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RESEARCH ARTICLE

Optimization of Polymerization Parameters for Enhanced Paracetamol Selectivity of Poly(1,4–diaminobenzene) Membrane Electrodes

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ABSTRACT: The electrochemical polymerization of 1,4-diaminobenzene in an aqueous KCl electrolyte at a controlled potential of 0.600 V resulted in the formation of an adherent poly(1,4-diaminobenzene) (PDB) film on a gold electrode. This polymeric membrane demonstrated selective permeability for paracetamol while effectively blocking ascorbic acid interference. A comprehensive investigation of polymerization parameters, including film thickness, monomer concentration, electrolyte concentration, and applied polymerization potential, was conducted to optimize the permselective characteristics of the PDB film. Voltammetric studies revealed that a polymeric film with a thickness corresponding to 7 mC charge exhibited the highest selectivity for paracetamol while completely suppressing ascorbic acid oxidation signals. Further analysis demonstrated that the optimal polymerization potential was 0.600 V vs. Ag/AgCl, where the peak oxidation current for paracetamol was maximized. Monomer and electrolyte concentrations were also optimized, with the best response obtained at 50 mM 1,4-diaminobenzene and 100 mM KCl. The findings suggest that the optimized PDB film functions as an effective permselective membrane, enabling highly sensitive and selective electrochemical detection of paracetamol. This approach offers a cost-effective and robust alternative to conventional sensors, addressing issues such as low sensitivity, poor stability, and the need for noble metal electrodes. The optimized polymer membrane holds potential for applications in pharmaceutical analysis and biomedical sensing, where precise and interference-free detection of paracetamol is essential.

Keywords: Paracetamol, Electrochemical polymerization, Poly(1,4-diaminobenzene), Selective membrane, Voltammetry, Interference suppression.

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1. INTRODUCTION

Paracetamol (PCT), also known as acetaminophen, is one of the most widely used oral analgesics and antipyretics. It is commonly prescribed for the relief of fever, headaches, and minor body aches and pains [1]. Due to its effectiveness and safety, PCT is extensively used for treating various conditions, including muscular aches, chronic pain, migraine headaches, and toothaches. Additionally, it is frequently administered to reduce fever and alleviate symptoms associated with the common cold, such as cough and body discomfort [2].

The pharmacological action of PCT is attributed to its ability to inhibit the production of prostaglandins in the central nervous system, which contributes to its analgesic effect. Its antipyretic property arises from its ability to modulate the thermoregulatory center in the hypothalamus [2]. Despite its widespread usage and safety profile, excessive intake of PCT or hypersensitivity reactions can lead to severe health complications, including hepatotoxicity, nephrotoxicity, and pancreatic disorders [3]. PCT overdose is one of the leading causes of acute liver failure worldwide, emphasizing the need for precise and reliable detection methods for monitoring its levels in pharmaceutical formulations and biological fluids.

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Several analytical techniques have been employed to determine PCT concentrations in various samples. Among them, high-performance liquid chromatography (HPLC) [4], spectrophotometric methods [5-10], capillary electrophoresis [11], amperometric biosensors [12], and electrochemical methods [13] have demonstrated significant utility. Among these, electrochemical techniques have gained prominence due to their inherent advantages, including high sensitivity, rapid response, simplicity, cost-effectiveness, and the potential for miniaturization in point-of-care diagnostics [14]. Electrochemical sensors offer a promising alternative for PCT detection, enabling real-time analysis with minimal sample preparation and lower reagent consumption compared to conventional chromatographic techniques.

A variety of electrochemical sensors have been developed for PCT determination using different electrode materials [15-20]. However, many of these sensors suffer from certain limitations, such as low sensitivity, poor stability, and reliance on expensive noble metals. This has driven researchers to explore alternative electrode materials that offer high performance while remaining cost-effective and readily available. The challenge lies in developing an electrochemical sensor for PCT detection that not only exhibits excellent sensitivity and stability but also utilizes low-cost electrode materials that can be fabricated with ease.

