AASCIT Communications
Volume 3, Issue 2
February 15, 2016 online
ISSN: 2375-3803

American Association for Science and Technology

Cyclic Voltammetry of Lead Nitrate with Acetyl Acetone Using Glassy Carbon Electrode

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Keywords

Cyclic Voltammetry, Lead Nitrate, Glassy Carbon Electrode, Acetyl Acetone

The redox behavior of lead nitrate was studied voltammetrically in presence and absence of acetyl acetone using both platinum and new made glassy carbon electrodes in 0.1 M KCl supporting electrode. Composition of the oxidation and reduction processes well done also with new graphene electrode from the three discussed electrodes, glassy carbon electrode gave better separated waves for both reduction and oxidation processes. All mechanisms were discussed. Comparison with other electrodes also done in literature like carbon paste electrode and hand drawn pencil electrode, our new prepared glassy carbon electrode gave better results.

Introduction

Using nano new electrode which depend on particle size [1-2] is needed. Nanomaterials have received increasing attention in various fields of science and technology [3-7].

Lead nitrate is attractive material which used in many industrial applications, including lead acid batteries [11-13], and oxidation of organic compounds [14-17].

In this work we tried to discuss the cyclic voltammetry for lead ions in the medium used.

Experimental

Lead nitrate, acetyl acetone and KCl from Merck were used without purification. In all experiments double – distilled water (conductivity equal 3 micro Simens) value is used.

Instrument DY2000, DY2000EN Multichannel Potentiostat was used for voltammetry measurements.

New glassy carbon electrode was prepared by polishing pure carbon cylinder with wool peace and Al_2O_3 powder, connected with copper wire and lastly isolated by heat shrank polymer. Graphene electrode was prepared by evaporation of small amount of graphene oxide put in carbon paste electrode and evaporates it under I.R. lamp.

Voltammetry analyzer using conventional three - electrode electrochemical cell to perform cyclic voltammetry (CV). Measurements were done by using graphene, and glassy carbon working electrodes. Platinum wire electrode as counter electrode and Ag/AgCl standard electrode were used.

Results and Discussion

The electrochemical behaviors for lead were studied voltammetrically [1-8]. The prepared electrodes were examined in a potential range from -2 to +2 V vs. Ag/AgCl electrode. Figures (1-6) showed the cyclic voltamogram for divalent lead ions in using 0.1 M KCl as a supporting electrode as done in literatures [3-9]. The scan rate for all measurements was fixed at 0.05 V/sec.

Normally lead divalent ions showed one quasi reversible reaction, one oxidation peak was appeared at -0.43 V in the forward scan and another reduction peak was appeared at -0.64 V at the reverse scan with a 190 mV separation peak potential (ΔE_P) was obtained in literature [1-6].

In our study here the reduction scan was in the forward direction and oxidation scan is in the reverse direction. Fig (1) shows two reduction peaks at -0.27 V and 0.0 V , whereas in the oxidation scan two waves were obtained at-0.33V and -0.53 V for the redox behavior of 1 mM Pb(II) in 0.1 m KCl vs Ag/AgCl electrode..

The reduction peaks (E_{PC}) appears at 0.0 V and -0.18 V in Fig. (1) versus Ag/AgClhas $I_{PC} = 2.7 \times 10^{-4}$ A using glassy carbon electrode are corresponds to two reduction steps of lead ions, each one consumes one electron.

The reduction reaction of Pb^{+2} is $Pb^{+2}(aq) + 2e^- \text{------}\rightarrow Pb(s)$

Lead ion in the solutions takes two electrons and becomes Pb.

The oxidation reaction of Pb is $Pb(s) \text{------}\rightarrow Pb^{+2}(aq) + 2e^-$

Occurs lead atom loses electron to become Pb^{+2} (aq) back to the oxidation.

The reduction peak is at -0.72 V for lead ion (lead nitrate) in presence of 0.1M KCl supporting electrode alone at the platinum electrode (Fig.6) which separated into two clear waves using glassy carbon electrode at 0 V and -0.18 V Which due to the reduction of divalent ion to monovalent ion and the second for the reduction of monovalent lead to lead metal.

