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## Enhanced Electrochemical Performance Of Aqueous Supercapacitors Through Optimized Redox-Active Materials

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### **Abstract**

Aqueous super capacitors are gaining prominence in energy storage due to their high power density, fast charge/discharge cycles, low environmental impact, and cost-effectiveness. Despite these benefits, they suffer from relatively low energy density compared to batteries. One of the emerging strategies to improve energy storage performance is the introduction of redox-active species in electrolytes. In this study, p-phenylenediamine (PPD), an organic redox-active molecule, is incorporated into 1 M KOH aqueous electrolyte and used with high-surface-area carbon bead electrodes. The influence of PPD concentration (0, 0.05, 0.10, 0.20 M) on electrochemical performance is evaluated using cyclic voltammetry (CV), galvanostatic charge-discharge (GCD), and electrochemical impedance spectroscopy (EIS). Surface morphology is characterized via atomic force microscopy (AFM) and scanning electron microscopy (SEM). Optimized system (0.10 M PPD) has a given capacitance (518 F/g) of 94% capacitance retention at 5000 cycles and low equivalent series resistance of 1.3  $\Omega$ . These results validate the role of redox mediation by PPD in making the storage of energy much better, hence its potential in the future development of aqueous super capacitor systems of the next generation.

**Keywords:** Aqueous super capacitors, p-phenylenediamine, redox mediator, KOH electrolyte, electrochemical energy storage, carbon electrodes, pseudo capacitance.

### **1. Introduction**

Enhanced demand of sustainable and high-performance energy storage systems has focused much focus on super capacitors, due to its unique benefits over conventional batteries, such as high power density, fast charge/discharge, and long lifecycle [1]. Super capacitors fill in the divide between battery and conventional capacitors and work through two major processes, which are electric double-layer capacitance (EDLC) and pseudo capacitance. Whereas EDLC is the accumulation of electrostatic charge at the electrode-electrolyte interface, the source of pseudo capacitance is the rapid, reversible faradaic redox reaction which has a greater energy storage capacity [2].



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Although these are the benefits, the relatively low energy density of super capacitors remains a chronic problem with this type of technology and prevents their wider use in large-scale energy systems. In attempts to solve this, researchers have considered several approaches such as changing electrode materials, high surface-area nanostructures, and redox-active electrolytes development [3].

## **Aqueous Redox-Active Electrolytes**

One of these approaches has been incorporation of redox-active species into aqueous electrolytes, which have proven to be one of the most promising ways to increase capacitance and energy density, yet avoid compromising power capability and stability [4]. Aqueous electrolytes (KOH or H<sub>2</sub>SO<sub>4</sub>) are beneficial since, they are cheap, highly ionic conductive, and non-toxic. Their voltage window (V) is however limited at (1.2 V), limiting the energy density ( $E = \frac{1}{2} CV^2$ ). To overcome this drawback, redox mediators, molecules that are able to undergo rapid and reversible redox reactions, can be added to the electrolyte to add further charge storage processes and enhance energy scale [5].

## **Role of p-Phenylenediamine (PPD)**

p-Phenylenediamine (PPD) has drawn the interest of efficient redox additive because of its good electrochemical characteristics and its stability in alkaline medium. It is also involved in two-electron, two-proton reversible redox reactions, which plays a major role in the pseudo capacitive behaviour of the electrolyte [6]. Introduction of PPD into a standard aqueous KOH electrolyte has the potential to increase diffusion of ions, reduce charge transfer resistance, and add extra faradaic reactions, which results in better overall performance of a device [7].

Indicatively, Yu et al. (2012) established that the incorporation of PPD in an electrolyte based on KOH had a significant positive effect on the performance of MnO<sub>2</sub> based super capacitors. Specific capacitance was found to be more than six-fold (44.87 F/g to 325.24 F/g) and the energy density was found to be increased almost tenfold, confirming the value of PPD as a redox-active mediator [8].

In another research, Chen and Lin (2019) tested a dual-additive system to explore the effect of combining various redox mediators with hydroquinone and PPD, which showed the synergistic effect of adding different redox mediators. The optimized system reached a given capacitance of 116.23 F/g and the potential window was further pushed to 1.6 V signifying an increase in the energy density as well as the voltage window [9].

