb²⁺ with a width from 20-25 nm and length of 50-100 nm as a special 3D network structure with high adsorbent capability towards Pb²⁺. Nafion, the polymer for cation exchange, is used as a conductive matrix where NHAP and the ionosphere can be closely bound to the surface of the electrode. With an accumulation time of 10 minutes at the open circuit potential, the electrode has linear range between 5.0 nmol and 0.8 μmol (Lin and Huang, 2017).

In real samples a new voltammetric process for measuring a trace lead (II) in a poly (4-vinylpyridine)/mercury film electrode (PVP / MFE), was developed through anodic square wave stripping voltammetry. The principal advantages are good interference resistance, fast identification and good electrode renewal without deoxygenation. Two modification methods were used: HAuCl₄ electrochemical reduction and Au NPs stabilized by citrate electrostatic adsorption. These and other studies have led us to believe that the Au NPs have acted as nuclear deposition plants for Hg and that the GC or ITO have been superior in mercury removal. In fact, with an ITO surface modified with electrostatically absorbed Au NPs, a remarkable detection limit of 1 μm·L⁻¹ of Hg was achieved (Alshamaileh, et al, 2016).

In the work discussed in this article, an appropriate method for the analytical determination of heavy metal samples has been investigated with the advantage of the use of Platinum/palladium electrodes in conjunction with chronoamperometric and square wave voltammetry.

2. Experimental

Both chemicals were listed as reagents and used as approved. By diluting certain chemical compounds into Milli-Q water system), aquatic solutions had been prepared. A water solution path containing the combination of the platinum/palladium electrodes used in this work. The average volume of the sample in the electroanalytical chemical cell was 20 mL in each trial. This contains platinum/palladium electrodes in glass cell, the reference electrode Ag/AgCl: 1 M of KCl and the platinum counter electrode. Experiment conditions of rotating disk voltammograms: Scan rate is 50mv/s, rotation speed is 1000 RPM, scan range is -0.2-0.85 V and peak potential is 0.724v.

