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Electrochemical characterization using of Cyclic Voltammetry

Abstract

Cyclic Voltammetry is a popular electrochemical study for studies the sample like capacitance, charge storage mechanism, redox and oxidation reaction.CV electrochemical cell setup and set the parameter of the CV like as scan rate , potential window etc.. In CV three electrodes reference electrode, working electrode and counter electrode about electrode materials and electrode configuration. Calculation the specific capacitance with various scan rate and charge storage mechanism used CV data. CV has many applications in various fields like as analytical chemistry, Biological and Pharmaceutical industry and material science.

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Introduction

Cyclic Voltammetry (CV) is an electrochemical analysis technique. CV is an electrochemical method that gives qualitative and quantitative information about redox-active of polymer electrolyte and any sample species by relating a cyclically changing potential to an electrode immersed in an electrolyte solution and calculating the resulting current. Since its inception in the mid-20th century, CV has become indispensable in fundamental research and practical applications such as sensor development, corrosion studies, battery research, and catalysis. Compared to traditional capacitors and batteries, electrochemical capacitors—also referred to as supercapacitors or ultracapacitors—have shown great stability, a high power density, and a moderate capacity for energy storage, making them viable power sources. Applications requiring high power with brief current bursts, such electric vehicle startup and uninterruptible power supply (UPS) systems, benefit greatly from their utilization. Two processes in supercapacitors store energy: redox reactions involving the electrode material and electrostatic interactions between the charged electrode surfaces and electrolyte ions. Activated carbon, metal oxides, and conductive polymers are common electrode materials used in electrical double-layer capacitors (EDLCs). The

electrode material's surface area, pore size, and pore geometry all have a significant impact on capacitance. These characteristics improve capacitance by making better use of the electrode surface. The mass, volume, and surface area of the electrode all affect specific capacitance. Capacitance values are frequently restricted because only a part of the pores actively contribute to the creation of double layers. Nonetheless, porous materials that have pore diameters that are in good proportion to the electrolyte ions are especially beneficial because they speed up ion transit, increase efficiency, and produce larger capacitance values [Josi et.al. 2018].

In summary, the electrolyte mostly determines the device's voltage and resistance, but the capacitance of the capacitor is primarily determined by the electrode material's capacity, expressed in F/g.The electrode's potential is linked to the oxidation or reduction happening at the electrode surface. Electrode ions in solution are forced to improvement or miss an electron by the voltage. A stronger reducing electrode is implied by a greater negative potential, while a stronger oxidizing electrode is implied by a higher positive potential. The electrode's potential is associated to the oxidation or reduction happening at the electrode surface. Electrode ions in solution are forced to gain or lose an electron by the voltage. A stronger reducing electrode is implied by a greater negative potential, while a stronger oxidizing electrode is implied by a higher positive potential. As a outcome, regulating an electrochemical cell's electrode potential can regulate the redox reaction inspiring on the electrode. Therefore, it is crucial to investigate the electrochemical reaction taking place at the electrode/electrolyte contact.

Because of its simple measurement process, CV is one of the greatest commonly used electrochemical techniques. A CV provides important information on the activity of chemically or electrochemically reactive species as well as the processes taking place at the electrode surface. As a result, CV is widely used in electrode characterization, reaction monitoring, and early investigations of novel systems. Because the curves include both kinetic and thermodynamic parameters and are affected by the analyte, electrolyte, and electrode situations, interpreting CV data can be difficult despite its value. To analyze electrochemical behavior and derive useful information from voltammograms, novices in electrochemistry must grasp the foundations of CV. A universal definition is challenging since different voltammogram patterns can be produced by different experimental setups.

In this work, discussed about the cyclic voltammetry principle, electrochemical cell setup, reference, working and counter electrode. A sort discussion about the electrode configuration, electrode material and how to set the experimental parameter like as potential window and scan rate. Calculate the specific capacitance with various scan rate and charge storage mechanism using by the Cyclic Voltammetry and its application in various fields like as biological, pharmaceutical, analytical chemistry and materials science studies.