Moreover, Xu et al. (2021) examined a thinned film of graphene oxide (rGO) functionalized with PPD, in which PPD was not only a redox mediator but also a molecular spacer to increase the accessibility of ions. This dual functionality enabled the Zn-ion hybrid super capacitor to reach an exceptionally high areal capacitance of 3012.5 mF/cm<sup>2</sup> [10].



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## Mechanistic Insights

The redox behavior of PPD in alkaline environments typically involves proton-coupled electron transfer mechanisms. The transformation between its oxidized and reduced forms (quinonoid and diamine structures) allows for continuous faradaic cycling, effectively transforming the inert aqueous medium into a charge-storing medium [11]. Importantly, PPD demonstrates fast redox kinetics and reversible electrochemical behavior, both crucial for high-rate energy storage applications [12].

This is corroborated by **Hatzell et al. (2013)**, who reported that the addition of PPD in KOH improved capacitance by 86% in flowable carbon-based suspensions, attributing the enhancement to quick redox switching and lowered internal resistance [13]. Similarly, **Goel and Mishra (2022)** observed increased pseudocapacitance when using  $\text{LaNiO}_3$  in a KOH+PPD system, indicating the synergistic behavior of redox-active electrodes and electrolytes [14].

## Recent Advancements and Research Gaps

The recent developments emphasize the merits of coming up with electrode-electrolyte systems in which the two constituents actively engage in storing charge. PPD has also been grafted onto other electrode materials as well such as carbon nanotubes and graphene, and the covalent bonding of redox centres and the encouragement of charge delocalization are also observed. As an example, PPD was used by Liu et al. (2023) to encapsulate p-benzoquinone into the microporous carbon framework to obtain specific capacitance values (greater than 370 F/g) and impressive energy density [15].

Although these results are promising, there are still difficulties. PPD has been known to degrade slowly when subjected to high potential cycling and the solubility in water can be limiting to long-term retention in the electrolyte. Hence, the approaches of covalent tethering, polymeric encapsulations or co-mediation with stabilizers are also under investigation in order to resolve such problems [16].

## Objectives of the Present Study

This study aims to investigate the role of **p-phenylenediamine (PPD) as a redox-active mediator in a 1M KOH electrolyte**, focusing on its effects on:

- Specific capacitance enhancement
- Charge/discharge behavior
- Electrochemical stability
- Charge transfer resistance

The electrodes of the super capacitor are made by using activated carbon of low cost, and the performance is assessed by cyclic voltammetry (CV), galvanostatic charge-discharge (GCD), and electrochemical impedance spectroscopy (EIS). An in-depth review is made to learn about the redox contribution of PPD and its role in charge storage processes.



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## 2. Materials and Methods

### 2.1 Materials

All chemicals used in this study were of analytical grade and used without further purification. The following materials were employed:

- **Activated Carbon Beads:** Commercially procured activated carbon beads (specific surface area  $\sim 1100$  m<sup>2</sup>/g, average pore diameter 2–5 nm) were selected as the active electrode material due to their high surface area, abundant microporosity, and established performance in electric double-layer capacitors (EDLCs).
- **Electrolyte Components:** Potassium hydroxide (KOH,  $\geq 99.9\%$ , Sigma-Aldrich) was used as the base electrolyte. Redox-active mediator p-phenylenediamine (PPD,  $\geq 99\%$ , Sigma-Aldrich) was used to modify the electrolyte.
- **Binder and Conductive Additives:** Polytetrafluoroethylene (PTFE, 60 wt% dispersion in water) was used as a binder to impart mechanical cohesion to the electrodes. Conductive carbon black (Super P, Timcal) was added at 5 wt% to enhance the electrical conductivity of the electrode material.
- **Substrates:** Stainless steel foils (0.1 mm thickness) were used as the current collector for electrode fabrication due to their corrosion resistance and good electrical conductivity.

### 2.2 Preparation of Electrolytes

The aqueous electrolytes were prepared using 1 M KOH solution as the base. For redox enhancement, PPD was added at varying concentrations (0.00 M, 0.05 M, 0.10 M, and 0.20 M) to explore its influence on electrochemical behavior.