In recent years, polymer-modified electrodes have attracted considerable attention for their potential in enhancing electrochemical sensor performance. Conducting polymers provide a unique combination of electrical conductivity, chemical stability, and the ability to facilitate electron transfer processes, making them suitable candidates for sensor applications. Previous studies [21-23] have demonstrated that electrochemically synthesized films of conducting polymers such as poly(3-methylthiophene), polyaniline, polypyrrole, poly(o-toluidine), poly(1,3-phenylenediamine), and poly(o-phenylenediamine) can effectively differentiate PCT from interfering substances such as ascorbic acid. These studies highlight the potential of polymeric films in improving the selectivity and sensitivity of electrochemical sensors.

Among the various conducting polymers, poly(1,4-diaminobenzene) has emerged as a promising material for electrochemical sensing applications. This polymer is characterized by its ladder-like structure with phenazine rings, which imparts excellent stability and electron transport properties [24-26]. The ability of poly(1,4-diaminobenzene) films to immobilize enzymes, prevent electrode surface fouling, and enhance selectivity makes them highly suitable for use in electrochemical sensors. Additionally, this polymeric film has been successfully employed as a permselective membrane for halogenide ions, further underscoring its utility in sensor development.

Despite these promising attributes, the application of poly(1,4-diaminobenzene) films for PCT detection remains relatively unexplored. The present study aims to address this gap by investigating the electrochemical preparation, optimization, and voltammetric characteristics of poly(1,4-diaminobenzene) as a PCT-selective polymeric membrane. By leveraging the unique properties of this polymer, we aim to develop a sensor that offers enhanced sensitivity, stability, and selectivity for PCT detection. The findings of this study could contribute to the development of cost-effective and efficient electrochemical sensors for pharmaceutical and biomedical applications, ensuring accurate monitoring of PCT levels in various sample matrices.

2. EXPERIMENTAL DETAILS

2.1. Materials

1,4-Diaminobenzene was purchased from Merck and recrystallized from water before use to ensure high purity (melting point: 139°C-142°C). This purification step was necessary to remove any potential impurities that could interfere with polymerization or electrochemical performance. All other chemicals used, including paracetamol, ascorbic acid, and potassium chloride (KCl), were of analytical grade and obtained from either Sigma or Merck, ensuring consistency in experimental conditions. Aqueous solutions were prepared using double-distilled water to minimize any unintended ionic interferences.

To ensure the reliability of the electrochemical measurements, solutions of ascorbic acid and paracetamol were freshly prepared before each experiment to prevent oxidation or degradation. The monomer solutions were purged with high-purity nitrogen gas for approximately 10 minutes before initiating polymerization to remove dissolved oxygen, which could otherwise interfere with the electropolymerization process. During electropolymerization, the solution was continuously blanketed with nitrogen gas to maintain an oxygen-free environment, thereby enhancing polymer film quality and reproducibility. Unless otherwise specified, 0.1 M aqueous sodium sulfate (Na2SO4) solution was used as the supporting electrolyte in voltammetric experiments to provide a stable ionic medium for electrochemical processes.

2.2. Instrumentation

All electrochemical experiments were conducted using a BAS electrochemical analyzer, which allowed precise control and monitoring of the electrochemical processes. A conventional three-electrode electrochemical cell setup was employed, comprising a gold working electrode (geometric area: 1.98 mm²), an Ag/AgCl reference electrode (BAS, MF-2063), and a platinum wire coil auxiliary electrode. The use of a gold electrode was chosen due to its excellent conductivity, chemical stability, and suitability for polymer film deposition. The Ag/AgCl reference electrode ensured a stable reference potential, while the platinum wire coil served as an efficient counter electrode.

