

Voltammetric Study of the Redox Current Peaks of Pb(II) Mediated by GCE in Normal Saline

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Abstract: Lead ion of sulfate was studied electrochemically analysis, using cyclic voltammetric technique by glassy carbon electrode (GCE) in normal saline solution (0.9%NaCl). The results of the study have been found oxidation and reduction current peaks of lead ion in normal saline at -550 mV and -650mV respectively. It was studied the electrochemical properties of the redox current peaks of Pb(II) with different reagents such as glucose, uric acid, urea and ascorbic acid in normal saline. The redox current peaks of Pb(II) were enhanced through using each of glucose, uric acid and urea, but in ascorbic acid the reduction current peak was reduced. Also, the other phenomena of the reduction current peak of Pb(II) was disappeared when using blood as an electrolyte because the lead ions act as oxidant pollutant in blood medium.

Keywords: Pb(II), Cyclic Voltammetry, GCE, Ascorbic Acid, Urea, Uric Acid, Glucose

1. Introduction

Lead compounds in environment causes pollution in air and officiates toxicity of human body. The scientists focused to studying the redox reactions of the pollutants such as heavy metals Mn(II), Hg(II), Cd(II) in different electrolytes using cyclic voltammetric technique [1-4].

Lead (II) sulfate (PbSO_4) is a colorless solid, which appears white in microcrystalline form. It is often seen in the plates/electrodes of car batteries, as it is formed when the battery is discharged (when the battery is recharged, then the lead sulfate is transformed back to metallic lead and sulfuric acid on the negative terminal or lead dioxide and sulfuric acid on the positive terminal). Lead sulfate is toxic by inhalation, ingestion and skin contact. It is a cumulative poison, and repeated exposure may lead to anemia, kidney damage, eyesight damage or damage to the central nervous system (especially in children). It is also corrosive - contact with the eyes can lead to severe irritation or burns [5, 6].

The solution phase voltammetry employed in the study of metal ion interaction with a selected ligand. Voltammetry behaviors of lead (II) ion in the presence of ligand with N-heterocycle compounds, such as nicotinic acid (pyridine-3-carboxylic acid) were being studied using cyclic voltammetry (CV) on a hanging dropping mercury electrode (HDME) [7].

Anodic stripping voltammetric studied to determination of Pb^{2+} or any metal ion. Cyclic voltammetric investigations of Pb^{2+} in 0.30 M acetic acid were characterised by a cathodic reduction peak on the initial negative scan resulting from the reduction of Pb^{2+} ions to Pb^0 [8].

Nitrogen compounds like urea and melamine are known to be commonly used for milk adulteration resulting in undesired intoxication; a well-known example is the Chinese episode occurred in 2008. The development of a rapid, reliable and economic test is of relevance in order to improve adulterated milk identification. Cyclic voltammetry studies using an Au working electrode were performed on adulterated and non-adulterated milk samples from different independent manufacturers [9].

The reduction of Pb (II) has been investigated in presence of different sodium salts as supporting electrolytes at a Hanging Mercury Drop Electrode (HMDE) using cyclic voltammetry. The correlation of cathodic peak current and anodic peak current, and the difference in cathodic peak potentials and anodic peak potentials with varying voltage scan rates indicates that the reduction of Pb (II) is reversible [10].

Voltammetric procedures for trace metals analysis in polluted natural waters using homemade bare gold disk microelectrodes. In filtered seawater samples, square wave

anodic stripping voltammetry (SWASV) is applied for analysis, whereas in unfiltered contaminated river samples, differential pulse anodic stripping voltammetry (DPASV) gave more reliable results. The peak potentials of the determined trace metals are shifted to more positive values compared to mercury drop or mercury-coated electrodes, with Zn always displaying 2 peaks, and Pb and Cd inverting their positions. These microelectrodes have been validated for natural sample analysis by use in an on-site system to monitor Cu, Pb and Zn, polluted by industrial activities. First results obtained on sediment core issued from the same location have shown the ability of this type of microelectrode for in situ measurements of Pb and Mn concentrations in anoxic sediments [11].

The reaction of alpha beta gamma delta-tetra(p-sulfophenyl)porphyrin (TPPS4) with Pb(II), Cd(II) or Cu(II) has been studied, and the spectra of the Pb(II)-TPPS4, Cd(II)-TPPS4 and Cu(II)-TPPS4 show the spectral absorption of these complexes with high sensitivity. The proposed analytical method has the advantage of high sensitivity, simplicity and high efficiency of interference-resisting [12].

The study serves to focus attention on the modification of multiwalled carbon nanotube with 2-(5-bromo-2-pyridylazo)-5-diethylaminophenol-Br-PADAP and its application for the development of a new, simple, and selective modified electrode in order to determine Pb(II) in standard alloys and water samples. The electrochemical method was based on open circuit accumulation of lead ions onto a 5-Br-PADAP-modified multiwalled carbon nanotube electrode and then their anodic stripping voltammetric determination. [13].

A new surface ion-imprinted Multi-walled carbon nanotubes (MCNTs), Pb(II) complex as functional monomer and template ion was presented for extracting and enrichment traces of Pb(II) ion. Parameters affecting the recovery of Pb(II) have been investigated [14].

In this work, Pb(II) sulfate compound was studied by electrochemical analysis using cyclic voltammetric technique to finding the redox current peaks properties of Pb²⁺ ion in normal saline as an electrolyte in present with each of AA,

glucose, uric acid and urea.

2. Experimental

2.1. Reagents and Chemicals

Lead sulphate PbSO₄ from Fluka (Germany), Ascorbic acid (AA) from Technicon chemicals Co. (Oreg. Tournai Belgique), KCl from SCRC (China) and other chemicals and solvents were of annular grade and used as received from the manufacturer. Double distilled water was used for the preparation of aqueous solutions. All solutions were deaerated with oxygen free nitrogen gas for 10-15 minutes prior to making the measurement.