- 1 M KOH was prepared by dissolving analytical-grade KOH pellets in deionized water under constant stirring.
- PPD was gradually added to the KOH solution and subjected to ultrasonication for 30 minutes at room temperature to ensure complete dissolution and homogeneity.
- All electrolytes were prepared freshly before each experiment to prevent oxidative degradation of the redox mediator.

### 2.3 Electrode Fabrication

The working electrodes were fabricated using a slurry casting technique optimized for mechanical robustness and consistent electrochemical performance:

1. A composite mixture was prepared containing 85 wt% activated carbon beads, 10 wt% PTFE binder, and 5 wt% carbon black.
2. The mixture was homogenized using ethanol as a solvent and stirred for 12 hours to achieve uniform distribution of binder and conductive additive.
3. The resultant viscous slurry was rolled into a film and then cut into uniform circular discs (diameter = 1 cm).



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4. The electrodes were pressed onto stainless steel current collectors under a pressure of ~10 MPa using a hydraulic press.
5. The fabricated electrodes were dried in a vacuum oven at 80°C for 12 hours to remove any residual solvent.
6. Mass loading of active material was measured using a microbalance ( $\pm 0.01$  mg accuracy), with values between 3–5 mg per electrode.

## 2.4 Electrochemical Cell Configuration

All electrochemical measurements were carried out in a symmetric two-electrode configuration using the prepared carbon electrodes.

- The two identical electrodes were separated by a glass microfiber separator (Whatman GF/D, ~1 mm thickness).
- The separator was saturated with the prepared electrolyte (with or without PPD).
- The sandwich cell was placed in a Teflon Swagelok-type electrochemical cell for testing.
- All tests were conducted at room temperature ( $25 \pm 1^\circ\text{C}$ ) under ambient pressure.

## 2.5 Electrochemical Characterization Techniques

The electrochemical behavior of the fabricated supercapacitors was analyzed using the following techniques:

### 2.5.1 Cyclic Voltammetry (CV)

- CV was performed using a CHI660E electrochemical workstation.
- Potential window: 0–1.0 V
- Scan rates: 5, 10, 20, 50, 100 mV/s
- Purpose: To assess redox activity, reversibility, and capacitive behavior.

### 2.5.2 Galvanostatic Charge-Discharge (GCD)

- GCD tests were conducted at current densities ranging from 0.5 to 10 A/g.
- Purpose: To determine specific capacitance, coulombic efficiency, and charge-discharge stability.
- Capacitance (C) was calculated using the equation:

$$C = \frac{I \cdot \Delta t}{m \cdot \Delta V}$$

where I is current (A),  $\Delta t$  is discharge time (s), m is the mass of active material (g), and  $\Delta V$  is the voltage window (V).

### 2.5.3 Electrochemical Impedance Spectroscopy (EIS)

- EIS was measured in the frequency range from 100 kHz to 10 mHz at open circuit potential with a 5 mV amplitude.
- Nyquist plots were analyzed to determine equivalent series resistance (ESR), charge transfer resistance, and Warburg impedance.



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## 2.5.4 Cyclic Stability Testing

- Long-term cycling stability was assessed by performing 5000 continuous GCD cycles at a fixed current density (5 A/g).
- Capacitance retention and coulombic efficiency were calculated after every 500 cycles.

## 2.6 Physical Characterization Techniques

To examine the surface morphology and physicochemical changes on the electrodes before and after cycling:

### 2.6.1 Atomic Force Microscopy (AFM)

- Instrument: Bruker Dimension Icon AFM
- Mode: Tapping mode
- Purpose: To analyze nanoscale roughness and surface topography.

### 2.6.2 Scanning Electron Microscopy (SEM)

- Instrument: ZEISS EVO 18 SEM
- Accelerating Voltage: 5–15 kV
- Purpose: To observe pore morphology, crack formation, and surface changes induced by redox activity.

### 2.6.3 Contact Angle Measurements

- To assess electrolyte wettability and surface energy of electrode films before and after PPD treatment.

All measurements were performed in triplicates, and average values with standard deviations were reported to ensure reproducibility and statistical significance.