For cyclic voltammetry (CV) experiments, a scan rate of 50 mV/s was used as a standard parameter to ensure

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controlled electron transfer kinetics and reproducible electrochemical behavior. The electrochemical setup was optimized to minimize background noise and enhance signal resolution, ensuring accurate characterization of the polymeric films and their interaction with paracetamol and ascorbic acid.

2.3. Preparation of Poly(1,4-diaminobenzene) Film

Before electropolymerization, the gold disc electrodes used as working electrodes were subjected to a rigorous cleaning procedure to remove any contaminants that could affect polymer film adhesion and electrochemical performance. The cleaning process followed a standard protocol [33], which involved sequential mechanical polishing with diamond polishing compounds and aqueous alumina slurry (Johnson Matthey Catalog Comp., USA) down to a final particle size of 0.5 µm. This polishing step was critical in achieving a smooth and uniform electrode surface, facilitating consistent polymer film formation.

Electropolymerization of poly(1,4-diaminobenzene) was carried out in a deaerated aqueous solution containing 1,4-diaminobenzene as the monomer and KCl as the supporting electrolyte. The use of KCl ensured adequate ionic conductivity and stable polymer film deposition. The polymerization process involved applying a controlled potential to induce the oxidative polymerization of 1,4-diaminobenzene, leading to the formation of a polymeric film on the gold electrode surface. Following polymerization, the resulting poly(1,4-diaminobenzene) films were thoroughly rinsed with deionized water to remove any unreacted monomer, supporting electrolyte residues, or loosely adhered polymeric material. Visual inspection of the electrode surface confirmed the formation of a thin, homogeneous polymeric film with a characteristic brownish color, indicating successful polymer deposition. The uniformity and adhesion of the polymeric film were essential for ensuring consistent electrochemical performance in subsequent voltammetric studies.

To optimize the polymerization parameters, differential pulse voltammetry (DPV) measurements were conducted in 0.1 M aqueous Na2SO4 solution (pH 6.0) containing 10 mM paracetamol or ascorbic acid. DPV was chosen for optimization due to its high sensitivity and ability to differentiate between closely spaced redox peaks, allowing for precise evaluation of the polymer film's selectivity and electrochemical behavior. The influence of polymerization conditions, such as monomer concentration, applied potential, and polymerization time, was systematically investigated to achieve optimal film properties for selective paracetamol detection.

3. RESULTS AND DISCUSSION

3.1. Cyclic Voltammogram of Poly (1,4-diaminobenzene) Film

Cyclic voltammetry (CV) was employed to investigate the electrochemical polymerization of 1,4-diaminobenzene on a gold electrode in 0.1 M KCl solution. Figure 1 displays the cyclic voltammograms recorded in the absence and presence of the monomer. A distinct oxidation process was observed, characterized by irreversible oxidation peaks at 0.26 V and 0.61 V. The occurrence of these peaks indicates the electrochemical oxidation of the 1,4-diaminobenzene monomer, initiating the polymerization process.