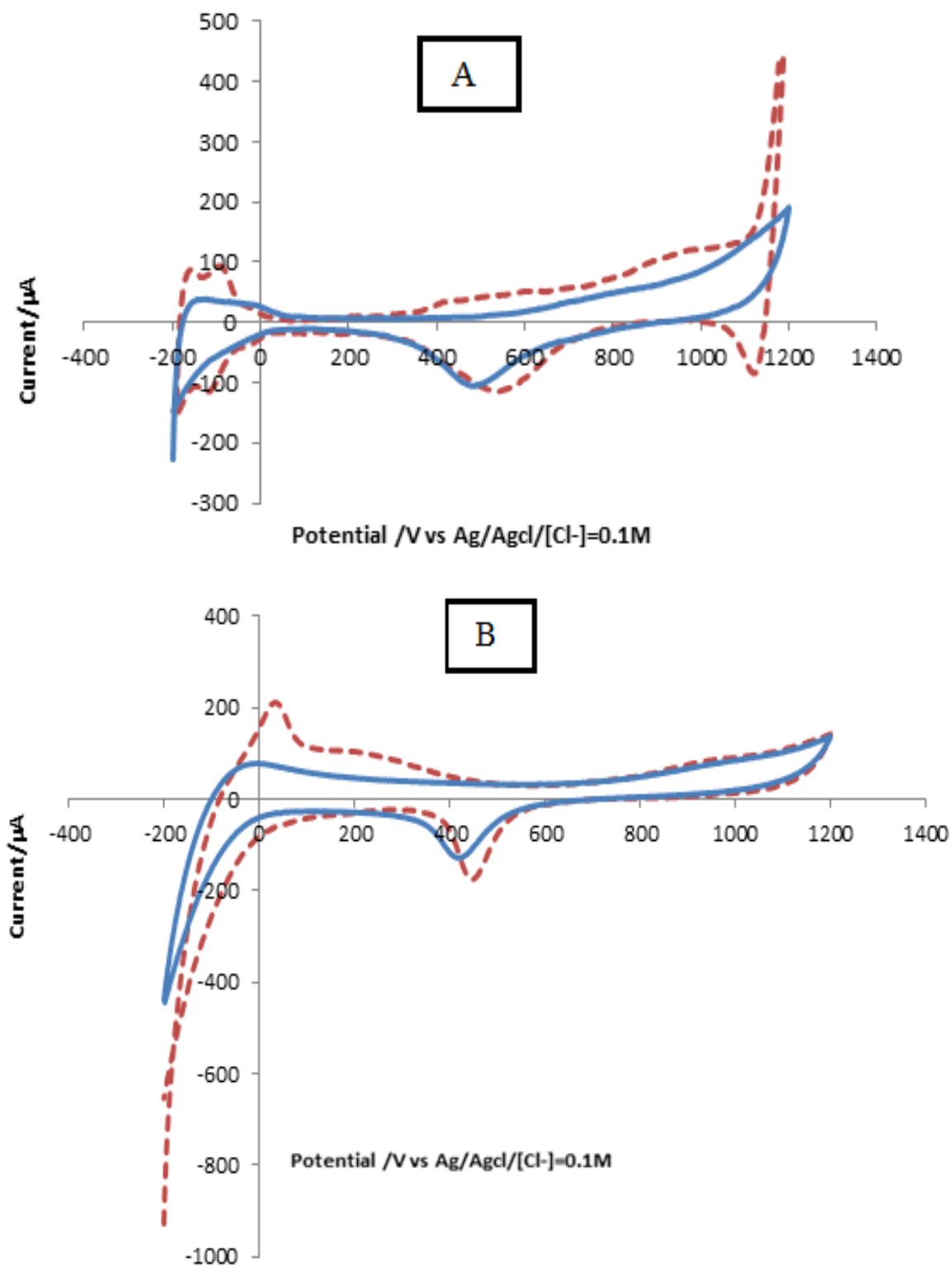
3. Analytical Procedure

First, the electrode was submerged in a heavy metal solution, which produced a chemical accumulation in an open circuit for iodine monolayer. The electrode was then removed from the electrode, washed in water and relocated to the real voltammetric membrane in which only one electrolyte (0.5 M H₂SO₄) was present. Different protocol in sample processing and in room temperature electrochemical experiments were performed.

The same electrochemical systems mentioned above have been used for this calculation before and after immersion in heavy metals containing solution. For AC voltage of 5-10 mV at 0 mV the frequencies from 100 KHz to 10 MHz can now be calculated. After 30 minutes of exposure, computer programs automatically test measurements made at rest potentials. Experiments have been carried out using the open circuit square-wave voltammetric system by deposit of the heavy metal. At the potential -1.0 to -0.4 V the accumulated metal was then anodized with a phase potential of 25 mV; 5 mV amplitude, 5 sec amplitude and 1 mV/s scan rate.

4. Results and Discussion

Figure 1 indicates the platinum electrode concentration of Antimony (II). With cyclic Voltammetry (CV), the voltammograms were obtained at an appropriate scan rate of 100 mV s^{-1} . Before a pre-concentration phase in Sb (II) solution, the base curve, showing no point, was obtained. As shown in Figure 2, after the pre-concentration phase the peak current revealed that the Sb (II) had been removed from the bulk solution by platinum electrode at different time immersion.



