

Cyclic Voltammetric (CV) Evaluation of Electro chemical Behaviors of $K_3 [Fe (CN)_6]$ and Uric Acid

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ABSTRACT

Cyclic Voltammetry has a multipurpose electroanalytical technique for the study of electroactive species, the method displays redox behaviour of chemical species inside a wide range potential. The current at the working electrode is observed as a three-cornered excitation potential is applied to the electrode. The purpose of this paper was to determine the diffusion coefficient (D) of the $K_3[Fe(CN)_6]$ and uric acid; electrochemical nature of $K_3[Fe(CN)_6]$ and uric acid, effects of varying the concentrations analytes on peak currents and peak splitting and scan rate CV on peak currents and peak splitting. All the reagents used for this experiment were analytical grade. $K_3[Fe(CN)_6]$ and uric acid solutions were prepared. The experiment has been implemented with the principles of cyclic voltammetry. The relationship between the scan rate and the peak current were investigated and the result shows that there is a direct relationship between scan rate and peak current that is $I_p \propto V^{1/2}$. As articulated on the above voltammograms, the peak current increases with the augmentation of the concentrations of $K_3[Fe(CN)_6]$, So that, the researcher conclude the manifestation of direct relationship between peak current and concentration of analytes. The experiment of this research shows that the increasing of diffusion coefficient (D) with increasing of concentrations /and scan rate. The peak currents and peak splitting fluctuate with variation of scan rate of CV. Similarly, the peak currents and peak splitting vacillate with variation of concentration of the analytes. CV therefore can be used as an indication of major analytical tool for the determination of the trace elements which are electro active in nature. The electrochemical nature of $K_3[Fe(CN)_6]$ and uric acid were determined; reversible and irreversible respectively.

Keywords: Cyclic Voltammetry; Ferricyanide; Uric Acid; Electrochemical Behaviour

INTRODUCTION

Cyclic voltammetry is a method used for investigating of the electrochemical behaviour of a system It was first described in 1938 and pronounced theoretically . Cyclic voltammetry is the most broadly used technique for obtaining qualitative information about electro chemical reactions. The power of cyclic voltammetry results from its ability to rapidly provide considerable information on the thermo dynamics of redox processes, on the kinetics of heterogeneous electron-transfer reactions and on coupled chemical reactions or adsorption processes. Cyclic voltammetry is frequently the first experimental approach performed in an electro analytical study, since it offers rapid location of redox potentials of the electro active species

and convenient evaluation of the effect of media upon the redox process. A cyclic voltammogram is obtained by applying a linear potential sweep (that is, a potential that increases or decreases linearly with time) to the working electrode. As the potential is swept back and forth past the formal potential, E^0 , of an analyte, current flows through the electrode that either oxidizes or reduces the analyte. The magnitude of this current is proportional to the concentration of the analyte in solution, which allows cyclic voltammetry to be used in an analytical determination of concentration [1].

CV has become a very popular technique for electrochemical studies of new systems, and has proved as a sensitive tool for obtaining information about fairly complicated electrode reactions. CV methods have found to have extensive

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applications for the evaluation of thermodynamic and kinetic parameters such as number of electrons change (n), heterogeneous rate constant (k_0), entropy (S), Gibb's free energy (G) and diffusion coefficient (D_0) etc., of a number of redox reactions and associated chemical reactions. These methods are especially useful in both oxidation and reduction process and to study the multiple electron transfer in an electrochemical reaction [2].

Potassium ferricyanide is a bright red salt with a chemical formula $K_3Fe(CN)_6$. The salt contains that the octahedral and coordinated $[Fe(CN)_6]^{3-}$ ion. It is soluble in acetonitrile-water media (1:1) and its solution shows some green yellow fluorescence. Like the other metal cyanides, solid potassium ferricyanide has a complicated polymeric structure. The polymer consists of octahedral $[Fe(CN)_6]^{3-}$ centres cross linked with K^+ ions that are bound to the CN ligands. The $K^+ \sim NCFe$ linkages break when the solid is dissolved in water-acetonitrile media. The $Fe(CN)_6^{3-} / Fe(CN)_6^{4-}$ redox couple is used as an example of an electrochemically reversible redox system used to study some basic concepts of Cyclic Voltammetry [$Fe(CN)_6]^{3-}$ consists of a Fe^{3+} centre bound in octahedral geometry to six cyanide ligands). The complex has Oh symmetry. The iron is low spin and easily reduced to the related ferrocyanide ion $[Fe(CN)_6]^{4-}$, which is a ferrous (Fe^{2+}) derivative. This redox couple is reversible and entails no making or breaking of Fe-C bonds: $[Fe(CN)_6]^{3-} + e^- \rightarrow [Fe(CN)_6]^{4-}$ [3].

