**INSTRUMENTAL ANALYSIS LABORATORY**

**NAME OF THE EXPERIMENT:** Cyclic Voltammetry **(**CV)

**DATE OF THE EXPERIMENT:** 24.04.2023 – 25.04.2023

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**GROUP NUMBER:** 6

**DATE SUBMISSION:** 1.05.2023

**CALCULATION PART**

**A)Determination of Diffusion Coefficient by CV and CA**

**Graph 1.** Cathodic Current vs Square root of Scan Rate Graph

**Cathodic Equation**

y = (2x10-6)x +(2x10-7)

R2 = 0,9908

**Randles-Sevcik equation:**

*i*p = (2.69x105).(n)3/2(A).(D)1/2 .(*v*)1/2 .C\*

m=(2.69x105).(n)3/2(A).(D)1/2 .C\*

From experiment,

Fe(.CN.)6+3 + e- Fe(.CN.)6+4 thus n equal to one.

***A***equal to the electrode area which unit is cm2 is 0,002

**C** equal to the concentration of summarization of Fe(.CN.)6+3 and Fe(.CN.)6+4 equal to,

4x10(-6) mol/cm3= 4 mM

Slope = (2x10-6) = (2.69x105).(n)3/2(A).(D)1/2 .C\*

**D** which is the diffusion coefficient equal to **3.048x104**

**Graph 2**. Anodic Current vs square root of Scan Rate Graph

**Anodic Equation**

y = (-2x10-6)x +(-1x10-7)

R2 = 0,9998

**Randles-Sevcik equation:**

*i*p = (2.69x105).(n)3/2(A).(D)1/2 .(*v*)1/2 .C\*

m=(2.69x105).(n)3/2(A).(D)1/2 .C\*

From experiment,

Fe(.CN.)6+3 + e- Fe(.CN.)6+4 thus n equal to one.

***A***equal to the electrode area which unit is cm2 is 0,002

**C** equal to the concentration of summarization of Fe(.CN.)6+3 and Fe(.CN.)6+4 equal to,

4x10(-6) mol/cm3= 4 mM

Slope = (-2x10-6) = (2.69x105).(1)3/2(0.002 cm2).(D)1/2 .( 4x10(-6) mol/cm3)

**D** which is the diffusion coefficient equal to **-9.506x104 cm2/s**

For,

5 seconds D equal to 8.28x10-6 cm2/s

10 seconds D equal to 8.46x10-6 cm2/s

**B)Determination of Ascorbic Acid in Tablet by CV using Standard Addition Method**

From the formula of M1.V1 =M2.V2

V0 = 3 mL sample solution

(0.1M).(V0) =(1x10-3M).(3 mL+V0) => V1 = 33µL =0,033mL

|  |
| --- |
| V1 |

(0.1M).(V1) =(2.5x10-3M).(3.033 mL+V1) => V2 = 46.6µL =0,0466 mL

V2

(0.1M).(V2) =(5x10-3M).(3.0796 mL+V2) => V3 = 81.0µL =0,0810 mL

V3

(0.1M).(V3) =(7.5x10-3M).(3.1606 mL+V3) => V4 = 85.3µL =0,0853 mL

V4

(0.1M).(V4) =(7.5x10-3M).(3.1606 mL+V4) => V5 = 90.1µL =0,0901 mL

V5

**Table 1.** Current (µA) and Concentration of Adding Ascorbic Acid (mM)

|  |  |  |
| --- | --- | --- |
|  | **Current (µA)** | **Concentration** **of Adding Ascorbic Acid** **(mM)** |
| KCl and tablet | 5.182 | 0 |
| KCl and tablet and V1 | 6.367 | 1 |
| KCl and tablet and V2 | 9.4780 | 2.5 |
| KCl and tablet and V3 | 13.90 | 5 |
| KCl and tablet and V4 | 19.13 | 7.5 |
| KCl and tablet and V5 | 23.64 | 10 |

**Graph 3.** Current (A) vs Concentration of Ascorbic Acid(mM) Graph

y = 1,8705x + 4,8888

R2=0,999

When current equal to zero that gives concentration

When y equal to zero,

1,8705x = -4.8888 => x= -2.614 mM this is concentration of Ascorbic acid Ascorbic Acid in Vitamin C solution may say Cdilluted