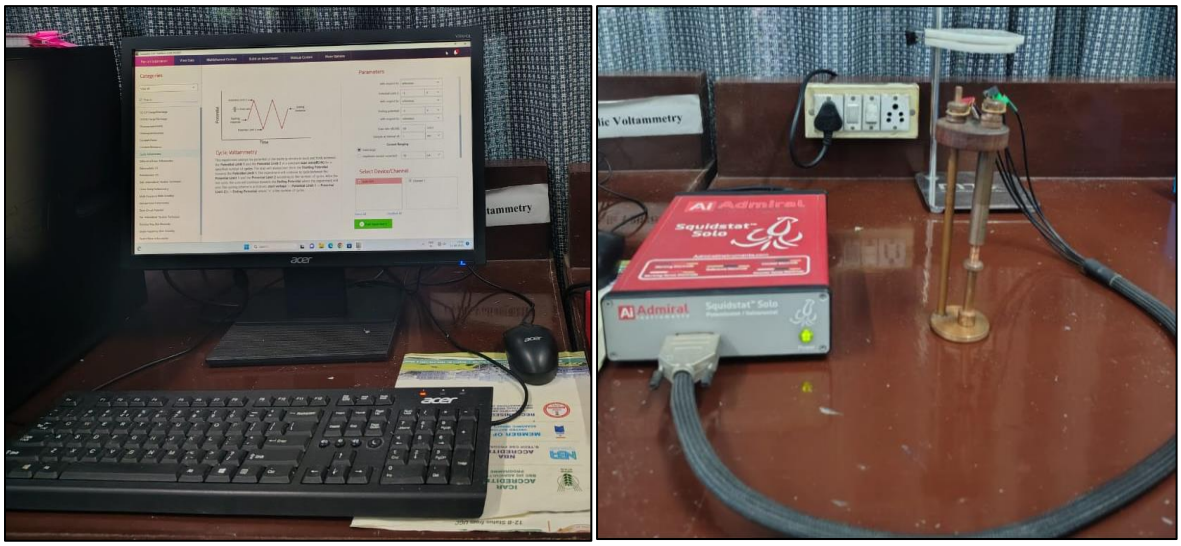

Figure 1: Cyclic Voltammetry set –up (Squidstat Solo from DTech solution).

2. Fundamental Principles of Cyclic Voltammetry

2.1 Electrochemical Cell Setup

A CV setup consists of a three-electrode system in this system have a one WE, RE and CE. Briefly describe about these electrode and configuration of two electrode systems, three electrode systems, four electrode systems and five electrode configurations.

Working electrode (WE):

In order to perform the intended electrochemical reactions, the working electrode (WE) is essential. An electrochemically inert electrode is typically chosen for assessing a species' redox potential in solution. Pt, Au, and carbon-based electrodes with well-defined surface areas, including glassy carbon or pyrolytic graphite, are popular options. Wire-shaped electrodes are not appropriate for measurements based on one-dimensional diffusion; instead, a flat electrode with an area significantly greater than the thickness of the diffusion layer is needed. Even though variables like double-layer capacitance, charging current, and gas adsorption behavior differ depending on the electrode material, they typically don't affect research on other electrode reactions as long as the applied current stays constant (unless the focus is on gas adsorption). The WE surface needs to be exceedingly clean in order to guarantee good repeatability. An ultrasonic cleaning procedure in ultrapure water is typically used after mechanical polishing using abrasives like alumina. Cleaning procedures, however, can vary from lab to lab and frequently rely on the electrode material, therefore initial optimization is crucial prior to measurement.

Reference electrode (RE):

During measurements, no current flows through the reference electrode (RE), which is in charge of regulating the working electrode's (WE) potential. An RE with a stable and highly reproducible potential is necessary to properly assess electrode reactions from a thermodynamic standpoint. Ag/AgCl (x mol dm^{-3} KCl) and $Hg/HgCl_2$ with saturated KCl (saturated calomel electrode, SCE) are frequently used in aqueous electrolyte systems. To avoid electrolyte cross-contamination, these REs usually employ porous glass or ceramic connections. Despite being able to be produced in a lab, trustworthy commercial versions are currently freely accessible. Additionally available are reservoir-type reversible hydrogen electrodes (RHE), which regulate the WE potential in accordance with the equilibrium established by proton activity and hydrogen partial pressure. For applications requiring long-term potential stability, salt bridges or double-junction reference electrodes are often used. provides a stable

reference potential.

Counter electrode (CE):

CE experiences a complementary redox reaction to that at WE and is also referred to as an auxiliary electrode. When oxidation occurs at CE, reduction occurs at WE, and vice versa. The CE must be able to handle a current higher than that seen at the WE in order for the reaction at the CE to be clearly ascribed to either electrolyte breakdown or the redox reaction of the active species. To lower the current density at the CE and guarantee that the polarization is connected to the electrode reaction at the WE, a mesh or coiled Pt or Au electrode is typically utilized. Its area is several dozen times greater than that of the WE. Mainly two type of electrode configuration -

Two Electrode Configuration-

The WC and WSC are joined and connected to the WE designated for the experiment in this electrode configuration. Together, the reference electrode, counter electrode, and counter sense electrode are linked to the CE designated for the experiment. Changes in the CE throughout the experiment have an impact on the potential at the WE because the WE's potential is fixed in relation to the CE. But it's also the most straightforward electrode arrangement shown in fig. (2) (Squidstat et.al.).

