**Experiment 6**

**Objective :**To study the electrochemical behavior of iron using cyclic voltammetry (CV) and to observe how varying scan rates affect the redox behavior and kinetics of iron.

**Materials and Equipment**

1. **Electrochemical Workstation** (e.g., Potentiostat/Galvanostat)
2. **Three-Electrode System**:

**Working Electrode**: Glassy Carbon Electrode (GCE) or Iron Electrode

**Reference Electrode**: Silver/Silver Chloride (Ag/AgCl) or Saturated Calomel Electrode (SCE)

**Counter Electrode**: Platinum Wire or Mesh

1. **Electrolyte Solution**: 0.1 M KCl or NaCl
2. **Iron Standard Solution**: Prepare a 1 mM solution of Potassium Ferrocyanide in deionized water.
3. **Deionized Water**
4. **Glassware**: Beakers, pipettes, and volumetric flasks
5. **Protective Equipment**: Lab coat, gloves, safety glasses

**Procedure**

**1. Preparation of Solutions**

1. **Iron Solution**:

Prepare a 1 mM iron solution by dissolving the appropriate amount of Potassium ferrocyanide in deionized water.Ensure the solution is well-mixed and filter if necessary to remove any particulates.

1. **Electrolyte Solution**:

Prepare a 0.1 M KCl or NaCl solution to act as the supporting electrolyte.

**2. Electrode Preparation**

1. **Cleaning the Electrode**:

Rinse the glassy carbon electrode (GCE) with deionized water.

Polish the GCE with alumina slurry (0.3 µm) on a polishing cloth, followed by rinsing with deionized water.

Rinse the counter electrode and reference electrode with deionized water.

1. **Electrode Assembly**:

Assemble the three-electrode system in a suitable electrochemical cell.

Ensure proper positioning and connection of the working electrode (GCE or iron electrode), reference electrode (Ag/AgCl or SCE), and counter electrode (platinum wire).

**3. Cyclic Voltammetry Measurement**

1. **Cell Setup**:

Fill the electrochemical cell with the 0.1 M electrolyte solution.

Insert the working, reference, and counter electrodes into the cell.

1. **Baseline Measurement**:

Perform a baseline cyclic voltammetry measurement in the electrolyte solution to establish a reference profile.

1. **Measurement with Different Scan Rates**:

Set up the cyclic voltammetry parameters on the electrochemical workstation:

* + - **Potential Range**: Typically from -0.5 V to 1.0 V (vs. Ag/AgCl)
    - **Scan Rates**: Perform measurements at multiple scan rates (e.g., 10 mV/s, 50 mV/s, 100 mV/s, 200 mV/s).
    - **Number of Cycles**: 2 to 3

Run the cyclic voltammetry scans for each scan rate, recording the voltammograms.

**4. Data Analysis**

1. **Peak Analysis**:

Identify the anodic and cathodic peaks in each voltammogram.

Measure the peak current (I\_peak) and peak potential (E\_peak) for each scan rate.

1. **Effect of Scan Rate**:

Plot the peak current versus scan rate to analyze the relationship between scan rate and redox current.

Analyze the peak potential shift with changing scan rates to study the reversibility and kinetics of the iron redox reactions.

1. **Kinetic Analysis**:

Use the Randles-Sevcik equation to analyze the diffusion-controlled processes if applicable



Ipeak​ is the peak current, nnn is the number of electrons transferred, A is the electrode area, D is the diffusion coefficient, v is the scan rate, and C is the concentration of the analyte.

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**Precautions**

* Always wear appropriate personal protective equipment (PPE) including lab coats, gloves, and safety glasses.
* Handle all chemicals with care and follow standard laboratory safety protocols.
* Dispose of iron-containing solutions and other chemical waste according to your institution’s hazardous waste disposal procedures.

**References**

* Bard, A. J., & Faulkner, L. R. (2001). Electrochemical Methods: Fundamentals and Applications. John Wiley & Sons.
* G. T. White, K. J. Gifford, & J. L. Gerlach. (2020). Cyclic Voltammetry: Practical Aspects of Working with Various Electrodes. Academic Press.

**Pre-Test Questions**

1. **What is the main purpose of performing cyclic voltammetry in this experiment?**
   * A) To measure the solubility of iron
   * B) To study the redox behavior of iron
   * C) To determine the boiling point of iron
   * D) To analyze the thermal stability of iron

**Answer: B) To study the redox behavior of iron**

1. **What does the scan rate refer to in cyclic voltammetry?**
   * A) The rate at which the sample is introduced into the cell
   * B) The rate at which the potential is changed
   * C) The rate of reaction at the electrode surface
   * D) The rate at which the electrode is cleaned

**Answer: B) The rate at which the potential is changed**

1. **Which electrode is typically used as the working electrode in cyclic voltammetry experiments?**
   * A) Platinum Wire
   * B) Glassy Carbon Electrode (GCE)
   * C) Silver/Silver Chloride Electrode
   * D) Saturated Calomel Electrode

**Answer: B) Glassy Carbon Electrode (GCE)**

1. **In cyclic voltammetry, what is the role of the supporting electrolyte?**
   * A) To provide a stable reference potential
   * B) To maintain ionic conductivity in the solution
   * C) To react with the analyte
   * D) To control the temperature of the solution

**Answer: B) To maintain ionic conductivity in the solution**

1. **What is typically observed in a cyclic voltammogram as a result of a reduction reaction?**
   * A) A peak at a positive potential
   * B) A peak at a negative potential
   * C) A flat baseline
   * D) No current response

**Answer: B) A peak at a negative potential**

**Post-Test Questions**

1. **When analyzing cyclic voltammetry data, what is plotted on the y-axis of a current versus potential plot?**
   * A) Potential
   * B) Scan Rate
   * C) Current
   * D) Concentration

**Answer: C) Current**

1. **How does increasing the scan rate affect the peak current in a cyclic voltammogram for a diffusion-controlled process?**
   * A) The peak current decreases
   * B) The peak current remains unchanged
   * C) The peak current increases
   * D) The peak current disappears

**Answer: C) The peak current increases**

1. **If the peak potential shifts to a more positive value with increasing scan rate, what does this indicate about the redox reaction?**
   * A) The reaction is reversible
   * B) The reaction is diffusion-controlled
   * C) The reaction is irreversible
   * D) The reaction is at equilibrium

**Answer: C) The reaction is irreversible**

1. **What information can be derived from the Randles-Sevcik equation in cyclic voltammetry?**
   * A) The pH of the solution
   * B) The concentration of the analyte
   * C) The temperature of the solution
   * D) The diffusion coefficient of the analyte

**Answer: D) The diffusion coefficient of the analyte**

1. **Which of the following is true when comparing cyclic voltammetry results obtained at different scan rates?**
   * A) The peak current is independent of scan rate
   * B) The peak current decreases with increasing scan rate
   * C) The peak current increases with increasing scan rate
   * D) The peak potential does not change with scan rate

**Answer: C) The peak current increases with increasing scan rate**