For the reasons above, potassium ferricyanide redox is a standard in electrochemistry. This redox couple is also used in: preparation of Prussian Blue (the deep blue pigment in blue printing), as a chemical reducer in photographic processing to remove silver from negatives and positives (dot etching), in wine production potassium ferricyanide has been used to precipitate out copper if copper has been used as a fungicide on the grapes. In histology, it is used to detect ferrous iron in biological tissue [4].

The main objective of this experiment was to illustrate the basics of cyclic voltammetry by studying the one-electron reversible redox system. The properties of the reaction: $Fe^{3+}(CN)_6^{3-} / Fe^{2+}(CN)_6^{4-}$ were investigated. The parameters of greatest interest for a reversible CV are the peak cathodic Potential (E_{pc}), the peak anodic potential (E_{pa}), the peak cathodic current (i_{pc}) and the peak anodic current (i_{pa}). The peak currents of a reversible process are given by the Randles-Sevcik equation: $i_p = (2.69 \times 10^5)n^{3/2}AD^{1/2}V^{1/2}$, Where A is the electrode surface area (cm^2) obtained using geometrical measurements, D is the diffusion coefficient (cm^2/s), C is the concentration of the electroactive species in the bulk solution (mol/cm^3) and V is the scan rate (v). Therefore, i_p is proportional to C and proportional to $V^{1/2}$. If A is known, then D can be calculated from the slopes of the linear [5].

Uric Acid (UA) (2, 6, 8-trihydroxypurine) is electroactive biomolecule, a white crystalline powder, soluble in water and is one of the important final end products of purine metabolism in the human body. In healthy persons the concentration of uric acid in urine is around 2 mM and in the blood it is in the range of 120 mM to 450 mM. Normally, the healthy human beings excrete about 400 to 700 mg through urine per day. Abnormal

levels of uric acid are symptoms of several physiological disorders. High levels of uric acid cause hyperuricemia, excess serum accumulation of uric acid in the blood can lead to arthritis (gout). The low concentration of uric acid leads to hyperuricemia and other diseases such as Lesch-Nyhan syndrome and heavy hepatitis. Uric acid is also a marker for the renal failure. Its quantitative determination in body fluids is necessary for the treatment of diseases [6].

The purpose of this paper is to determine the diffusion coefficient (D) of $K_3Fe(CN)_6$ and uric acid; electrochemical nature of $K_3Fe(CN)_6$ and uric acid; the effects of fluctuating concentrations of $K_3Fe(CN)_6$ and uric acid on peak currents and peak splitting and the effects of shifting scan rate of the CV on peak currents and peak splitting [7].

MATERIALS AND METHODS

Reagents

All the reagents used for this experiment were analytical grade, a 100 ml stock solution of 10 mM $K_3Fe(CN)_6$ in 0.1 M KNO_3 was prepared. Successive dilutions of this solution were done. 0.1 M $HClO_4$ was made for cleaning the working electrode during analysis of $K_3Fe(CN)_6$, 1 mM EtOH was made for cleaning the working electrode during analysis of uric acid, 5 mM uric acid and deionized water were used [8].

Apparatus

In the experiment underneath, cyclic voltammeter, Arrangement of a three electrode glass cell was used together, glassy carbon working electrode (Diameter of glassy carbon = 0.3 cm, radius = 0.15 cm) was used, the Ag/AgCl reference electrode, and the platinum wire as a counter electrode [9].