Adding acetyl acetone shifts the reduction and oxidation peaks favouring more complexation using glassy carbon electrode but on using graphene electrode not clear diagrams were obtained indicating strain in the electronic device.

The three electrode cell system was used.

The peaks separation ΔE_P defined as $\Delta E_P = E_P - E_{PC}$ was 32.

Which close to the theoretical value 28.5mV for reversible transfer, due to the internal resistance of the electrode, the shape is slightly distorted from rectangular and close to parallegram indicating the main contribution to capacitance is the change and discharge of the double layer.

Knowing ΔE_P the Gibbs free energies for reduction and oxidation can be evaluated and all the different thermodynamic parameters can be easily estimated [10-21].

Bare carbon paste electrode was used as new electrode for cyclic voltammetry of lead ion in literature which showed one quasi reversible reaction one, oxidation peak was appeared at -0.43 V and another reduction peak was appeared at -0.64 V [5]. Other scientists used glassy carbon of radiometer analytical type with about same results [22]. Cyclic voltammetry for lead ions were studied at hand drawn pencil electrode also which gave better results than the others and uses for determination by stripping voltammetry [23].

Quasi reversible / one oxidation

Acetyl acetone showed significant voltammetry changes in peaks in both peaks current and potential. This is due to the electrostatic interaction between acetyl acetone and Pb^{+2} ions leading to electrostatic interaction [22-32]. The enhancement in peak currents using acetyl acetone is due to increscent of Pb(II) in the electrode surface by the complex formation between acetyls acetone and lead nitrate.

The behaviour was studied using different concentrations of lead nitrate solutions at 292.15K were done by using different electrode techniques were done and presented in Figs. (1-6).

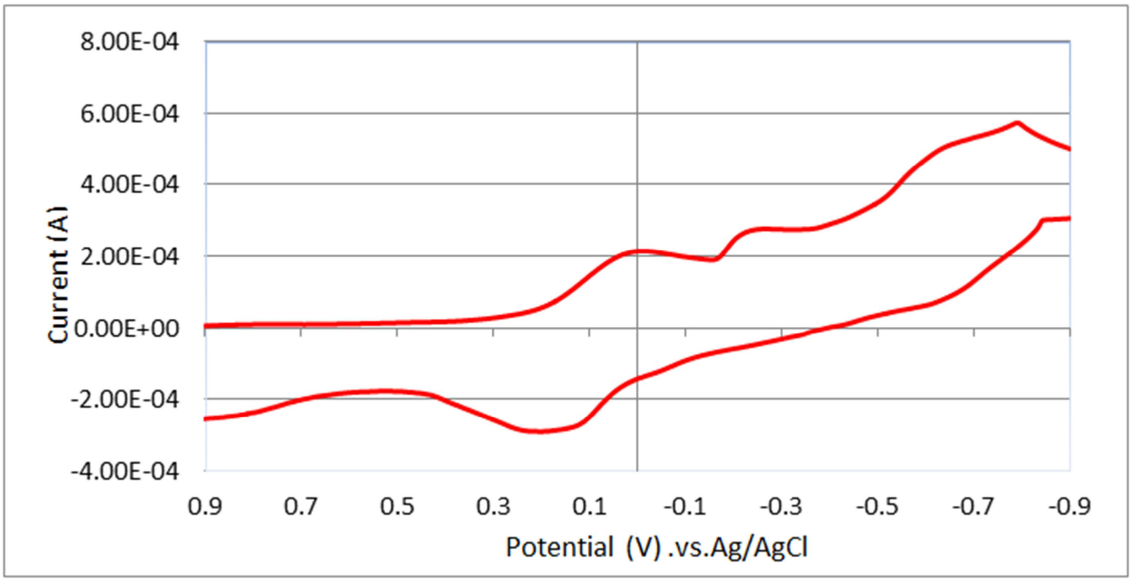

Fig. (1). *Cyclic voltamogram relation E in Amp. and V. vs. Ag/AgClfor1mM* $Pb(NO_3)_2$ *in 0.1 M KCl using glassy carbon electrode.*