2.2. Apparatus and Procedures

Instruments: EZstat series (potentiostat/glvnostat) NuVant Systems Inc. pioneering electrochemical technologies USA. Electrochemical workstations of Bioanalytical system with potetiostate driven by electroanalytical measuring softwares was connected to personal computer to perform Cyclic Voltammetry (CV), an Ag/AgCl (3M NaCl) and Platinum wire (1 mm diameter) was used as a reference and counter electrode respectively. The glassy carbon working electrode (GCE) was used in this study and cleaning with alumina grand.

3. Results and Discussion

3.1. Cyclic Voltammetry of Glassy Carbon Electrode in Normal Saline

There is good potential area of glassy carbon electrode in normal saline (0.9% NaCl), which can used this electrolyte to study the heavy metal as a pollutant in environment such as Pb(II). It was found that the potential area from -2mV to 1.5mV is good rang to study the electrochemical properties of heavy metals in normal saline using GCE as shown in Figure 1.

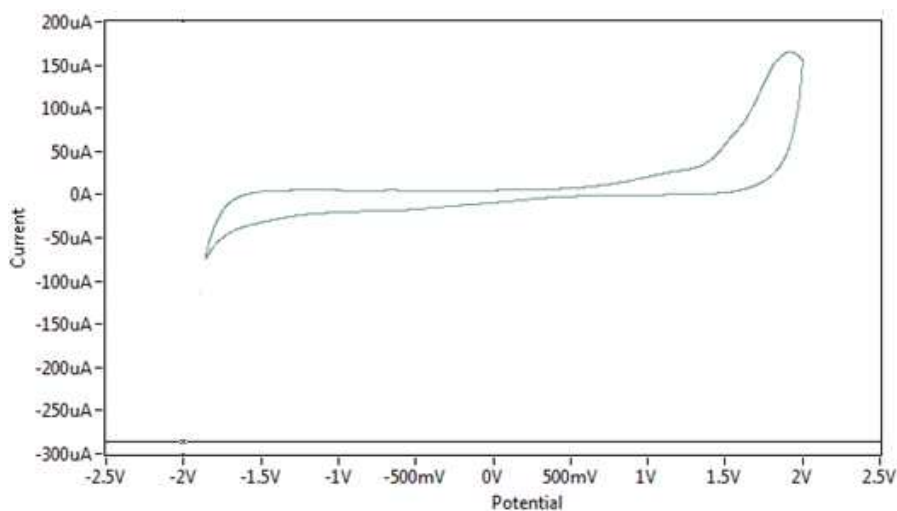


Figure 1. Cyclic voltammogram of normal saline as electrolyte by GCE at scan rate 100mVsec⁻¹ versus Ag/AgCl as reference electrode.

3.2. Cyclic Voltammetry of (PbII) in Aqueous Solution

It was found that different aqueous electrolyte act different redox current peaks of the Pb(II) ions which enhancement the oxidation- reduction reactions in these electrolytes as in the following different electrolytes.

3.2.1. In Normal Saline

Figure 2 illustrated the oxidation and reduction current peaks of Pb(II) in normal saline at -550mV and -650mV respectively, using GCE by cyclic voltammetric technique. It was chosen normal saline as an electrolyte to studying the electrochemical properties of one of important pollutants in environment which is lead compound and comparison with other electrolytes.

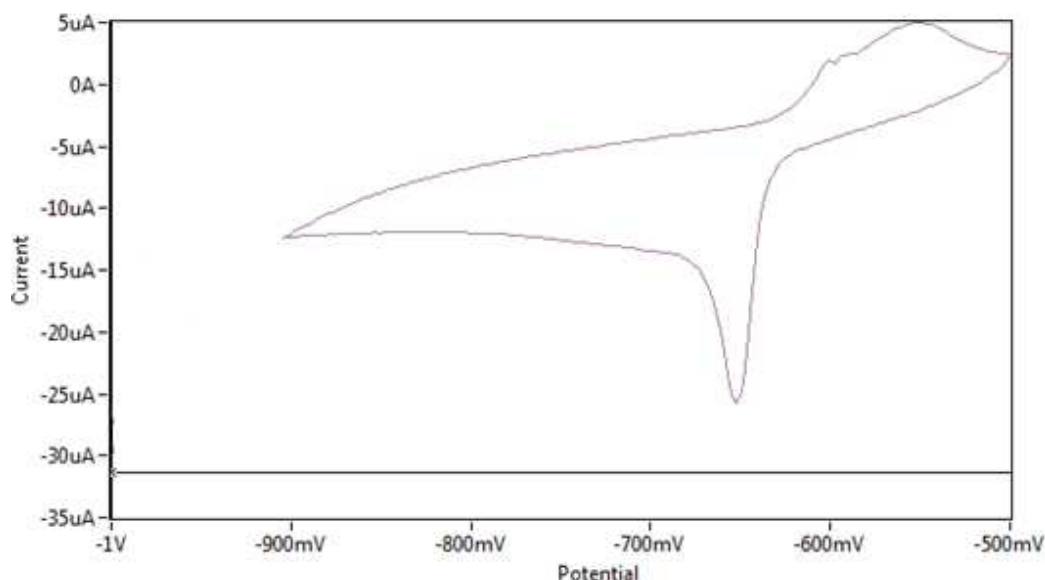


Figure 2. Cyclic voltammogram of 10mM Pb(II) in normal saline as an electrolyte by GCE at scan rate 100mVsec^{-1} versus Ag/AgCl as reference electrode.