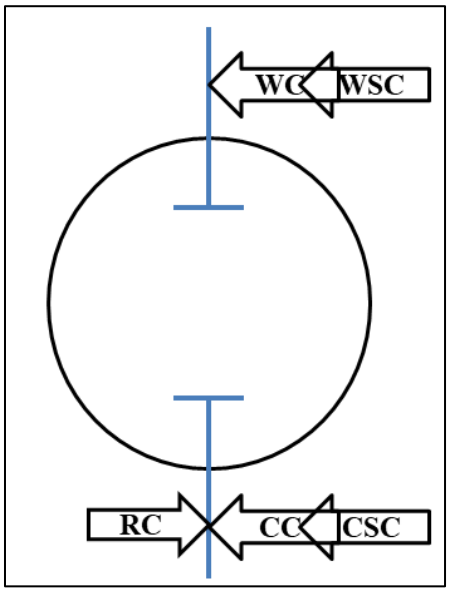

Figure 2: Two-electrode Configuration

Three Electrode Configuration-

The most widely used arrangement in analytical electrochemistry and physics. The WE designated for the experiment is connected to the WC and WSC in this manner. The CE designated for the experiment is linked to the CC and CSC. The RE and the RC are linked. Changes in the CE throughout the experiment have no effect on the potential at the working electrode since the working electrode's potential is applied or measured in relation to an independent RE Shown in Fig.(3) (Squidstat et.al.).

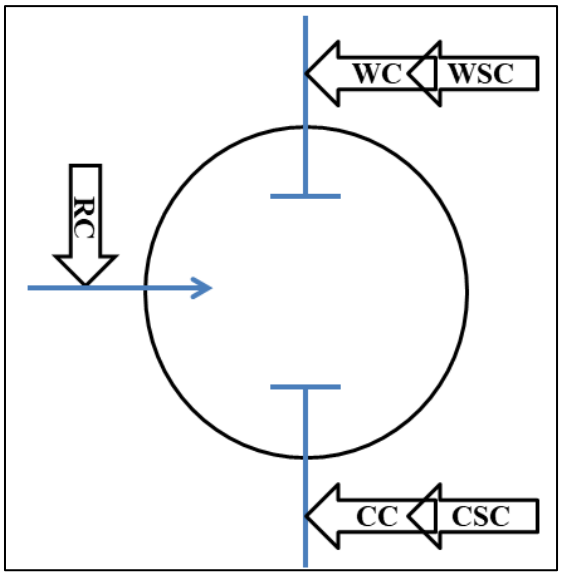

Figure 3: Three – electrode Configuration

2.2 Potential Sweep and Current Response

In material science, cyclic voltammetry (CV) is a popular electrochemical method for examining interfacial characteristics, electrode kinetics, and redox processes. This technique creates a triangle waveform by sweeping the working electrode's (WE) potential linearly over time from an initial potential (E_i) to a vertex potential (E_v), then reversing the process. Plotting the resultant current against the applied potential creates a cyclic voltammogram, which offers important insights into material behavior and electron transfer reactions. Faradaic and non-Faradaic currents are the two primary components that make up the overall current response in CV. The Faradaic current is caused by the oxidation or reduction of electroactive species that displays the charge transfer processes at the electrode surface. Non-Faradaic current, the capacitive current, however, is caused by the charging and discharging of the electric double layer. To comprehend the electrochemical performance and working nature of the materials under investigation, one should be capable of isolating these contributions.

2.3 Redox Mechanism and Reversibility

When interpreting the results of cyclic voltammetry (CV), the degree of reversibility and the redox process are essential factors. The ability of the oxidized and reduced forms of a given species to easily change into each other over the timescale of the electrochemical experiment is referred to as reversibility. The voltammogram has sharp, symmetric shapes of the peaks and the separation between the peaks (ΔEp) is close to the theoretical value of about 59mV/electron at 25ºC showing that in the ideal reversible system, this interconversion process is fast enough to establish equilibrium at the electrode surface. This type of behavior recommends that the process is primarily accurate through diffusion and that the electron transfer kinetics is rapid enough. Instead, many systems can be classified as being quasi-reversible or irreversible as they do not follow this ideal case. Under such circumstances, slow electron transfer rates, chemical reactions which are linked or other kinetic limitations cause bigger peaks, higher ΔEp values and distorted voltammogram profiles. These variations provide great information about the underlying electron transport kinetics and chemical reactions in the system. Since the efficiency of the electron transfer has a direct influence on the performance of materials in such applications as catalysis, energy storage, and sensor manufacturing, a clear understanding of the level of reversibility is crucial to the evaluation

of the electrochemical characteristics of materials (Khan et.al. 2023).