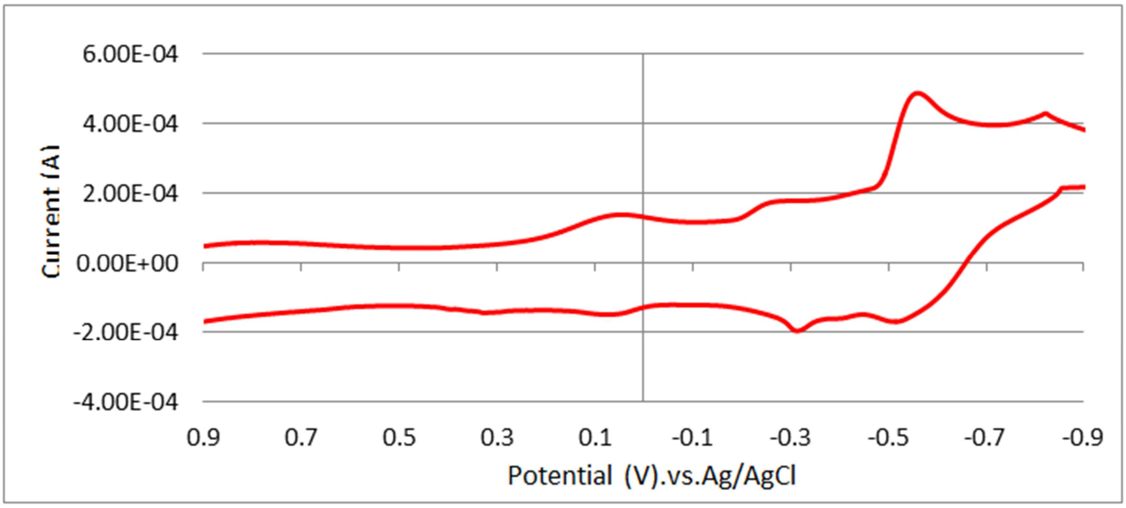

Fig. (2). *Cyclic voltamogram, relation E in Amp. and V.vs. Ag/AgClfor2mM* $Pb(NO_3)_2$ + $1x10^{-5}$ *M acetyl acetone in 0.1 M KCl using glassy carbon electrode.*

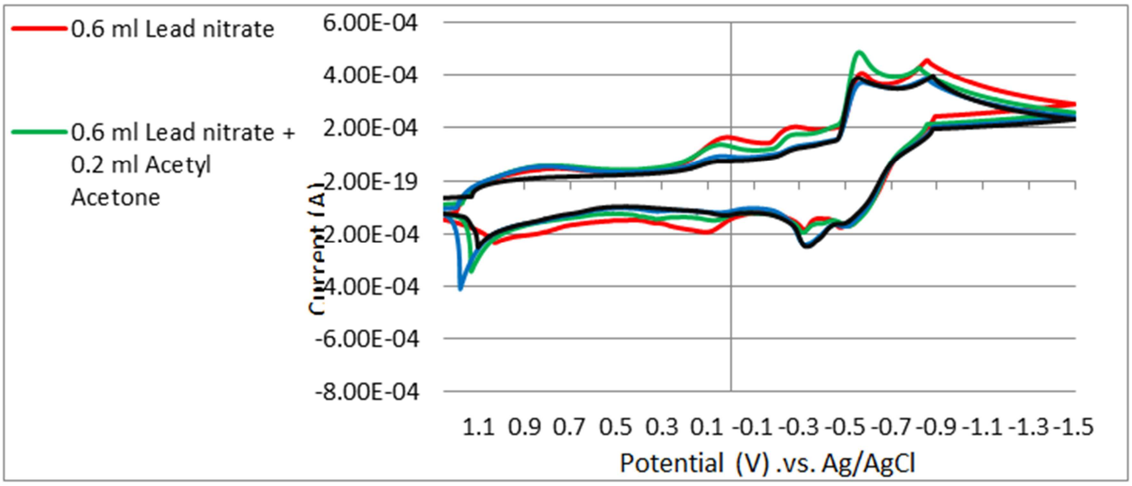

Fig. (3). *Comparison between adding (0.6 ml 0.1M)* $Pb(NO_3)_2$*, 2 mM , (0.6 ml* $Pb(NO_3)_2$ + $1.007x10^{-5}$ *M acetyl acetone (0.2 ml), (0.6 ml* $Pb(NO_3)_2$ + $1.307x10^{-5}$ *M acetyl acetone (0.4 ml)and (0.6ml* $Pb(NO_3)_2$ $1.314x10^{-5}$ *M acetyl acetone (0.6 ml) in 0.1 M KCl using glassy carbon electrode.*