3.2.2. In KCl Solution

KCl solution the other electrolyte was used to studying the redox current peaks of lead ions. It was found a weak sensitivity of the redox current peaks of lead ions in KCl comparison with normal saline as shown in Figure 3.

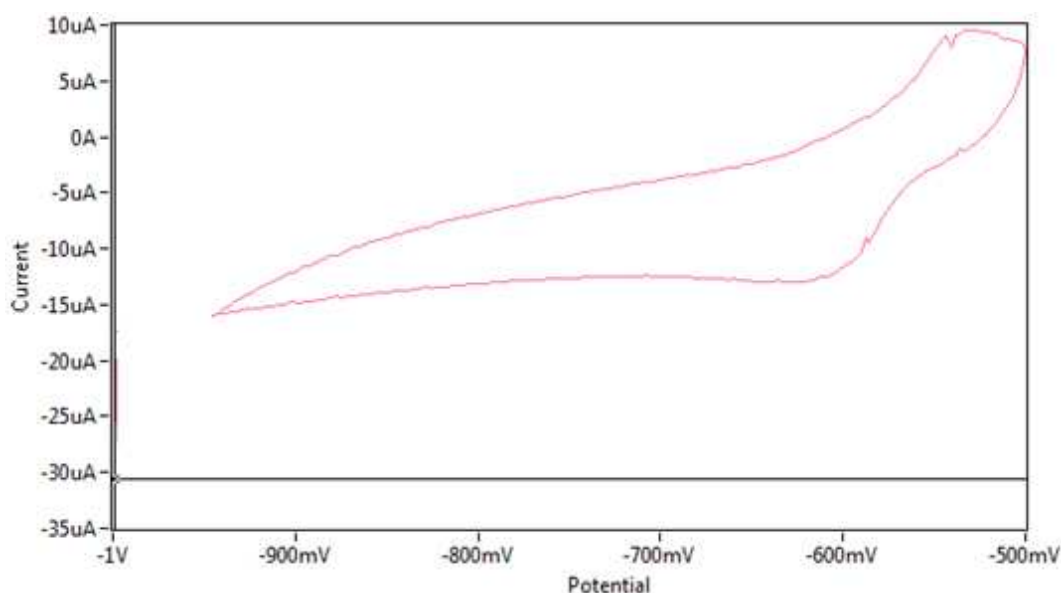


Figure 3. Cyclic voltammogram of 10mM Pb(II) in 1M KCl using GCE at 100mVsec^{-1} versus Ag/AgCl as reference electrode.

3.2.3. In Blood Medium

The redox current peaks of Pb(II) was studied in blood medium as an electrolyte to finding the effect of blood medium on the redox peaks. Figure 4 illustrated that lead ions act an oxidative reagent in blood medium with enhancement the oxidation

current peak of Pb(II) and disappearing the reduction current peak. So, it was used normal saline as a good electrolyte to explanation the redox current peaks of lead ions in this study.

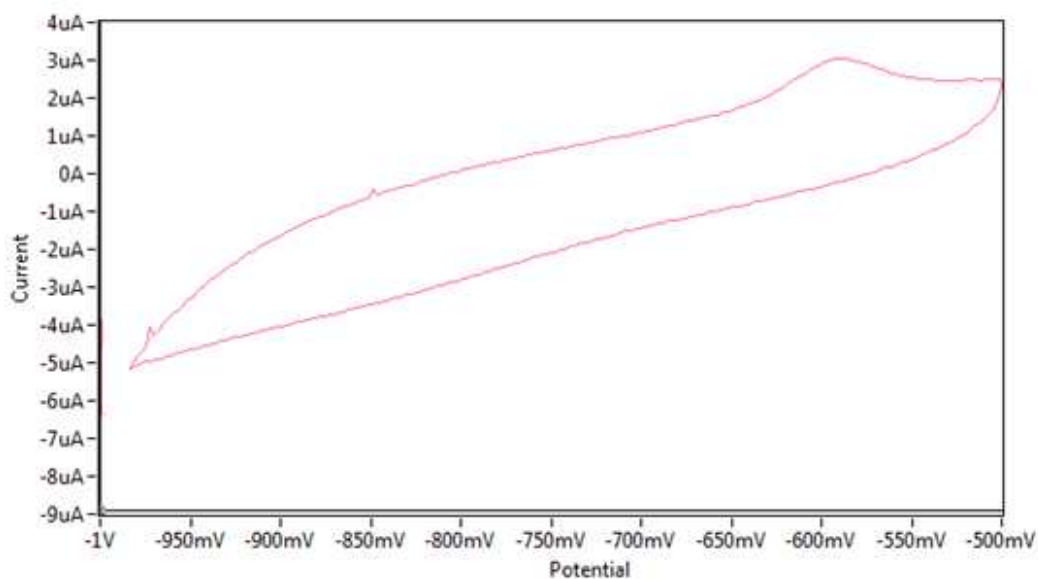


Figure 4. Cyclic voltammogram of 10mM Pb(II) in blood medium using GCE at 100mVsec^{-1} versus Ag/AgCl.

3.3. Enhancement Study of Redox Pb(II) in Normal Saline

It was chosen normal saline as a good electrolyte in this study for the enhancement of the both oxidation and reduction current peaks of lead ions. The normal saline was used as electrocatalyst in analytical study of the lead ions for enhancement of the redox current peaks as Pb(II) = Pb(IV).

3.3.1. Effect AA on the Redox of Pb(II)

The lead ions in normal saline act as an oxidative reagent with appearing of oxidation current peak at -550mV of the voltammogram as shown in Figure 2. It was found that the oxidation and reduction current peaks of lead ions in present of AA degreasing for appearance in normal saline. So, AA less affected on the oxidation and reduction reactions of lead ions in normal saline as shown in Figure 5.