3. Instrumentation and Experimental Procedure

3.1 Potentiostat

A potentiostat controls the potential applied to the WE and measures the resulting current. It is essential for precise potential control and sensitive current measurement. The key electrical device in cyclic voltammetry is a potentiostat, which applies and maintains the exact voltage at the working electrode (WE) in relation to a steady reference electrode while also measuring the current that results via the counter electrode. It is essential for precise, sensitive measurements in material science since it depends on feedback control, usually through operational amplifiers, to maintain tight potential regulation even when the electrochemical reaction circumstances change (Yamada et.al. 2022).

3.2 Electrode Materials

Glassy carbon, platinum, and gold are common WE materials in modern electrochemical research and each has unique advantages: Noble metals like Pt and Au offer higher conductivity and catalytic activity, while glassy carbon offers a broad stable potential window and chemical inertness. However, careful surface preparation is essential to getting consistent, dependable outcomes. Advanced activation techniques that improve performance and reproducibility have been reported in recent literature. For instance, activated GCEs with improved sensitivity and stability in epinephrine detection were created by a two-step electrochemical pretreatment of a glassy carbon electrode using cyclic voltammetry. Furthermore, as a convincing alternative to conventional polishing, alternative cleaning methods including oxygen plasma exposure have demonstrated enhanced voltammetric response and quasi-stable surface characteristics over time **(**Galloni et.al. 2025).

3.3 Experimental Parameters

Several important experimental settings have a significant impact on the quality and interpretability of cyclic voltammetry (CV) data.

Scan rate (v): The speed at which the voltage is swept across the working electrode is determined by the scan rate Fig. (5), which normally ranges from 10 mV/s to 1 V/s. It has a direct impact on the voltammogram profile and peak current. Diffusion-controlled processes take Centre stage at slower scan speeds, producing distinct peaks. Higher scan speeds, on the other hand, can change peak potentials and increase capacitive currents, which can reveal information on the kinetics of electron transport. It is common practice to differentiate between diffusion-controlled and surface-confined processes using the connection between peak current and the square root of the scan rate.

Potential window: The solvent's and the supporting electrolyte's electrochemical stability determine the critical potential range selection Fig. (4).If this window is exceeded, electrode deterioration, gas evolution, or solvent breakdown may result. Therefore, choosing a suitable potential window guarantees that just the analyte's redox behaviour is recorded.

Concentration of supporting electrolyte:

An inert supporting electrolyte at a high enough concentration reduces solution resistance (iR drop) and inhibits the effects of charged species migration. By guaranteeing that mass transit happens primarily by diffusion, this enhances the accuracy and repeatability of CV measurements.