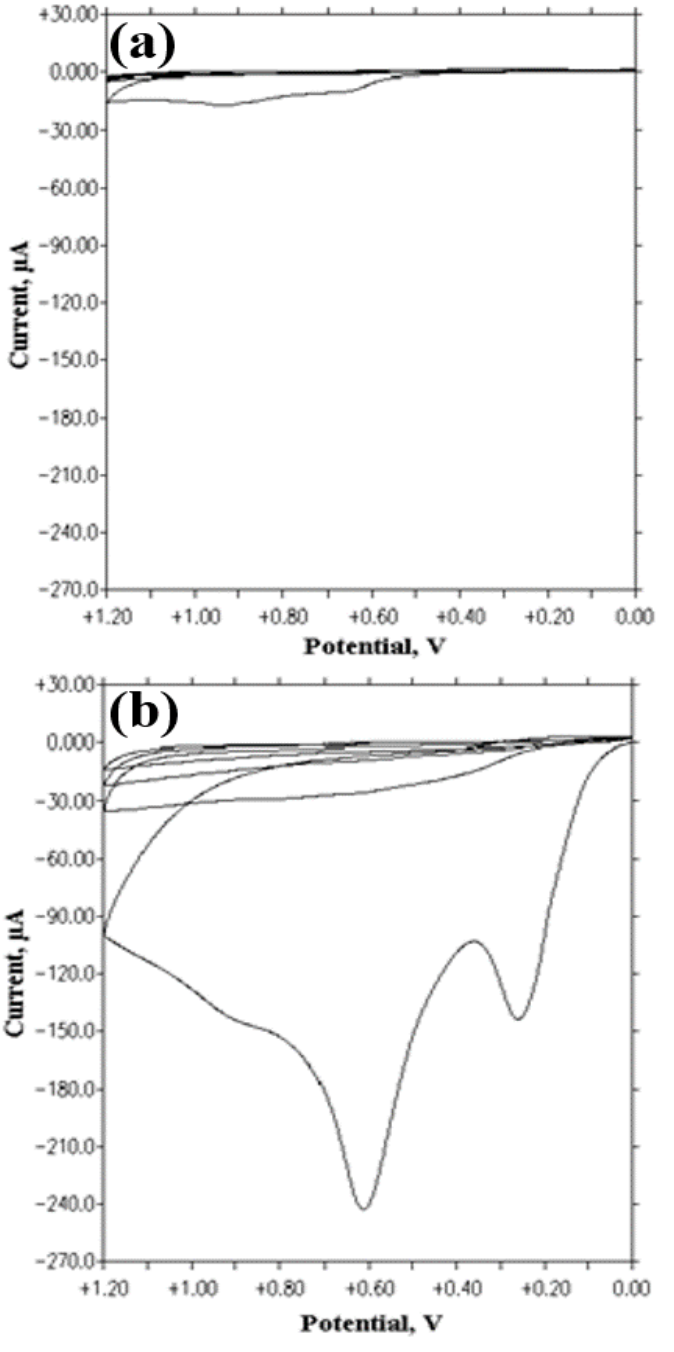


Fig. 1. Cyclic voltammograms obtained with a bare Au electrode in 0.1 M KCl (A), and in 0.1 M KCl + 50 mM 1,4-diaminobenzene (B). Scan rate: 50 mV/s.

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As electropolymerization progressed, the oxidation currents diminished in subsequent cycles, suggesting that the poly(1,4-diaminobenzene) film hindered further monomer oxidation. The formation of a stable, thin, and insoluble polymeric film was confirmed by its persistence on the electrode surface even after extensive cycling. The optimized polymerization process was achieved by holding the potential at 0.600 V for approximately 45 seconds, yielding a well-adhered film with desirable electrochemical properties.

3.2. The Effects of Film Thickness

The electrochemical performance of the polymeric film was significantly influenced by its thickness, which was controlled by adjusting the charge consumed during polymerization. Figure 2 illustrates the relationship between film thickness and peak current response for ascorbic acid and paracetamol. As the film thickness increased, the oxidation current for ascorbic acid exhibited a continuous decline, ultimately reaching negligible levels beyond a charge of 7 mC. This trend suggests that thicker films provide a greater barrier to ascorbic acid penetration, effectively preventing its oxidation at the electrode surface. Conversely, the response to paracetamol initially increased with film thickness, reaching a peak at 7 mC, beyond which further increases in thickness resulted in reduced current response.

The observed behavior can be attributed to the selective permeability of the polymeric film, which acts as a molecular sieve, favoring the diffusion of paracetamol while suppressing the oxidation of ascorbic acid. The optimal film thickness of 7 mC was selected for subsequent experiments, ensuring maximum sensitivity for paracetamol detection while eliminating interference from ascorbic acid.