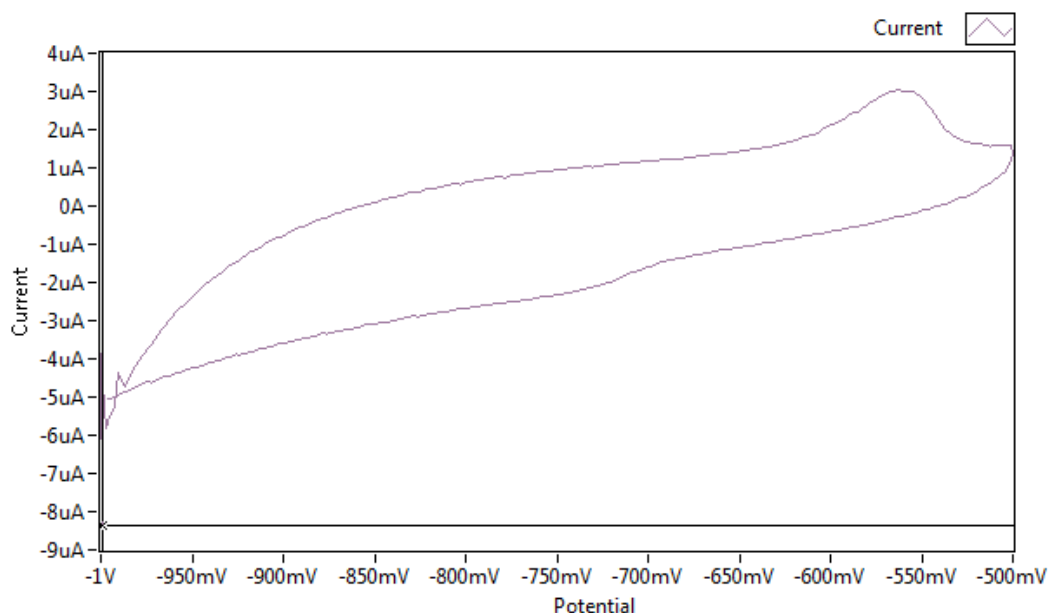


Figure 5. Cyclic voltammogram of 10mM Pb(II) in normal saline as an electrolyte in present of 10mM AA by GCE at scan rate 100mVsec^{-1} versus Ag/AgCl as reference electrode.

3.3.2. Effect Glucose on the Redox of Pb(II)

The electrochemical studied of redox current peaks of lead

ions in normal saline present with glucose solution which causes enhancement the oxidation current peak of Pb(II) as

shown in Figure 6. Gradually, the addition of glucose solution at different concentration to the lead ions in the voltammetric cell using normal saline as a supporting electrolyte causes enhanced the oxidation current peak of Pb(II) as shown in Figure 7, the relationship between cathodic current against the different concentration of glucose solution with the equation of $y=0.5714X+4.667$ with good sensitivity of $R^2=0.979$. it means that glucose act as

electrocatalyst for enhanced the oxidation current peak as oxidative effect of lead ions in normal saline, but the reduction current peak of Pb(II) appears no affected from the relationship between anodic current against the different concentration of glucose as shown in figure 8 the linear line illustrated as a horizontal straight line with equation $y=0.2432X+32.176$ and $R^2=0.2736$.

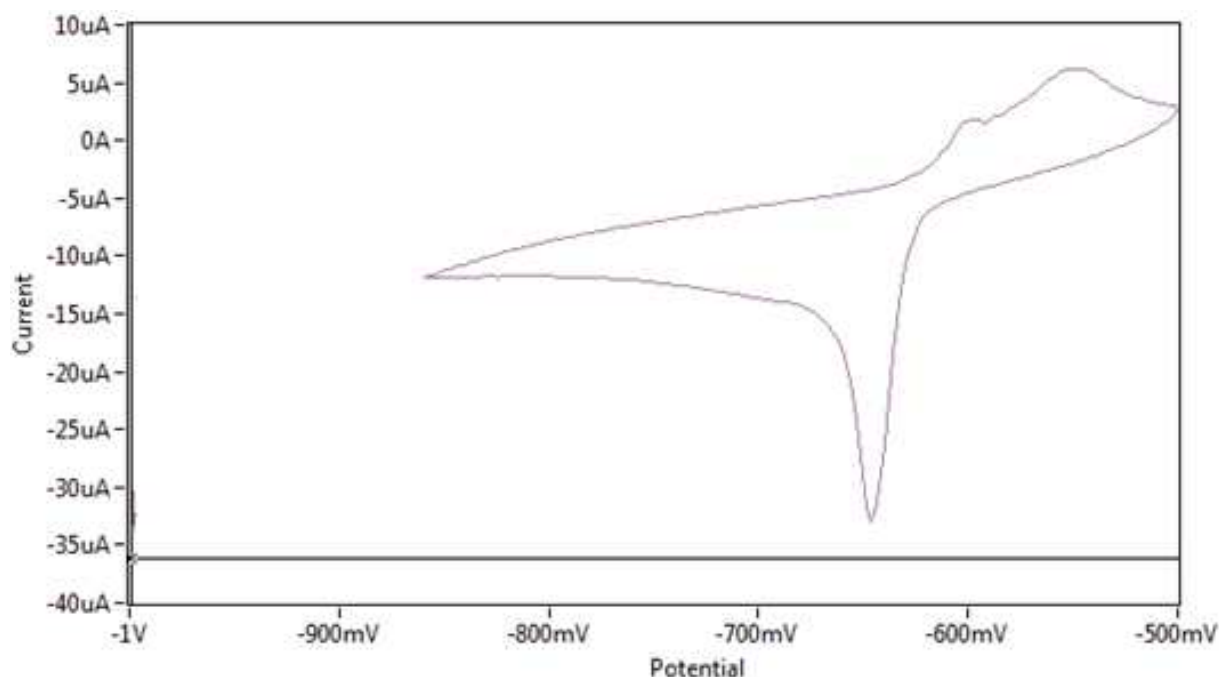


Figure 6. Cyclic voltammogram of 10mM Pb(II) in normal saline as an electrolyte in present of 10mM glucose by GCE at scan rate 100mVsec^{-1} versus Ag/AgCl as reference electrode.