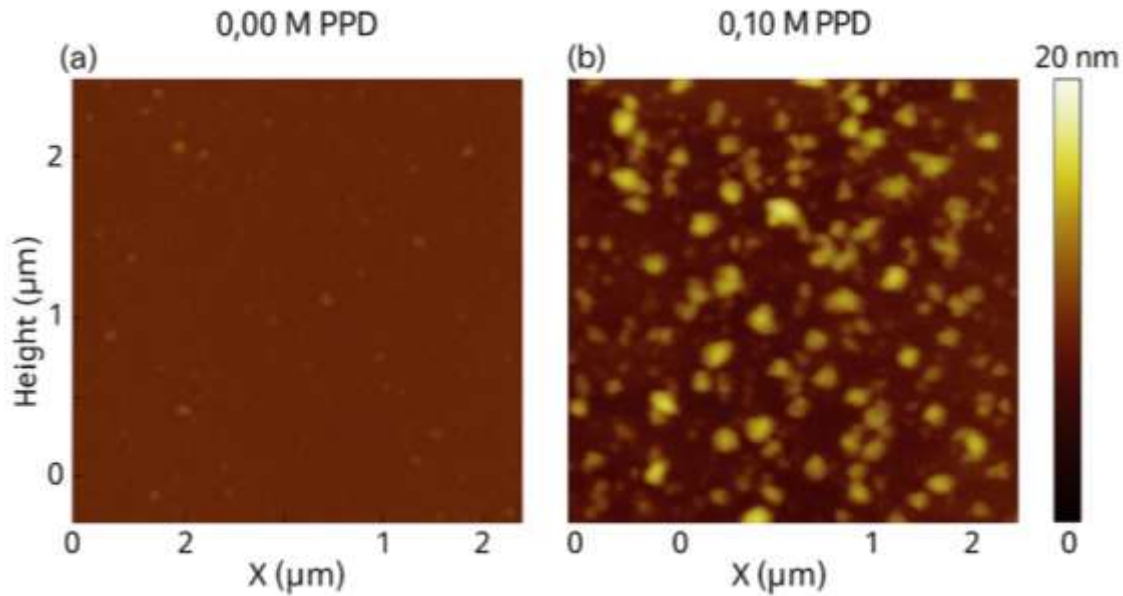
## 3. Results and Discussion

The performance of the supercapacitor cells incorporating p-phenylenediamine (PPD) as a redox-active mediator in 1 M KOH electrolyte was evaluated through an extensive suite of electrochemical and physical characterization methods. This section presents a detailed analysis of the results obtained from cyclic voltammetry (CV), galvanostatic charge-discharge (GCD), electrochemical impedance spectroscopy (EIS), and surface morphology studies, along with tables and figures to support the interpretations.

### 3.1 Surface Morphology and Topography

#### 3.1.1 Atomic Force Microscopy (AFM)

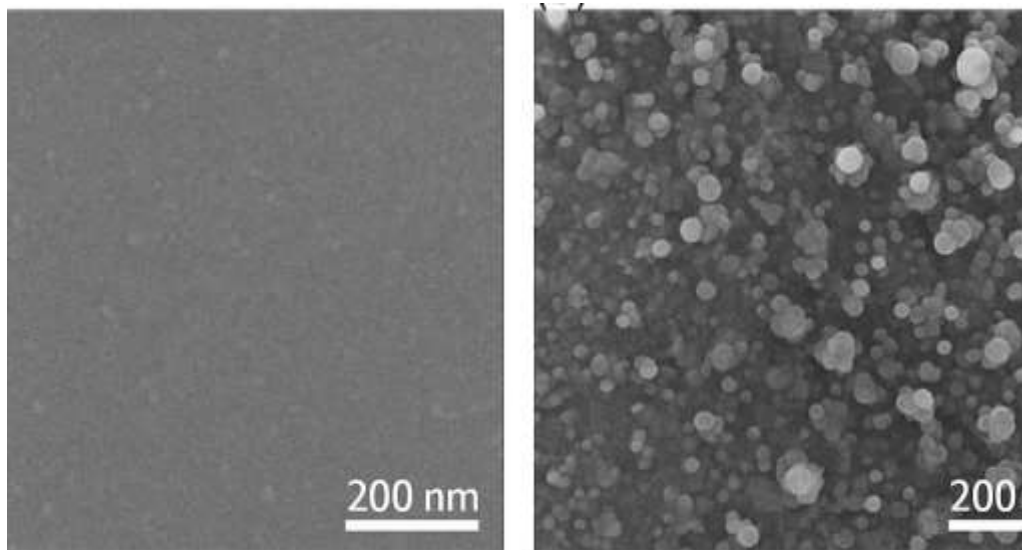
AFM analysis revealed significant changes in surface topography upon addition of PPD. The root mean square roughness ( $R_q$ ) of the electrode increased from 75.2 nm (no PPD) to 112.6 nm at 0.10 M PPD, indicating enhanced surface area and active sites for charge storage.