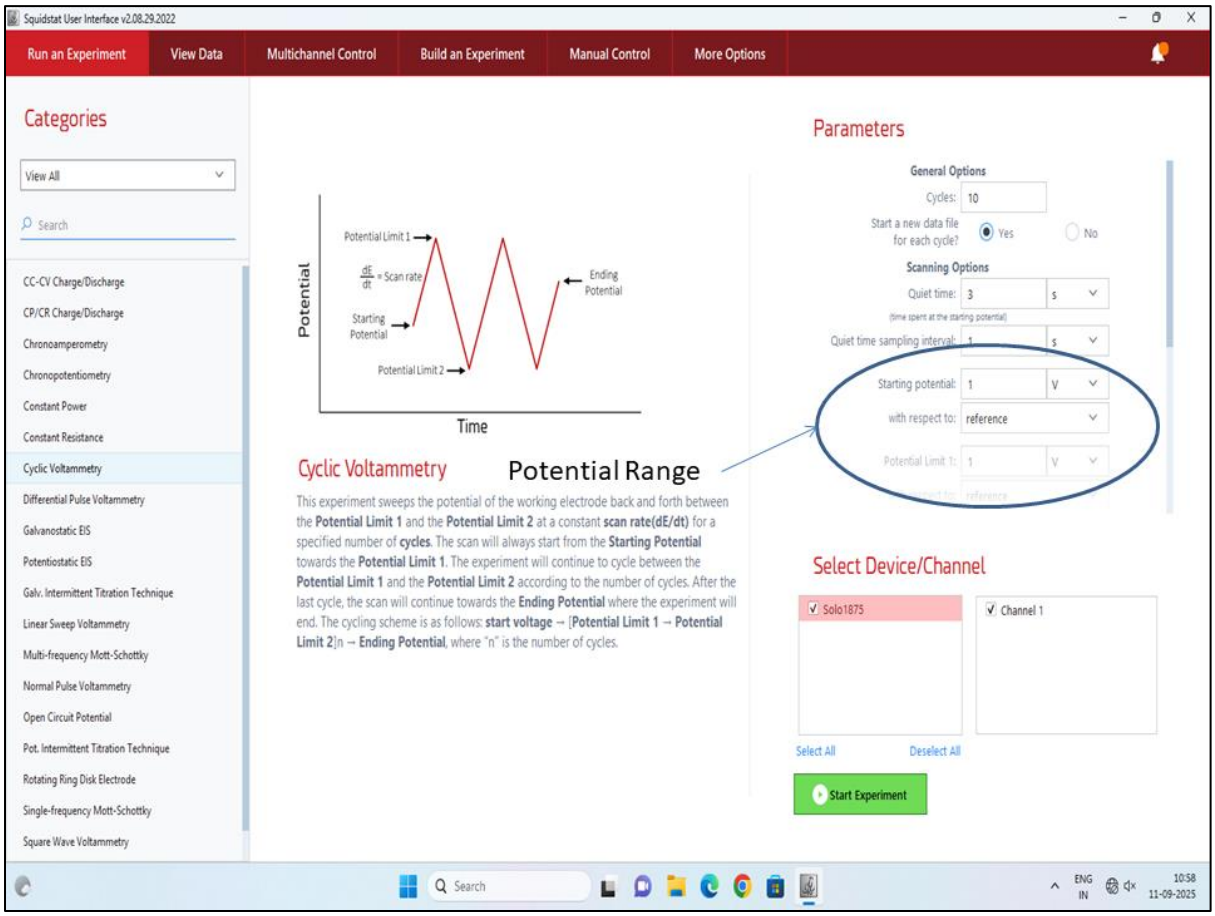

Figure 4: Potential window measurement in CV parameters.