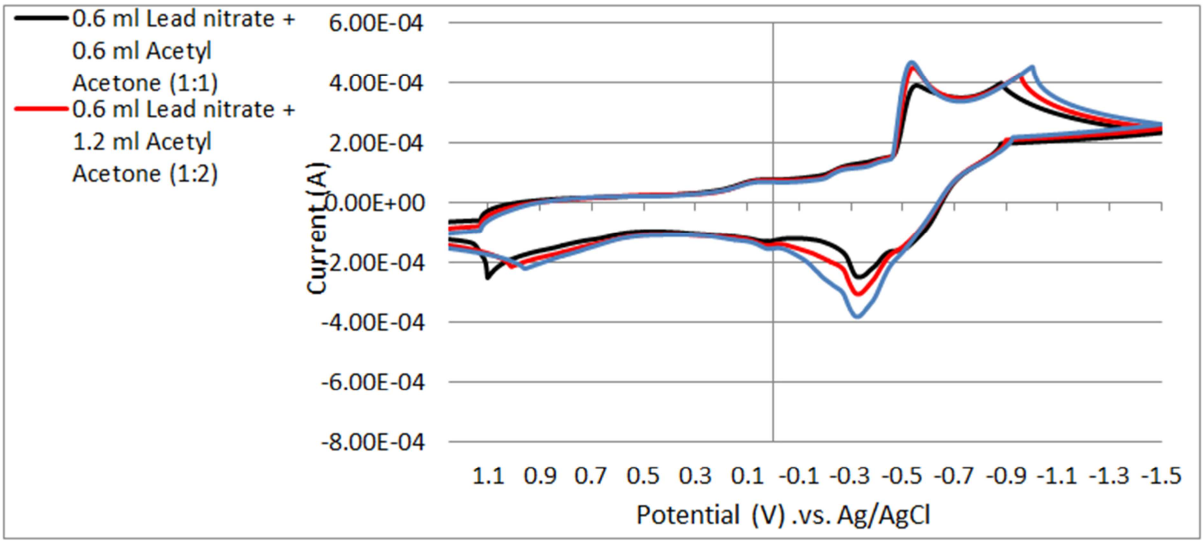

Fig. (4). *Comparison between adding (1:1), (1:2) and (1:3) (M/L) ($Pb(NO_3)_2$: acetyl acetone) in 0.1M KCl using glassy carbon electrode.*