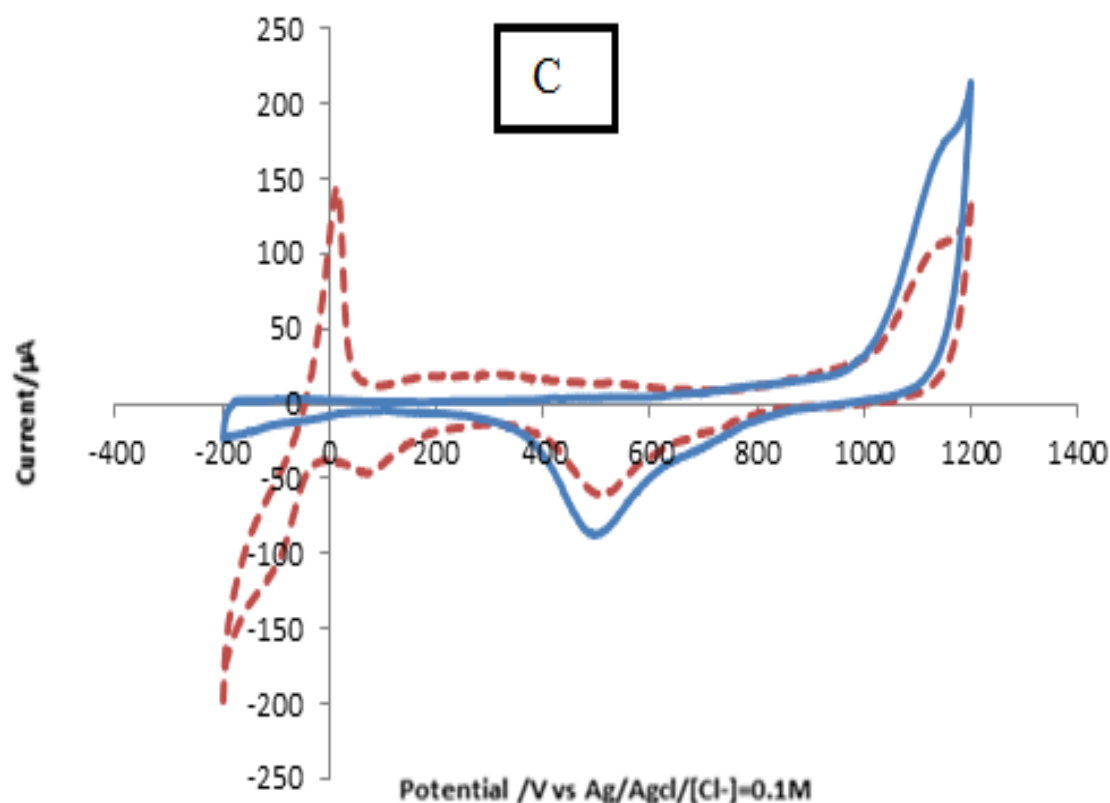


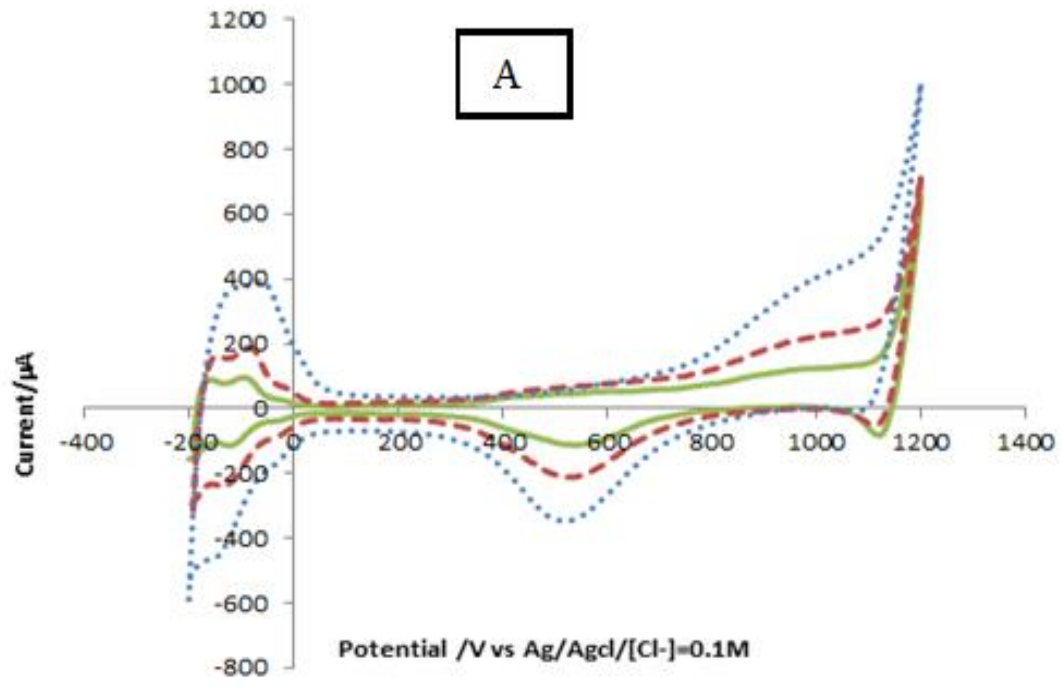
Figure 1. Cyclic voltammograms of platinum electrode before and after incubation with Antimony (II). The scan rate was 100 mV s⁻¹. (A) [Sb (II)] = 3x10⁻⁴ M and (B) [Cu (II)] = 5x10⁻³ M (C) [I] = 4x10⁻² M.