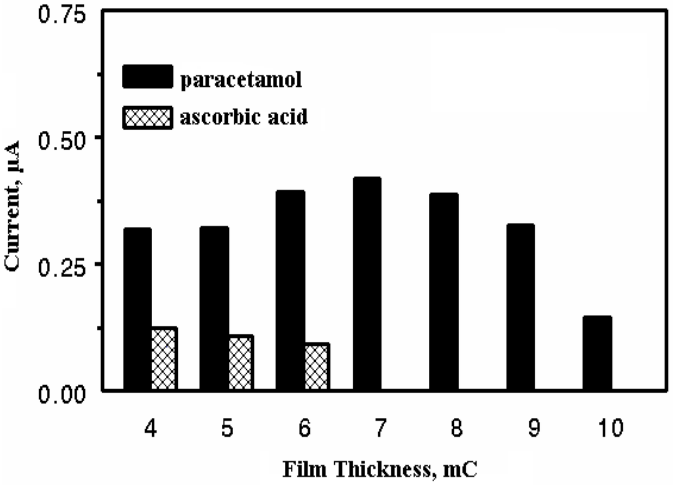


Fig. 2. Effect of film thickness on the response.

3.3. The Effect of Polymerization Potential on the Response to Paracetamol

To determine the optimal polymerization potential, films were prepared at varying potentials ranging from 0.4 V to 0.8 V, and their electrochemical responses to paracetamol were analyzed (Figure 3). The peak current exhibited a gradual increase with increasing polymerization potential, reaching a maximum at approximately 0.600 V, beyond which further increases led to a decline in response.

The observed trend suggests that polymerization at 0.600 V produces films with optimal electrochemical activity, ensuring sufficient conductivity while maintaining selectivity for paracetamol. Lower potentials may result in incomplete polymerization, yielding less stable films, whereas excessive potentials could lead to over-oxidation, reducing film conductivity and impeding analyte diffusion. Thus, 0.600 V was identified as the ideal polymerization potential for subsequent studies.

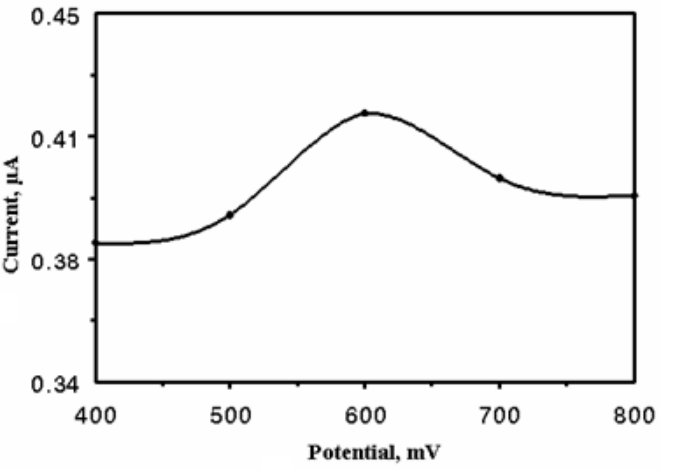


Fig. 3. Effect of polymerization potential on the response.

3.4. Effects of Monomer and Electrolyte Concentrations

The concentration of 1,4-diaminobenzene in the electropolymerization solution played a crucial role in determining the film properties. As shown in Figure 4, the peak current response for paracetamol increased with increasing monomer concentration, reaching a plateau at 50 mM. Higher concentrations led to faster polymerization, reducing the overall deposition time while ensuring uniform film formation.

Similarly, the choice of supporting electrolyte significantly influenced polymerization efficiency and film stability. As depicted in Figure 5, the highest paracetamol response was obtained using 100 mM KCl, indicating its suitability as a supporting electrolyte. Additionally, the effect of different electrolytes on the voltammetric response of the polymer electrode was examined, including Na2SO4, NaNO3, NaCl, LiCl, Mg(NO3)2, CaCl2, and NaClO4. Among these, Na2SO4 provided the highest peak current response, suggesting superior ionic conductivity and electrochemical stability under the experimental conditions.