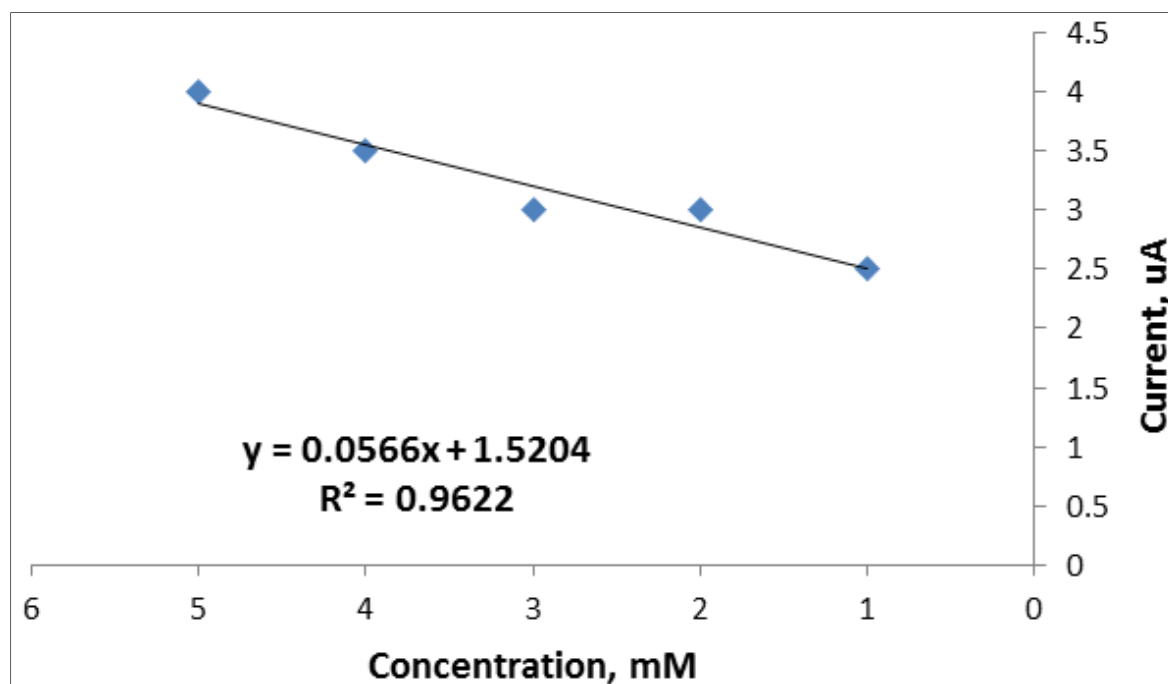


Figure 7. Plotting of anodic current peak of Pb(II) in different concentrations of glucose using normal saline as supporting electrolyte.

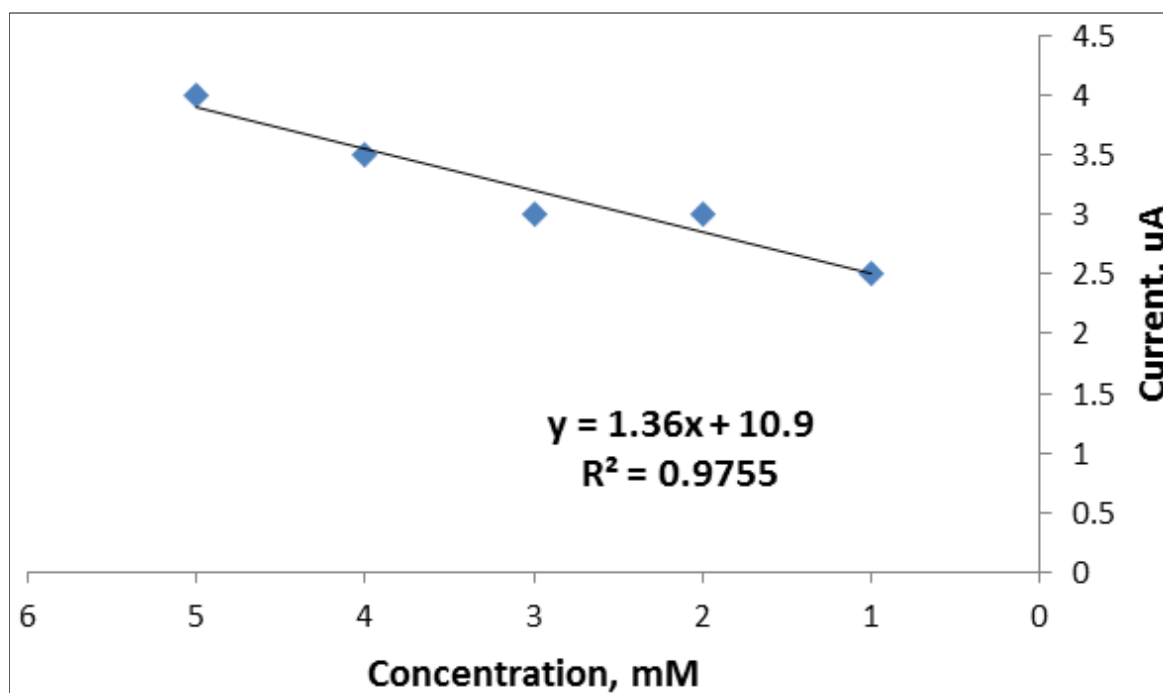


Figure 8. Plotting of cathodic current peak of Pb(II) in different concentrations of glucose using normal saline as supporting electrolyte.