Figure 1 displays the rotating disk platinum electrode impedance diagram in 3X10⁻⁴ M of Antimony and 5x10⁻³ M of copper is solution, prior to and after pre-concentration phase. The data demonstrate explicitly that when the Pt is subjected to antimony (II), its capacitance at the interface is increased. In the event of adsorption on the electrode surface, this finding is in line with the literature (Alkhalwaldeh et al, 2020). As part of an optimized process that results in electrolytic reduction of the heavy metal in the changed coating, we offer empirical evidence for the Sb (II) adsorption on the surface. The observed decrease of the resistance to charge transfer often results in the conductivities of the additive electrode that can be clarified with the inclusion of Antimony on the surface of the electrode.

The layer powder on platinum surfaces was analyzed by cyclic voltammetry on the electrode before and after the pre-concentration stage in antimony solution. As seen in Figure 2, Sb (II) and Cu (II) has been observed with a pair of stable and well-defined redox peaks. This findings suggest that after pre-concentration in the antimony solution Sb (II) and Cu (II) are present in or / and appetite coating platinum electrodes. The result reveals that the platinum electrode surface is an important element in the Sb (II) and Cu (II) accumulation cycle, indicating the systemic manipulation of anodic surface processes by cyclic voltammetry with some critical advantages.

The Platinum electrode immersion time in Sb (II) and Cu (II) solution had a significant effect on the surface state and the impedance of the electron transmission. It can be shown that with the introduction of the adsorption time, the diameter of the semicircle has decreased to greater frequencies. There was no dramatic improvement at the interface electron transmission resistance, suggesting that Sb (II) and Cu (II) adsorption exceeded saturation point, when the soaking time in the antimony solution was more than 20 min (in Figure 2).

The tests of these antimony ultra-traces are shown to be extremely suitable in water samples where a maximum suggested residual stipulation is 10 μg/kg suggesting that antimony (II) can be studied in natural water samples (Almatarneh et al., 2019).