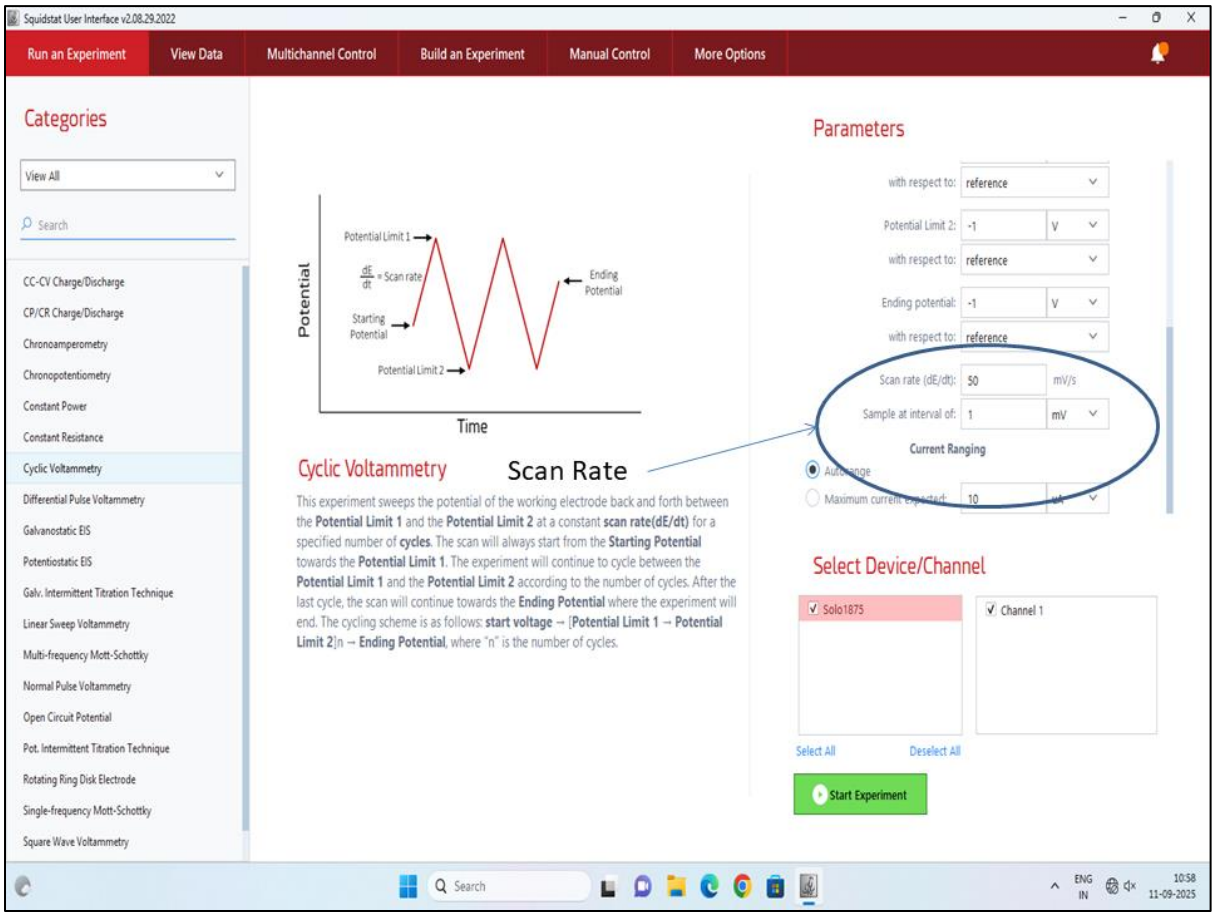

Figure 5: Scan rate measurement in CV parameter.

4. Data Interpretation

Calculate the Charge Storage Mechanism

For the analysis of CV, at varying scan rates, it displays the quasi-reversible reaction. First, we used equation (1), which examines the link between current (i) and scan rate (v), to identify the charge storage mechanism and examine the electrochemical behavior at different scan rates.

$$i = a.v^b \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \quad (1)$$

This charge storage mechanism's type is indicated by the exponent 'b'. According to Kumar et al. (2025a), a diffusion-controlled process is indicated by b = 0.5, whereas a surface-controlled (capacitive) process is indicated by b = 1. B-values such as b= 0.014, 1.0, 0.19, 0.038, and 0.59 were calculated at scan speeds of 10 mV/s, 20 mV/s, 40 mV/s, and 80 mV/s, respectively.

Calculate the Specific Capacitance

Computes capacitance and identifies the potential window for the electrical characteristics of any sample using cyclic voltammetry (CV). The CV was also used to test the electrical characteristics of the sample, including its capacitance. It is taken through the stainless steel electrode in the two-electrode circuit's design.This engagements two-electrode circuit that provides the specific capacitance using by this formula, Eqn. (2) (Kumar et.al. 2025b).

$$C_s = \frac{A}{2mk(V_2 - V_1)} \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (2)$$

Where m, V_1 and V_2 signify mass of the BPE, initial and final potentials, C_s is the capacitance and K is the scan rate and A is the area of the CV Curve.

Charge Storage Mechanism and Specific Capacitance

For analysis potential window and capacitance analysis of PVA CS with KI blend polymer electrolyte used the CV. For sample analysis, CV (model name Squidstat Solo) is utilized. Other works of literature are contrasted with the voltammogram (Sharma et.al. 2024). It is utilized with the 1x1 cm^2 stainless steel electrode in the circuit's two-electrode design.

Table 1: Information about K (Scan Rate) and C_S (Specific Capacitance) for the PVA:CS:KI.

Scan Rate (K) (mV/s)	**Specific Capacitance (C_S) (F/g)**
10	12 X 10^{-5}
20	1.4 X10^{-4}
40	2.25 X10^{-4}
80	1.57 X10^{-4}

Additionally, CV was utilized to examination the electrical characteristics of the prepared Sample, including its specific capacitance and potential window (N. Dobre et.al. 2014). Squidstat Solo is used for Sample capacity analysis. Squidstat Solo employs two-electrode circuit architecture with a 1x1 cm^2 stainless steel electrode. The potential window is directly proportionate to allow the capacity of charge flow between the potential range 1.78 to 1.98 V in the electrolyte. The cyclic voltammetry used to measurement the specific capacitance from equation (2), CV curve figure (6) and calculate the charge storage mechanism using by equation (1). The specific capacitance changes with changes in scan rate.