3.3.3. Effect Uric Acid on the Redox of Pb(II)

Uric acid as an oxidative reagent was used to studying the effect of redox current peaks of lead ions in normal saline as a supporting electrolyte. Figure 9 shows the oxidation reduction current peaks of Pb(II)=Pb(IV) in normal saline. When it was used uric acid as oxidative reagent act to enhancement the both of redox peaks of the lead ions as shown in figure 10, the relationship of anodic current peak

against the different concentration of uric acid in normal saline with equation of $y=0.0566X+1.5204$ and good sensitivity $R^2=0.9622$. Also, the effect of uric acid on the cathodic current peak of Pb(II) in normal saline was gradually enhanced as shown in figure 11 from the relationship of the reduction current against the different concentration of uric acid with equation $y=1.36X+10.9$ and good sensitivity $R^2=0.9755$.

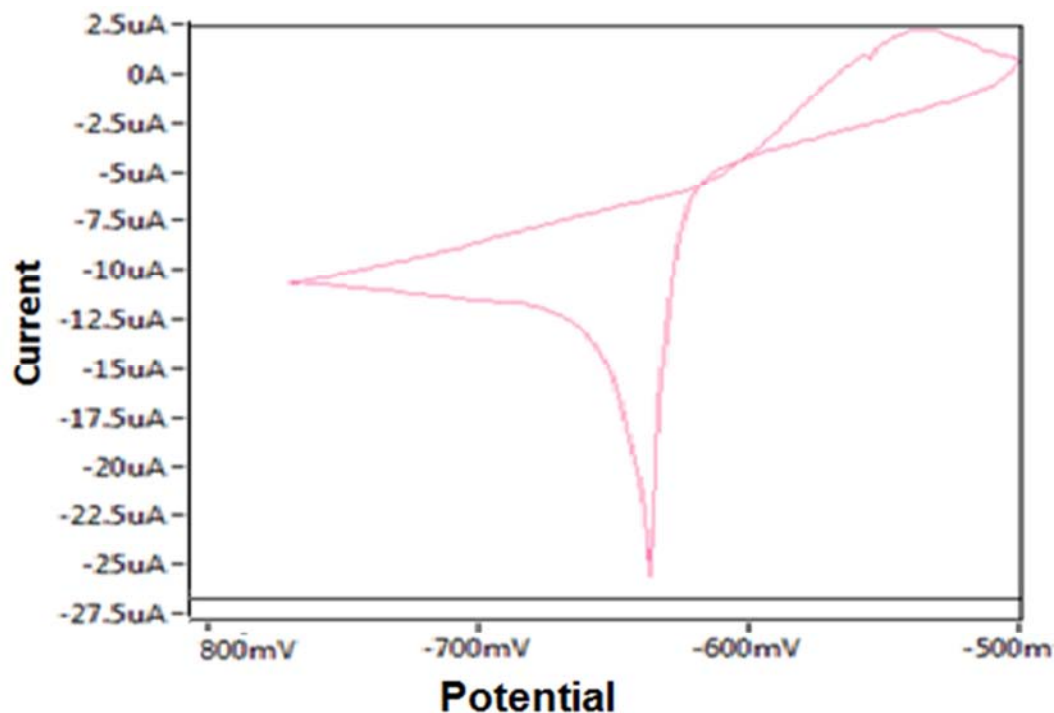


Figure 9. Cyclic voltammogram of 10mM Pb(II) in normal saline as an electrolyte in present of 10mM uric acid by GCE at scan rate 100mVsec^{-1} versus Ag/AgCl as reference electrode.