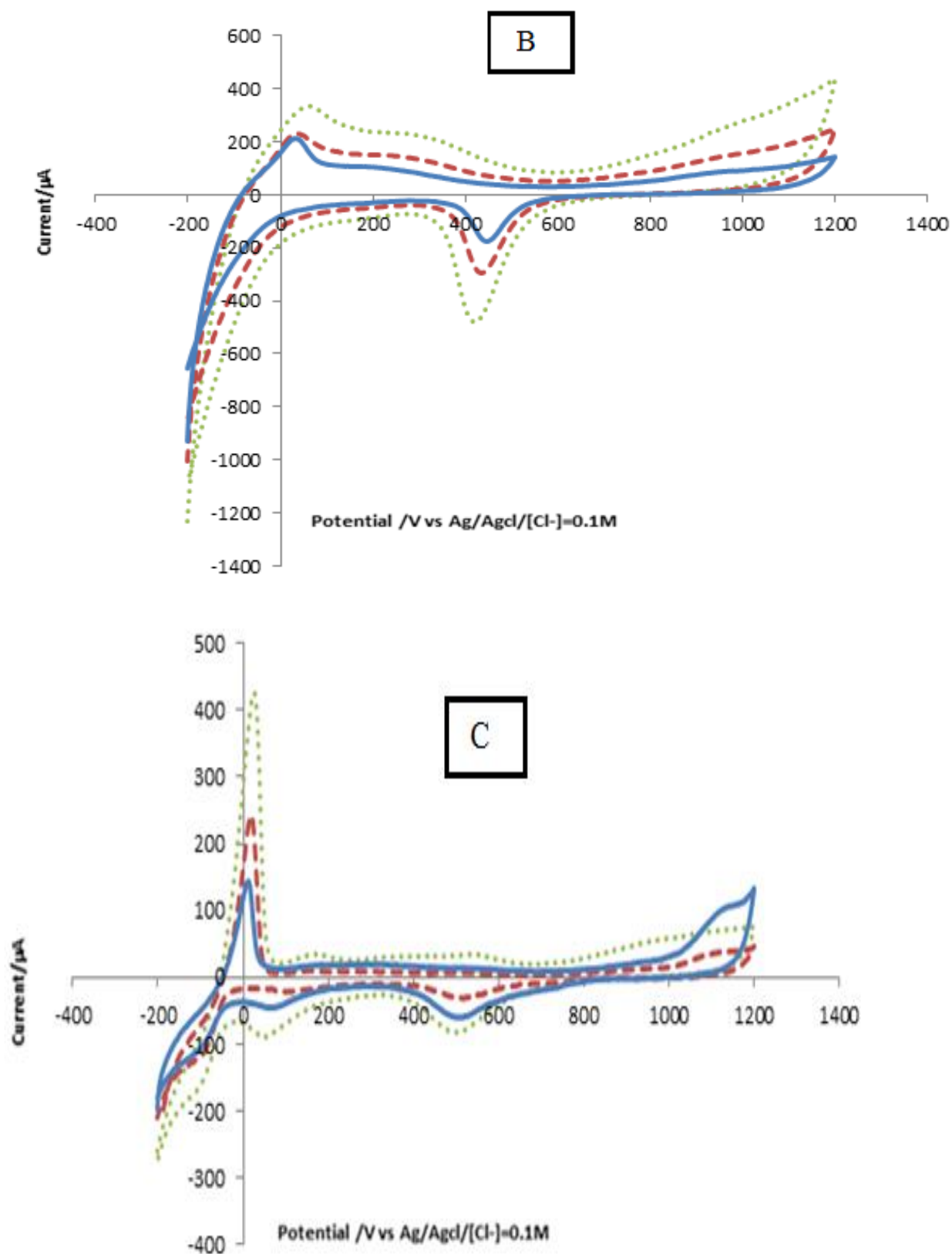


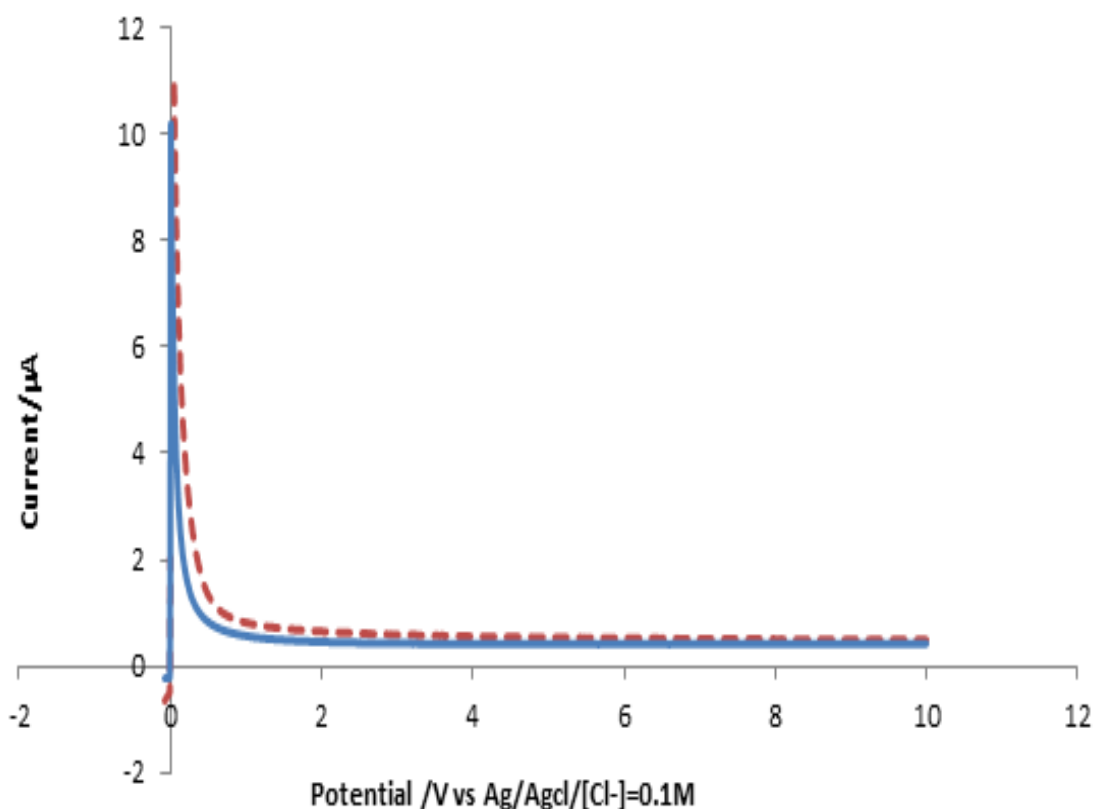
Figure 2. Cyclic voltammograms of platinum electrode with Antimony (II) at different immersion time, (the immersion time is 10, 15, and 20 min). During scan rate 100 mV s^{-1} ; concentration of (A) $[\text{Sb (II)}] = 3 \times 10^{-4} \text{ M}$ and (B) $[\text{Cu (II)}] = 5 \times 10^{-3} \text{ M}$ and (C) $[\text{I}] = 4 \times 10^{-2} \text{ M}$.

The layer powder on platinum surfaces was analyzed by cyclic voltammetry on the electrode before and after the pre-concentration stage in antimony solution (Figure 3). As seen in Figure 4, Sb (II) has been observed with a pair

of stable and well-defined redox peaks. This findings suggest that after pre-concentration in the antimony solution Sb (II) is present in or / and appetite coating platinum electrodes. The result reveals that the nanoparticle is an important element in the Sb (II) accumulation cycle on the platinum electrode surface, indicating the systemic manipulation of anodic surface processes by square wave voltammetry with some critical advantages.

The tests of these antimony ultra-traces are shown to be extremely suitable in water samples where a maximum suggested residual stipulation is 10 $\mu\text{g}/\text{kg}$ suggesting that antimony (II) can be studied in natural water samples. The lower concentration detection is 0.4 ppm and higher concentration of metal is 100 ppm.

The test plot produced in the above conditions (20 min deposition time) as shown in Figure 3. Linear reaction to a sensitiveness (path) of 0,0301 μA per antimony and copper 1 μmol ($R^2= 0,991$) was reached in platinum nanoelectrodes to 20 M. The lower detection limit is 3 and the quantitative limit is 4, which were five times the normal variance of the results obtained for a white person, were 8.5×10^{-8} M and 9.5×10^{-7} M respectively.