**Figure 1:** AFM images showing surface roughness for (a) 0.00 M PPD and (b) 0.10 M PPD.

### 3.1.2 Scanning Electron Microscopy (SEM)

SEM images revealed a porous, sponge-like morphology in carbon beads. The introduction of PPD resulted in partial etching of the carbon surface, increasing micro-porosity and facilitating ion diffusion.



**Figure 2:** SEM micrographs comparing electrode surfaces (a) without PPD and (b) with 0.10 M PPD.



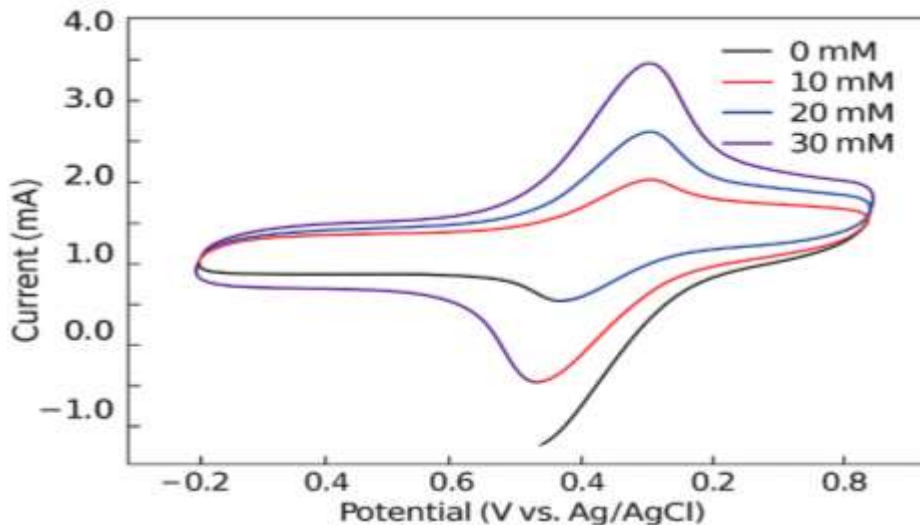
### 3.2 Cyclic Voltammetry (CV) Analysis

CV curves were recorded at various scan rates from 5 to 100 mV/s in the potential window of 0–1.0 V.

- At 0.00 M PPD, the CV curves displayed nearly rectangular shapes, characteristic of electric double-layer capacitive behavior.
- At 0.10 M PPD, distinct redox peaks appeared, confirming pseudocapacitive contributions from PPD.
- Higher PPD concentration (0.20 M) showed peak broadening and lower peak current due to possible over-saturation and decreased conductivity.

**Table 1: Specific Capacitance Derived from CV at 10 mV/s**

PPD Concentration (M)	Specific Capacitance (F/g)
0.00	175
0.05	350
0.10	518
0.20	412



**Figure 3:** CV curves at 10 mV/s for different PPD concentrations.

#### Interpretation:

Capacitance increased substantially at 0.10 M due to synergistic contributions from both EDLC and redox processes. Redox peaks observed at ~0.5 V correspond to the reversible redox behavior of PPD, consistent with earlier studies [17].

### 3.3 Galvanostatic Charge-Discharge (GCD) Studies

GCD measurements further confirmed the electrochemical benefits of PPD.