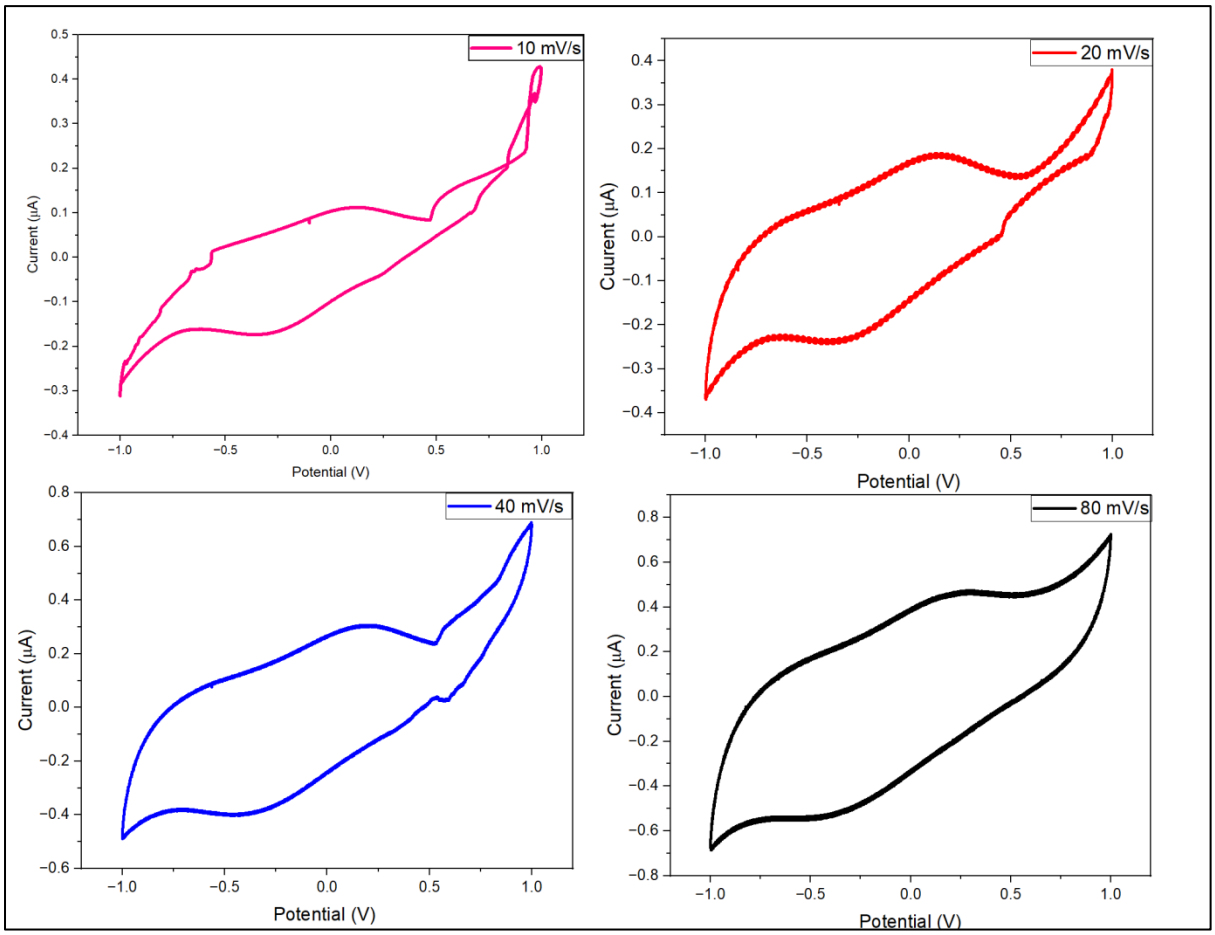

Figure 6: Cyclic Voltammetry curve different scan rate 10, 20, 40 and 80 mV/s.

5. Applications of Cyclic Voltammetry

5.1 Analytical Chemistry

CV applies correlation between the current magnitude at peak current and analyte concentration to determine the analyte, even small biomolecule, medicine, and antioxidant quantities. Indicatively, the amplitude of the maxima of cyclic voltammetry has a direct correlation with the concentration of redox-active species, a phenomenon that has been utilized to determine the range of antioxidants in biological samples like plasma. CV provides the unique potential values at which redox transitions in substances take place. It is possible to identify and analyze redox-active species in complex matrices using these values. CV can help to determine the formation and strength of chemical complexes by tracking the shift in peak or current with the addition of the ligands or the interacting species (Wang et.al. 2021).