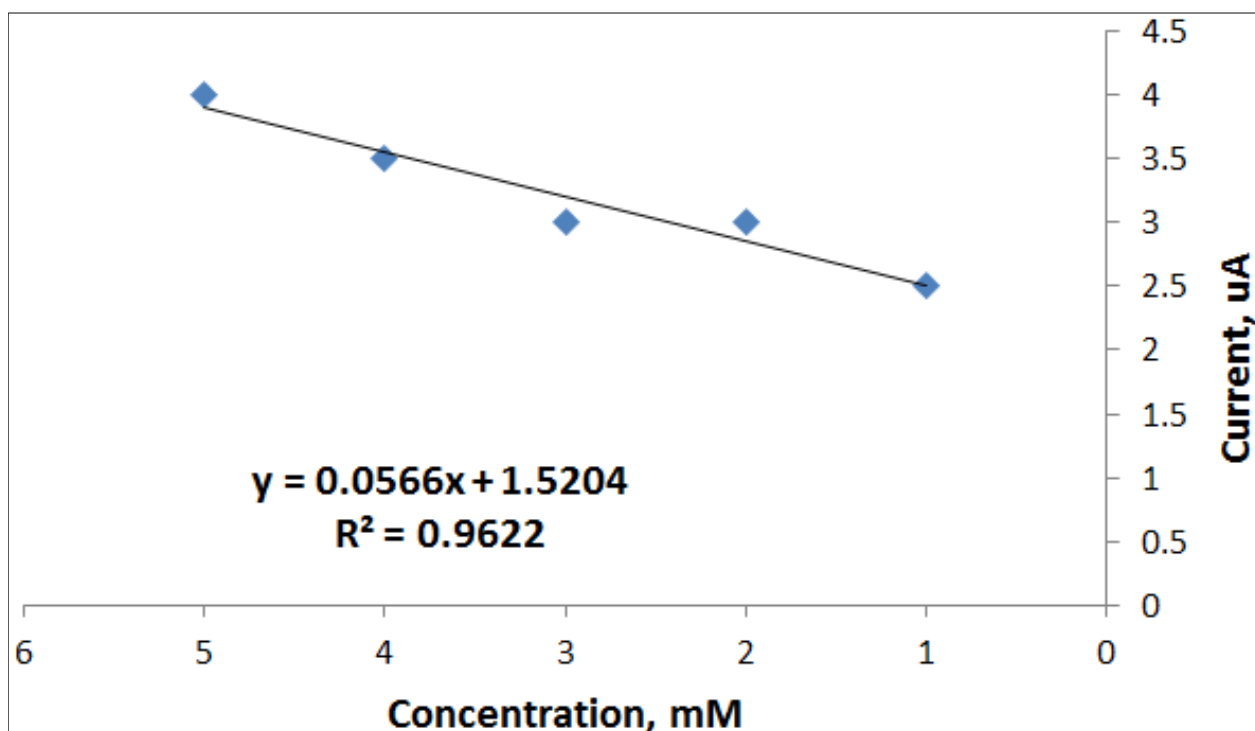


Figure 10. Plotting of anodic current peak of Pb(II) in different concentrations of uric acid using normal saline as supporting electrolyte.

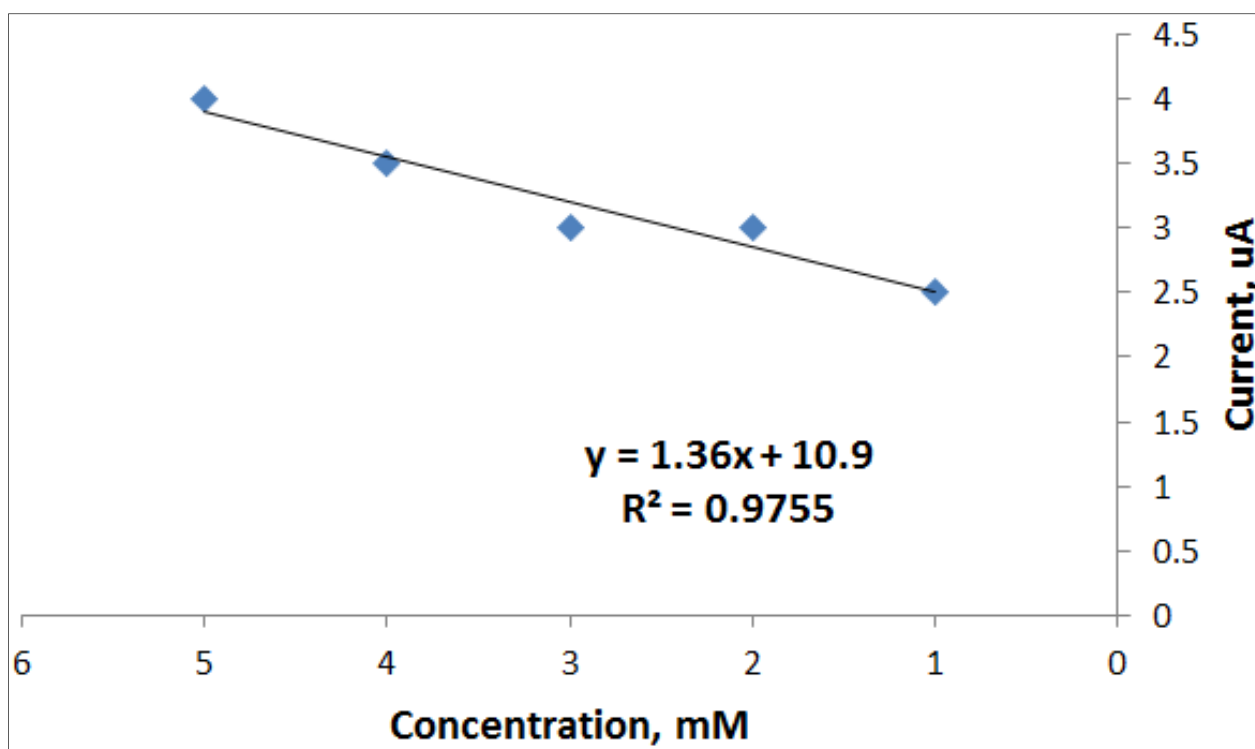


Figure 11. Plotting of cathodic current peak of Pb(II) in different concentrations of uric acid using normal saline as supporting electrolyte.

3.3.4. Effect Urea on the Redox of Pb(II)

Urea is the other oxidative reagent was used to study the effective of enhancement the anodic current peak of lead ions in normal saline as a supporting electrolyte as shown in figure 12. The relationship between the anodic current peak against the different concentration of urea as shown in figure 13, the linear relationship with equation of $y=0.35X+2.15$ and good sensitivity $R^2=0.9423$.