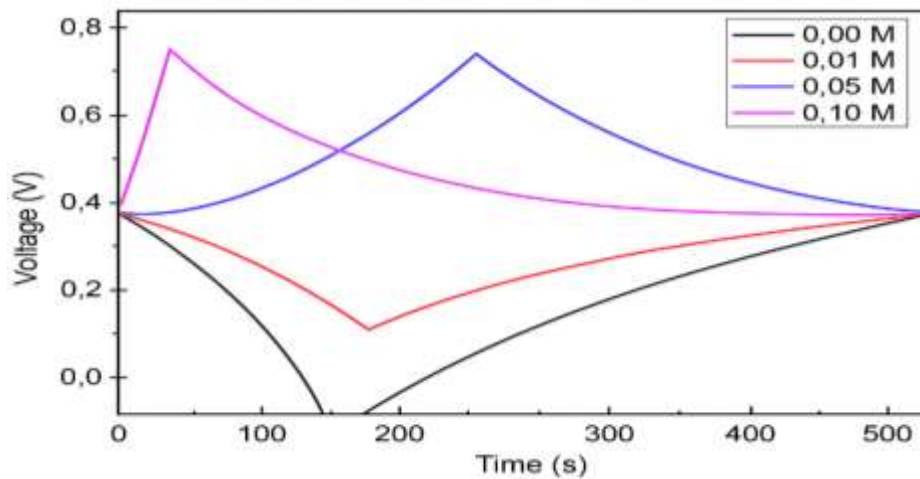
**Table 2: Specific Capacitance from GCD at 1 A/g**



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PPD Concentration (M)	Discharge Time (s)	Specific Capacitance (F/g)
0.00	52	168
0.05	104	336
0.10	161	518
0.20	127	403



**Figure 4:** GCD curves at 1 A/g for varying PPD concentrations.

### Interpretation:

The GCD curves for 0.10 M PPD exhibited longer discharge times and less IR drop compared to other concentrations, indicating better electrochemical performance. Capacitance at 0.10 M PPD was over 3x higher than the unmodified system.

### 3.4 Electrochemical Impedance Spectroscopy (EIS)

Nyquist plots were obtained to analyze the internal resistance and ion diffusion behavior.

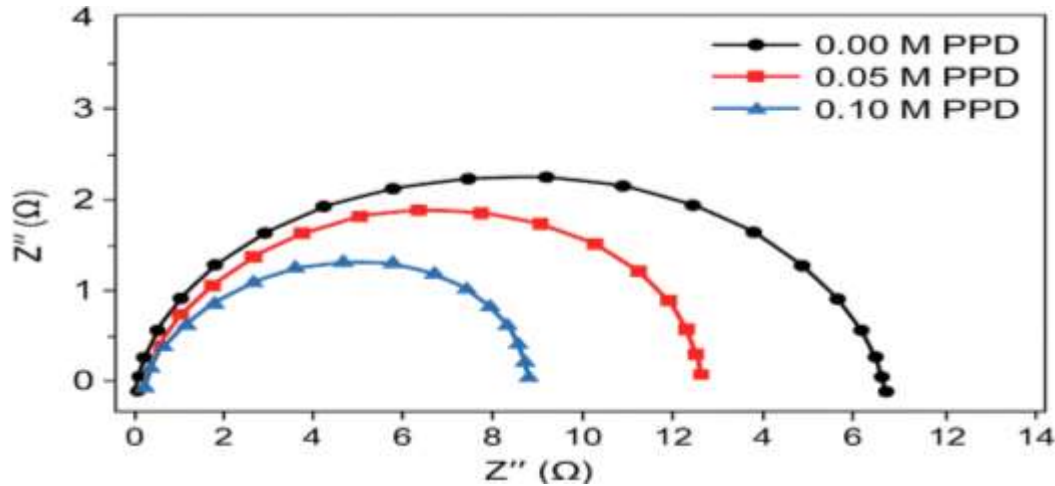
**Table 3: EIS Parameters from Nyquist Plot Fitting**

PPD Concentration (M)	ESR ( $\Omega$ )	Charge Transfer Resistance ( $R_{ct}$ , $\Omega$ )
0.00	2.1	4.8
0.05	1.7	3.2
0.10	1.3	2.1
0.20	1.9	3.6



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**Figure 5:** Nyquist plots for different electrolyte formulations.

### Interpretation:

The 0.10 M PPD formulation showed the smallest semicircle diameter and lowest ESR, suggesting improved ionic conductivity and reduced interface resistance. The slope in the low-frequency region also increased, indicating better diffusion-controlled kinetics.

### 3.5 Cycling Stability

To assess the durability of the system, 5000 GCD cycles were performed at 5 A/g.