1 mL tablet solution to 2 mL KCl thus dilution factor is equal to 3,

Molecular weight of Ascorbic Acid = 176.12 g.mol-1 **[1]**

176.12 g.mol-1=0,0113811 g = 13,8111 mg ascorbic acid

**Experimental Value:** 13,8111 mg in 40 mg tablet then weight/weight ratio in percentages equal to,

**Theoretical Value:** 1000 mg Ascorbic Acid in 4500 mg in Vitamin C tablet

**Error %=**

**INSTRUMENTATION PART**

Reference Electrode

Function Generator

RECORDER

Cell

E

i

Working

Electrode

Auxiliary Electrode

Potentiostat

In this experiment, the cyclic voltammetry, device was used to determine, the amount of ascorbic, acid in vitamin C. It acts as a potentiostat function generator in the cyclic voltammetry device. There are working electrodes, reference electrodes, and auxiliary electrodes in our solution cell. Ag/AgCl, electrode is used as the reference electrode in the experiment. As an example of another reference electrode Hg/Hg2Cl2, and Hydrogen electrodes. ,A Platinum working electrode is used as the electrode where, the reaction takes place. In the experiment, a counter, electrode made of a platinum wire, coil was used to conduct electricity and measure using current. Basi® C-3, Cell Stand device was used to prepare the cell, which is the, electrochemical ,reaction that happened Gamry Echem, Analyst™ software was used to make the data coming from the cell measurable..

**POST-LAB QUESTIONS**

**1)** The differences between 2 and 3 electrode systems can be understood by explaining the properties of 2 and 3 electrode systems thus,

**3 electrodes system:**

* It contains working, counter, and reference electrodes.
* The working electrode's potential is measured by the, reference electron, through which no current is flowing, while the counter electrode is passing current.
* The working electrode's potential is measured relative to the reference electrode, which has a fixed value,although the current transition occurs between the counter and, the working electrode.
* One-half of the cell is measured.
* The potential or potential difference of the working electrode, is not dependent on the,, counter electrode.

**2 electrodes system:**

* It has working and counter electrodes.
* The counter,electrode is in the system as the reference electrode.
* The values of the, current going back and forth between the two electrodes are measured.
* The potential, of the electrodes have an instability due to current, so the potentials of, which reactions occur cannot be determined.
* The whole cell is measured.

**2)** High concentration is used to increase the conductivity of the solution to eliminate the electric, field coming from the electrolyte and to create electrostatic attraction to reduce the, transition to the electrode surface. This method can also be used to minimize cell resistance.

**3)** The voltage drop causes distortions in voltammograms. The reference electrode is placed ,close to the working electrode to prevent or reduce this voltage drop**.**

**4)** The auxiliary electrode, also known as the counter electrode, is used for voltametric analysis ,of electric current in three-electrode systems or other reactions taking place inside the cell. It acts as an intermediary to provide the input ,potential to the working electrode. It helps to, complete the circuit by allowing the charge to flow ,it can also be used in two-electrode systems. It provides cyclic voltammetry and linear sweep voltammetry methods, among the electrochemical techniques.

**5)** In order to examine the change in the working electrode, the system needs to be out of balance,, with the potential change in the current. Reference electrodes are needed to make the potential change because they help to record the changing current. If the reference electrodes carry current, it is difficult to read the poteintial changes in the working electrode, as in two-electrode systems, because of the potential changes because it becomes dıfficult to determine which electrode it belongs to. Therefore, it is preferred to use a three-electrode system.

**6)** If the system proceeds with diffusion control in cyclic voltammetry, it has a linear behavior with the square root of the current peak scanning motive. ,On the other hand, if the current has a duration with the scanning rate, it is not a diffusion-controlled system. This situation can be, explained as follows ,charge transfers can occur in a diffusion-controlled manner from those that happen rapidly; this situation is related to the actives, from the electroactive species close to the surface.

**7)**If, electrochemical reactions occur in a system in which electroactive species move spontaneously from an area of high concentratıon to areas where the concentration is lower, then a diffusion-controlled system is said to exist.

**REFERENCES**

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