5.2 Materials Science

The functioning of conductive polymers and composites is assessed regarding their redox activity, capacitive behavior and charge storage capabilities by using CV. An example is that where graphene electrodes in a supercapacitor are reduced, reduced graphene oxide (rGO) electrodes have practically rectangular current voltage characteristic (CV) traces, which is an indicator of enhanced double-layer capacitance. CV is a fundamental method when analyzing how battery materials can be inserted into the battery and how this diffusion process can be kinetically explored, electrochemically, and how it relates to stability. CV has demonstrated the importance of

full reduction in lithiation with the condition of complete reduction to permit more stability and capacity in such hybrid systems as CuO–graphene oxide anodes. Similarly, CV also has found use in the investigation of sodium-ion electrodes to establish diffusion coefficients and understand pseudo-capacitance behavior. CV can also measure protective finishes, monitor surface reactivities, and examine cases of corrosion by using diverse voltammogram (Li et.al. 2025).

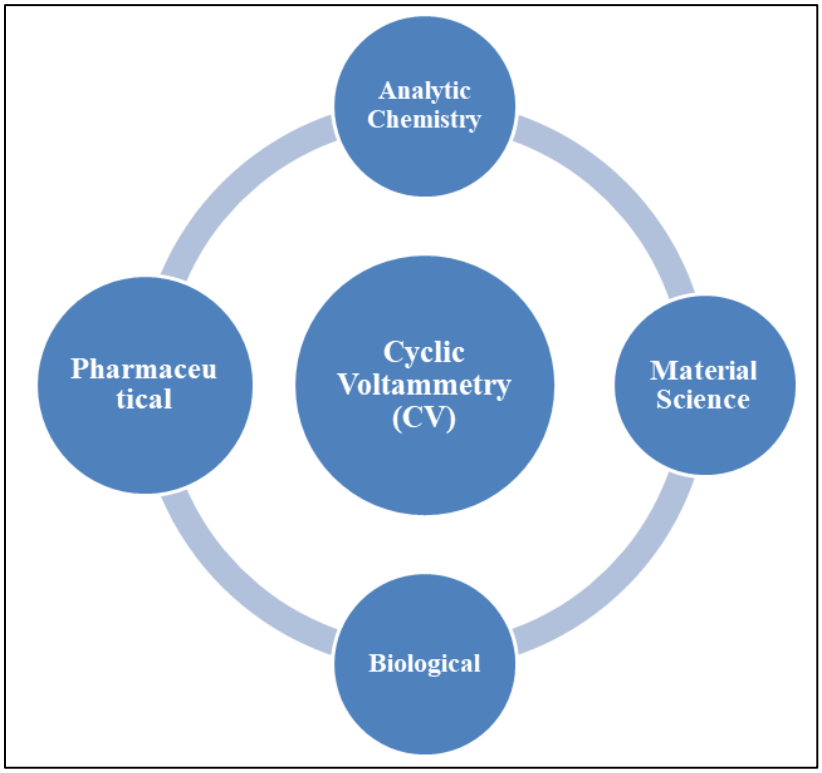

Figure 7: A schematic diagram representing various application of Cyclic Voltammetry.

5.3 Biological and Pharmaceutical Applications

CV is useful in biosensor construction and characterization of redox processes of enzymes. Among them, the immobilization of laccase on an electrode modified using β-cyclodextrin polymer should be mentioned as an example, making it possible to immobilize laccase so that the antioxidant capacity can be reliably measured in real-world samples, including tea infusion. Enzyme inhibition-based sensors based on cytochrome P450, which have been at least in part used to measure pharmaceutical substances and estimate inhibition constants, have also been applied to CV. In the case of drug compounds and their metabolites, CV facilitates the analysis of the metabolite alterations, binding dynamics, as well as other detection limits. Nanomaterial-enhanced voltammetric sensors can detect biomolecules like dopamine, uric acid, ascorbic acid in a sensitive manner in a clinical, food, and environmental situation (nanomaterials used include metal nanoparticles or carbon nanostructure) (Harun et.al. 2025).

Conclusion

Here discussed about the fundamental aspects of CV were systematically explored, including its operating principle, electrochemical cell configuration, and the roles of reference, working, and counter electrodes. Appropriate electrode material and electrode configuration, potential and scan rate were also discussed as major experimental parameters that are important to provide reliable measurements. Specific capacitance was tested within varying scan rates in order to comprehend charge- storage behavior and the electrochemical process behind

the behavior. This paper shows that CV is an informative technique on redox processes and capacitive performance and is a helpful and versatile method of characterizing energy-storage materials. Moreover, the former generalizability of CV to analytical chemistry, materials science, biological systems and pharmaceutical studies reveals its significance in reaction kinetics, electroactive species and new electrode materials research. Altogether, the publication supports CV as a critical method of fundamental research and advanced applications in both elementary and progressive studies and technology it is the heart of the electrochemical research.

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