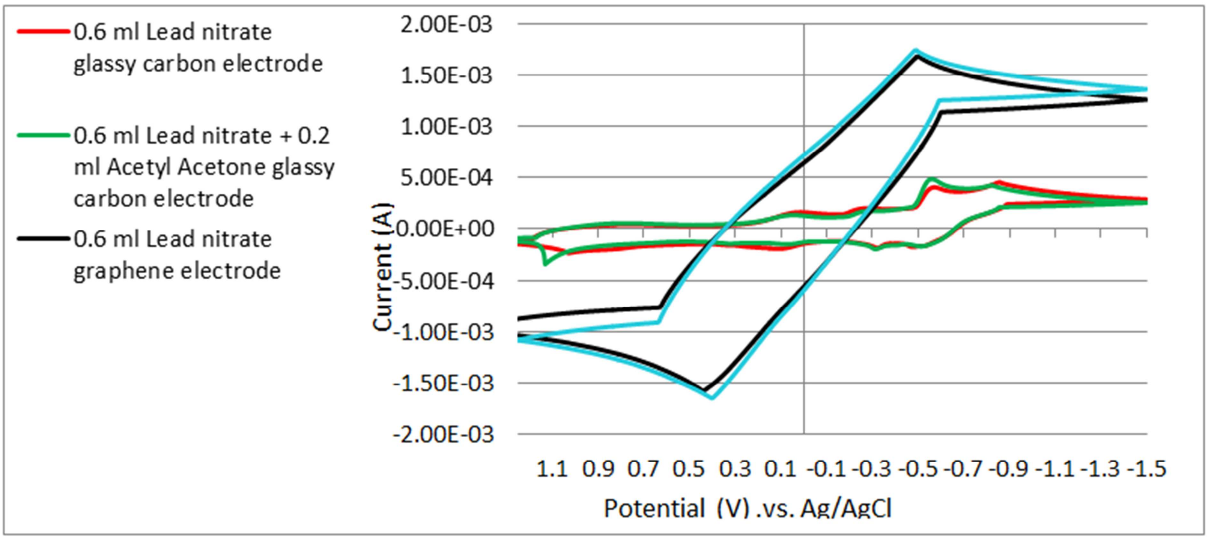

Fig. (5). *Comparison between glassy carbon electrode, graphene electrode versus silver-silver chloride electrode (0.6 ml $Pb(NO_3)_2$ + 0.2 ml (1mM) acetyl acetoneand 1.314x10-4 M acetyl acetone.*

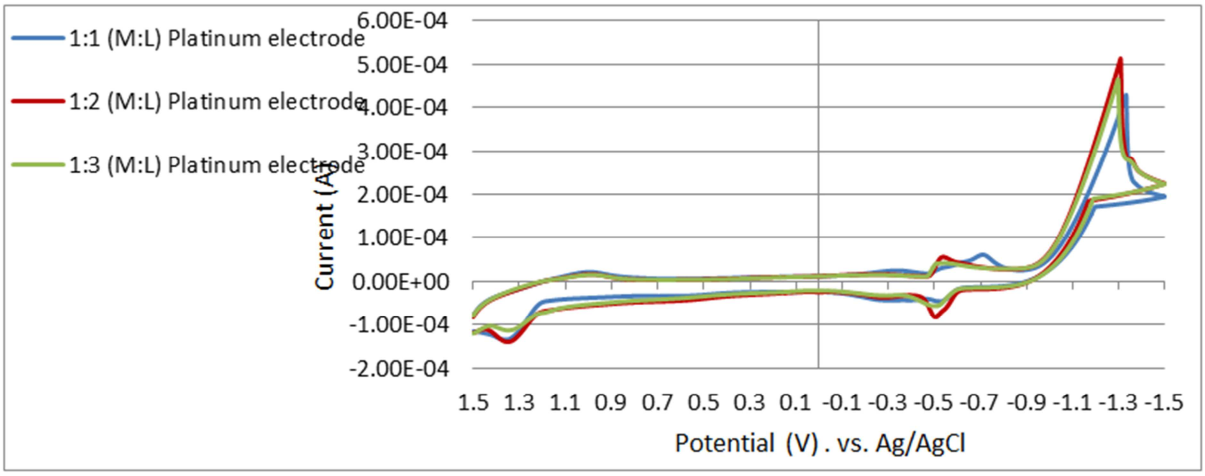

Fig. (6). *Voltamograms of different ratios, (1:1), (1:2) and (1:3) ($Pb(NO_3)_2$: acetyl acetone) in 0.1M KCl in platinum electrode versus silver-silver chloride electrode.*

Conclusion

This work is concentrated on the use of different electrodes for cyclic voltammetry of lead ions in solution. New simple glassy carbon electrode was prepared and used for studying the redox behaviors of lead ions. Glassy carbon electrode show bitter clear waves which is supported by the addition of acetyl acetone. From many working electrodes used for measuring cyclic voltammetry of lead ion like carbon paste electrode, readymade carbon electrode, hand drawn pencil electrode, platinum disk electrode , graphene electrode and the here prepared electrode from pure carbon peace, the last one gave better defined waves. This new electrode can also used for following the complex behaviors in solutions. ■

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