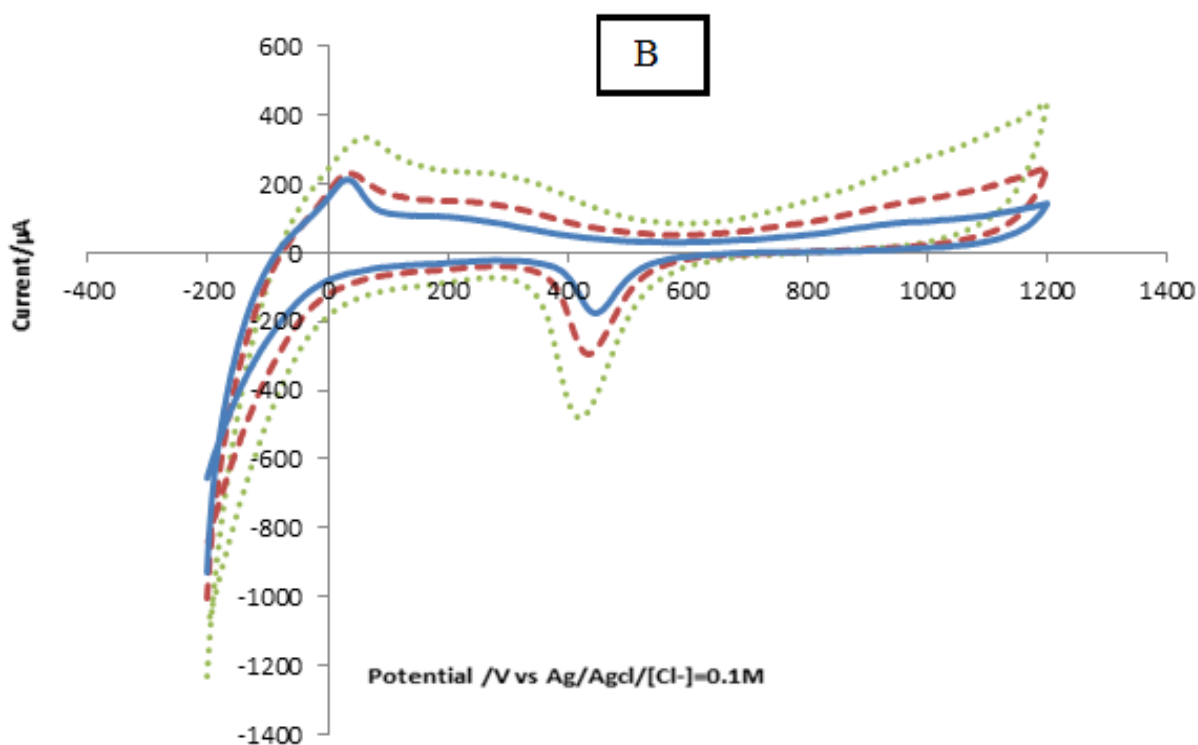


Figure 3. CA of antimony and copper calibration plot generated using the optimum conditions determined at pH 6.5 and 0.2 mol.l^{-1} Antimony nitrate.

The method for electroplating the electrode surface with bismuth is critical to obtaining satisfactory performances. Three methods exist to realize a bismuth deposit. The first method, *ex situ* plating, consists of electroplating the bismuth film before transferring the electrode into the sample solution for analysis. The plating conditions are variable but acidic media are recommended as bismuth ions are easily hydrolyzed as high pH. Plating solutions containing $5\text{--}200 \text{ mg}\cdot\text{L}^{-1}$ Sb (II) are used with

5. Conclusions

Electrochemical sensors have a number of advantages as surveillance tools: they have a high sensitivity, good stability, low costs, and simple operation. It is used widely to track emissions, life sciences, food and clinical diagnosis. It was shown that with cyclic and square wave voltammetry, one well-defined anode peak could be achieved at a power of 0.74 V versus Ag/AgCl. The Sb oxidation accumulated on platinum nanoelectrode takes place. Antimony (II) detection limit of $8.85 \cdot 10^{-8} \text{ mol L}^{-1}$ was much higher than those mentioned in the literature when experimental conditions for square wave voltammetry were optimized, and this sensor displayed a broad linear reaction range, good sensitivity, and reproductively. Nevertheless, nanoparticle platinum was the only electro catalyst that would perform the reaction at an acceptable rate. This led to a substantial search for the theoretical electro-catalysis of the square wave voltammetry.

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