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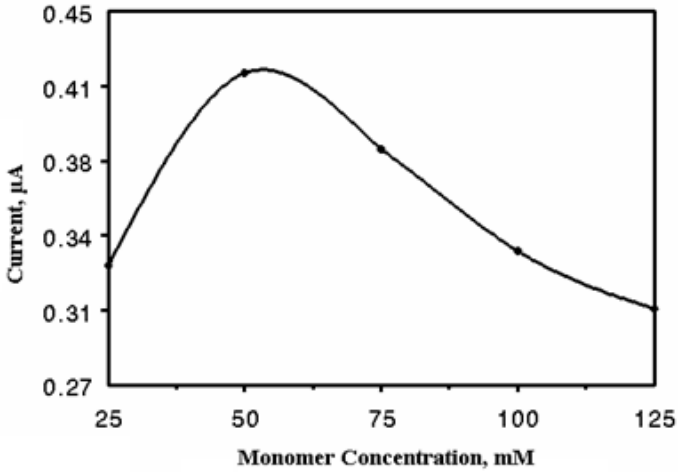


Fig. 4. Effect of 1,4-diaminobenzene concentration on the response.

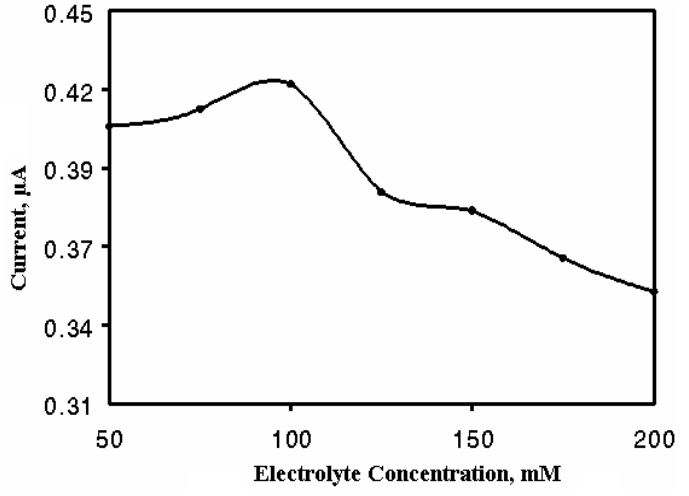


Fig. 5. Effect of KCl concentration on the response.

3.5. Voltammetry Characteristics of the Optimized Poly(1,4-diaminobenzene) Films

Differential pulse voltammetry (DPV) was employed to compare the electrochemical behavior of ascorbic acid at both bare and poly(1,4-diaminobenzene)-modified gold electrodes (Figure 6). At the bare electrode, ascorbic acid exhibited a distinct oxidation peak at approximately 0.40 V. However, at the polymer-modified electrode, this peak was completely suppressed, confirming the film's effectiveness in preventing ascorbic acid oxidation.

Conversely, as demonstrated in Figure 7, the polymer electrode permitted the oxidation of paracetamol, even in the presence of ascorbic acid. A series of DPV scans were conducted at increasing paracetamol concentrations (10–50 mM), revealing a linear increase in peak current response. This selective permeability highlights the film's ability to differentiate between paracetamol and ascorbic acid, allowing for accurate and interference-free detection of the target analyte.

Additionally, repeated voltammetric measurements in a binary mixture of paracetamol and ascorbic acid confirmed the stability of the polymer film. The observed reproducibility in voltammetric responses indicates that the film maintains its structural and electrochemical integrity over successive runs, further validating its suitability for analytical applications.