Analytical procedure

The glassy carbon working electrode was refined using a solution of 0.1 M $HClO_4$ and cleaned with distilled water beforehand experimentation to have renewed working surface and to ensure good electron transfer. The solutions that prepared above was added to the cell and carefully eliminated with Oxygen-free Nitrogen 15 minutes before running a CV. In addition, the solution was stirred between experiments in order to restore initial conditions, but it was not stirred during the experiment. 0.1 M KNO_3 supporting electrolyte was to control electrode potentials, eradicate the transport of electroactive species from travelling in the electric field gradients, preserve persistent ionic strength and keep constant pH, this guaranteed a diffusion controlled electrode process. The glassy carbon working electrode was rinsed very well between experiments. The CV was run from -0.2 to 0.8 V, the CV was completed at different scan rates. In addition, the CV was run from -0.2V to 0.8 V by varying the concentration of $K_3Fe(CN)_6$.

To realize electrochemical nature of uric acid, a cyclic voltammetry was run for 0.005 M uric acid with different scan rate, from -0.2 to 1.4 V. The glassy carbon working electrode was rinsed very well between experiments and immersed in 0.001 M EtOH for 30 minutes. The electrode was removed from that the solution and rinses it thoroughly; the experiments were conducted at the room temperature (25°C). Finally, and that

voltammograms of the analytes ($K_3Fe(CN)_6$ and uric acid) were fashioned and interpreted [10].

RESULTS

a. Effects of scan Rate

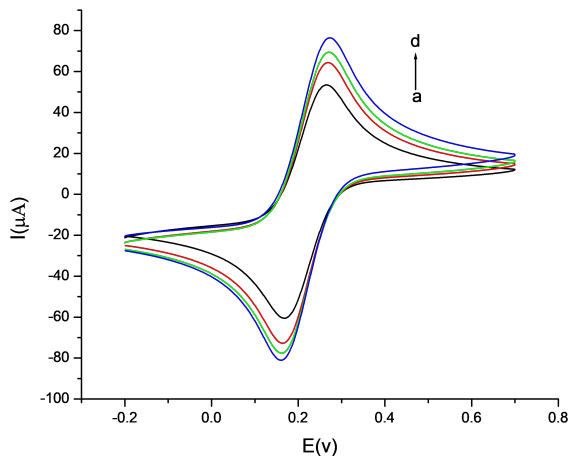


Figure 1: Voltammograms of 5 mM $K_3Fe(CN)_6$ at different scan rate.

Table 1: Voltammetry results of 5 mM $K_3[Fe(CN)_6]$ at the different scan rate.

Scan Rate (mVs^{-1})	Ia(A)	Ic(A)	Ea(V)	Ec(v)
100	63.4	-63.36	0.265	0.169
150	76.31	-77.1	0.268	0.164
175	81.64	-82.31	0.271	0.162
200	87.1	-87.2	0.273	0.161

b. Effects of concentration

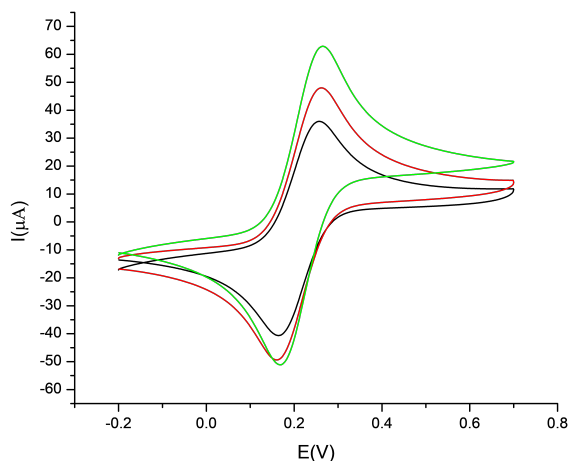


Figure 2: Voltammograms at scan rate $100 mVs^{-1}$ and different concentrations of $K_3Fe(CN)_6$.

Table 2: Voltammetry results of $K_3 [Fe(CN)_6]$ at scan rate $100 mVs^{-1}$ and different concentration.