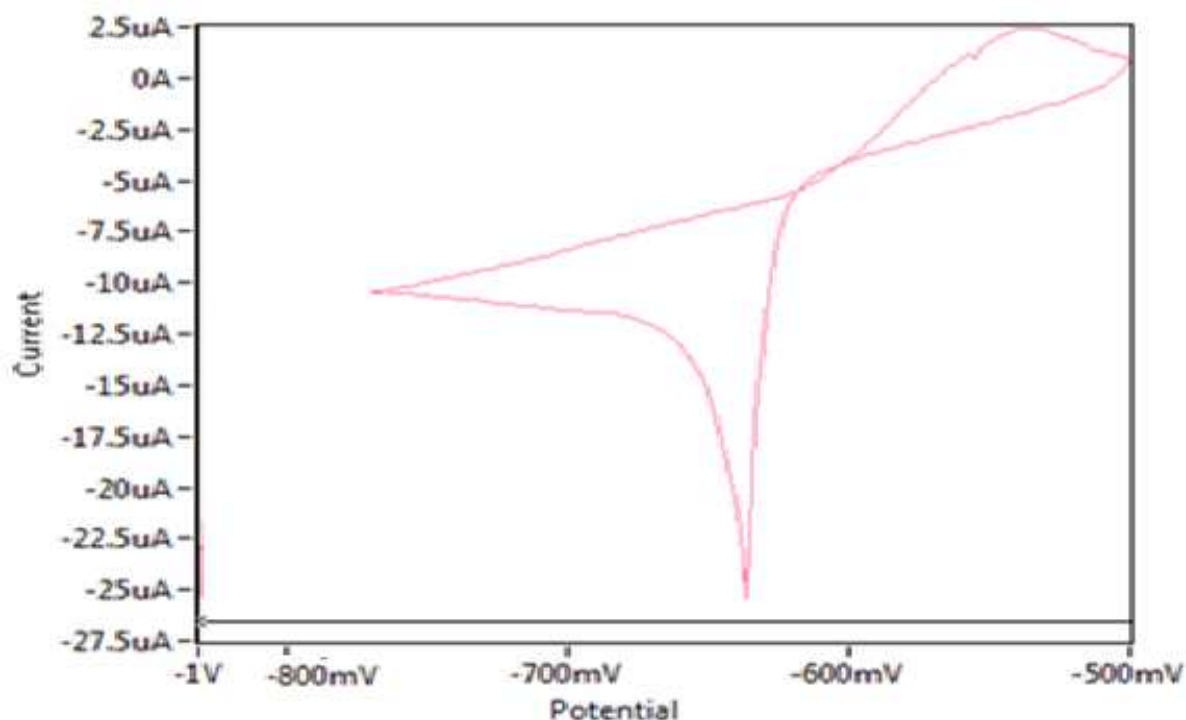


Figure 12. Cyclic voltammogram of 10mM Pb(II) in normal saline as an electrolyte in the presence of 10mM urea by GCE at scan rate 100mVsec⁻¹ versus Ag/AgCl as reference electrode.

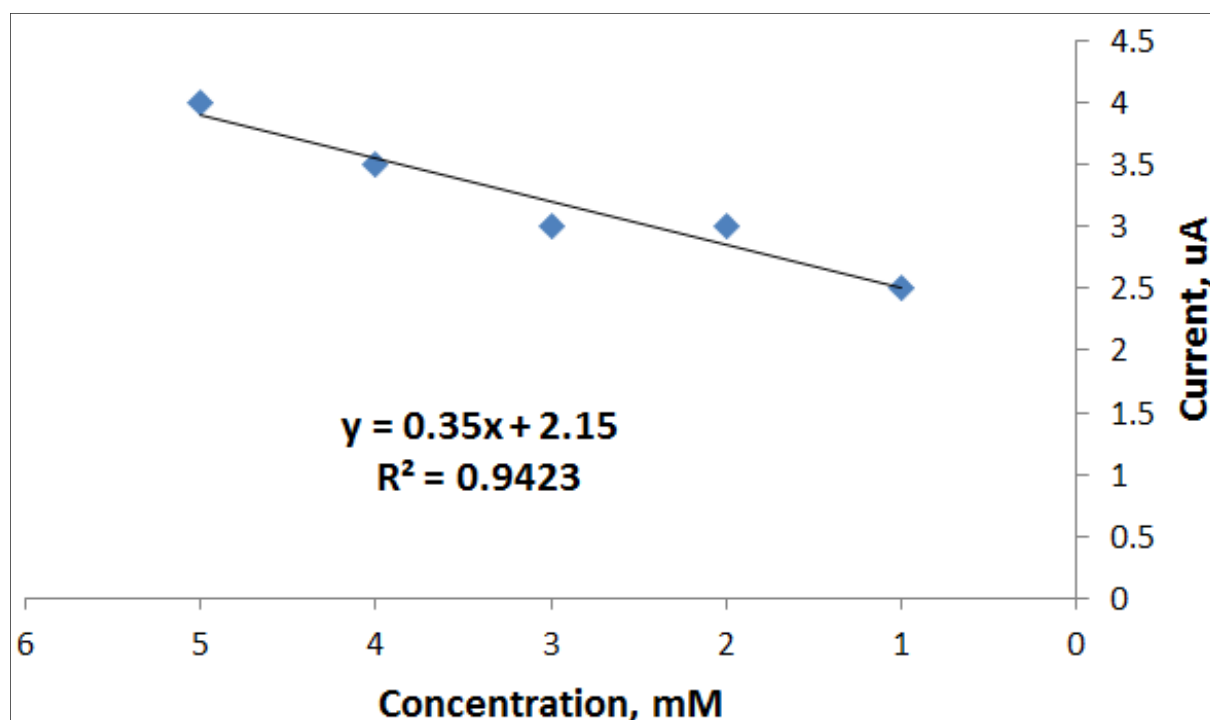


Figure 13. Plotting of anodic current peak of Pb(II) in different concentrations of urea using normal saline as supporting electrolyte.

4. Conclusion

It was chosen normal saline as a supporting electrolyte to studying the effect of different oxidative reagents (such as glucose, urea and uric acid) and antioxidative reagent (such as ascorbic acid) on the lead ions by cyclic voltammetric

technique. It was found that no affected of AA on the anodic and cathodic current peaks of the lead ions in normal saline. But, it was found for other oxidative reagents (glucose, urea and uric acid) that redox current peaks of lead ions were affected with these reagents by enhancement of the both redox current peaks. The electrochemical study of lead ions in normal saline as a supporting electrolyte was aimed to

finding the oxidation reduction process of lead compound introductive to studying in blood medium.

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