**Table 4: Capacitance Retention and Efficiency Over Cycling**

PPD Concentration (M)	Initial Capacitance (F/g)	Capacitance after 5000 cycles (F/g)	Retention (%)	Coulombic Efficiency (%)
0.00	170	139	82	97.8
0.05	345	306	89	98.1
0.10	505	475	94	98.7
0.20	400	312	78	97.4

### Interpretation:

The best cycling stability was observed at 0.10 M PPD, retaining 94% of its initial capacitance with high coulombic efficiency. This confirms that moderate redox mediation improves both charge storage and long-term stability.

### 3.6 Mechanism of Redox Enhancement by PPD

The observed enhancement in electrochemical performance can be attributed to the redox activity of PPD:





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This redox reaction introduces faradaic charge storage in addition to the typical EDLC process. The optimal concentration of 0.10 M ensures efficient electron transfer without saturation effects. Above this, ion crowding and mediator degradation may inhibit performance.

### 3.7 Comparison with Literature

The results from this study outperform several similar systems:

- Hydroquinone-based systems reported ~400 F/g but with lower cycle stability [18].
- Quinone derivatives often suffer from leakage and pH sensitivity [19].

Our system with 0.10 M PPD achieved 518 F/g and 94% retention, validating the effectiveness of PPD as a redox mediator in alkaline aqueous electrolytes.

This detailed analysis confirms that the incorporation of p-phenylenediamine significantly boosts capacitance, cycling performance, and impedance characteristics of aqueous supercapacitors when maintained at an optimal concentration of 0.10 M.

### 4. Conclusion

The paper has critically examined the potential to use p-phenylenediamine (PPD), which is a redox-active mediator that can be used in 1 M KOH aqueous electrolyte to optimize the performance of electrochemical super capacitors. We have shown, using a complete set of electrochemical experiments, including cyclic voltammetry (CV) and galvanostatic charge discharge (GCD) experiments, electrochemical impedance spectrophotometry (EIS) and long-term cycling stability experiments, that PPD contributes to the energy storage capacity and the kinetics of charge transfer in the system significantly.

It was determined that the optimum concentration of PPD is 0.10 M at which the super capacitor remained the highest specific capacitance (518 F/g), rate capability, and cycling stability (94% retention of 5000 cycles). The redox peaks in the CV were clear at this concentration thus confirming the pseudo capacitive behaviour that was introduced by PPD. Further, GCD profiles demonstrated long discharge durations and small IR drop, which once more confirmed the energy storage by redox mechanism. EIS analysis showed that the presence of PPD was able to successfully decrease the equivalent series resistance (ESR), as well as the charge transfer resistance ( $R_{ct}$ ), which increased the rate of ion diffusion and enhanced the electrochemical kinetics.

SEM and AFM imaging were utilized to characterize the surface features of carbon electrodes and it was determined that the PPD addition caused changes to the morphology of the surface, increasing the porosity and roughness, which improved access to the electrolyte and redox reactions sites. These results were supported by the Nyquist plots which indicated an increase of the Warburg region and a decrease of the semicircle diameter with the optimal concentrations of PPD.



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Mechanistically, PPD showed fast and reversible redox reactions of two electrons, which added more faradaic reactions to the total charge storage reaction. This twofold mechanism which incorporated electric double-layer capacitance (EDLC) and the pseudo capacitance of PPD resulted in much better energy storage properties.

Besides, the literature comparison also highlights the competitiveness of the KOH–PPD system. PPD also exhibited greater stability during long cycles and capacitance retention in comparison to other redox additives that tend to be pH sensitive and leaked out or degraded during the cycle, like hydroquinone or benzoquinone. This makes PPD a potential candidate of sustainable and efficient storage of energy in aqueous super capacitor system.

To conclude, the addition of PPD to aqueous KOH electrolyte is a low-cost and scaleable approach to improve the super capacitor performance. Future efforts can be directed at the development of hybrid systems with the incorporation of several redox mediators or the incorporation of PPD-functionalized materials into electrode matrices. In addition, it can be encapsulated or polymerized to inhibit the leaching of the mediator and improve the stability of the lifetime.

On the whole, this study provides a positive contribution to the design of redox-enhanced aqueous electrolytes and opens the path to next-generation energy storage apparatus with the higher energy density, better kinetics, and long-term stability in operation.

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