Conc.(mM)	Ipa	Ipc	Epa	Epc
2	42.46	-43.9	0.257	0.164
3	53.7	-53.7	0.263	0.161
5	63.4	-64.3	0.265	0.169

C. Uric Acid at different scan rate

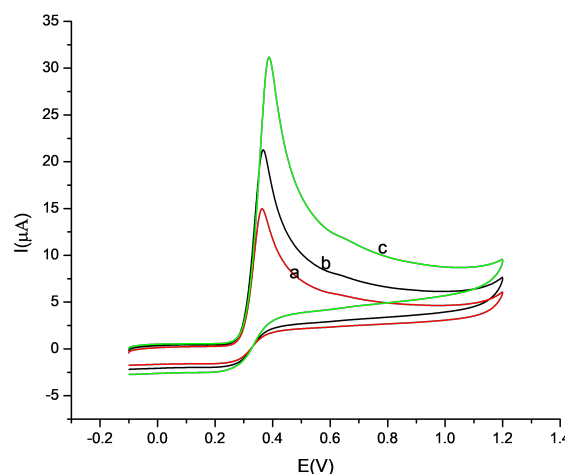


Figure 3: Voltammograms of 5mM Uric Acid at different scan rate.

Table 3: Voltammetry results of 5 mM Uric Acid at different scan rate.

	Scan rate in mVs^{-1}	Current(A)	E(v)
A	100	13.5	0.363
B	125	20.2	0.367
C	150	29.1	0.387

Calculations

Electrode area (A) = $\pi r^2 = 3.14 \times (0.15 \text{ cm})^2 = 0.07065 \text{ cm}^2$; $\Delta E_{peak} = 59 \text{ mV}/n$, $n = 59 \text{ mV} / (0.265 - 0.169) = 0.89 = 1$; $I_p = I_c - I_a = (63.4 - 63.36) \text{ A} = 126.76 \text{ A}$

Determination of diffusion coefficient (D) (cm^2/sec)

For $K_3Fe(CN)_6$ at $5 \text{ mM } K_3Fe(CN)_6$ and $100 \text{ mV}/s$ scan rate: $I_p = (2.69 \times 10^5) n^{3/2} v^{1/2} D^{1/2} A C$ [1], $D^{1/2} = I_p / ((2.69 \times 10^5) n^{3/2} v^{1/2} A C)$, $D^{1/2} = 126.76 / (2.69 \times 10^5 (1)^{3/2} \times (100 \times 10^{-3})^{1/2} \times 0.07065 \times 5)$, $D^{1/2} = 126.76 / 0.300493 \times 10^5 = 421.9707 \times 10^{11}$, $D = 178059.2716 \times 10^{10} \text{ cm}^2/se = 1.78 \times 10^{-5} \text{ cm}^2/sec$.

For Uric Acid at different 5 mM, $I_p = 2.99 \times 10^5 n (\alpha n \alpha)^{1/2} A C D^{1/2} V^{1/2}$ [1], $D^{1/2} = I_p / 2.99 \times 10^5 n (\alpha n \alpha)^{1/2} A C V^{1/2}$, $D = (13.5 / 2.99 \times 10^5 (\alpha n \alpha)^{1/2} (1) \times (100^{3/2} \times 10^{-3})^{1/2} \times 0.07065 \times 5)^2 = 1.595 / \alpha n \alpha \times 10^{-7} \text{ cm}^2 / \text{se}$.

DISCUSSION

Electrochemical behaviors of $K_3[Fe(CN)_6]$ and uric acid were examined, as a result $K_3[Fe(CN)_6]$ under goes redox reaction(forward and back ward scan of electron) while uric acid under goes only for ward scan so that both gives voltammograms which have different shapes which are shown in Figure. 1, 2 and3. The relationship between the scan rate and the peak current were investigated and the result shows that there is a direct relationship between scan rate and peak current that is $I_p \propto V^{1/2}$. As articulated on the above voltammograms, the peak current increases with the augmentation of the concentrations of $K_3[Fe(CN)_6]$, So that, the researcher conclude the manifestation of direct relationship between peak current and concentration of analytes. The experiment of this research shows that the increasing of diffusion coefficient (D) with increasing of concentrations /and scan rate. The peak currents and peak splitting fluctuate with variation of scan rate of CV. Similarly, the peak currents and peak splitting vacillate with variation of concentration of the analytes.

CONCLUSION

The cyclic voltammetry method was successfully applied to check the electro chemical behaviours' of $K_3[Fe(CN)_6]$ and uric acid. The dependence of peak current on scan rate of CV and concentration of electraolyte analyte were examined under optimal conditions. CV therefore can be used as an indication

of major analytical tool for the determination of the trace elements which are electro active in nature. The electrochemical nature of $K_3[Fe(CN)_6]$ and uric acid were determined; reversible and irreversible respectively.

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