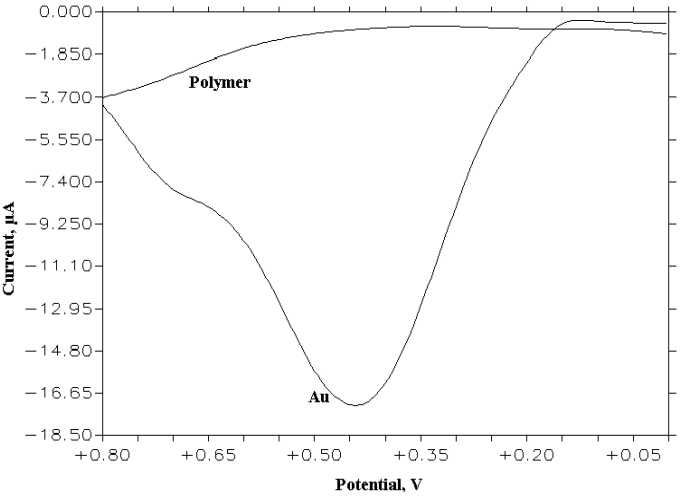


Fig. 6. DPVs of 10 mM ascorbic acid at the bare and polymer electrodes.

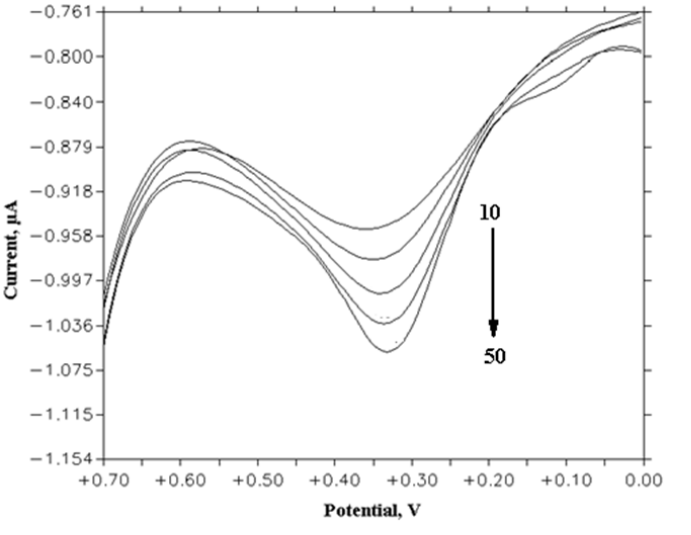


Fig. 7. DPV of 10 mM ascorbic acid at various concentrations of paracetamol (10–50 mM).

3.6. Mechanistic Insights into Film Selectivity and Performance

The selective exclusion of ascorbic acid and preferential oxidation of paracetamol can be attributed to the physicochemical properties of the poly(1,4-diaminobenzene)

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film. The polymer matrix likely possesses inherent electrostatic interactions and steric hindrance effects that influence analyte diffusion. The molecular size and charge characteristics of ascorbic acid may render it less compatible with the polymer's microstructure, while paracetamol, being structurally distinct, diffuses more readily.

Furthermore, the optimized film thickness and polymerization conditions contribute to an enhanced sensing performance, ensuring maximum signal intensity for paracetamol while suppressing background interference. The results suggest that poly(1,4-diaminobenzene) films can serve as effective electrode modifiers for selective electrochemical sensing applications.

The findings from this study have significant implications for the development of electrochemical sensors for pharmaceutical and biomedical applications. The ability to selectively detect paracetamol in the presence of interfering substances such as ascorbic acid underscores the potential of poly(1,4-diaminobenzene)-modified electrodes in complex sample matrices.

This study demonstrates the successful electropolymerization of 1,4-diaminobenzene on a gold electrode, yielding a stable, selective, and electroactive polymer film. The optimized film effectively suppresses ascorbic acid oxidation while permitting the detection of paracetamol, showcasing its potential for electrochemical sensing applications. The results highlight the importance of controlling polymerization conditions, including film thickness, polymerization potential, and electrolyte composition, to achieve optimal sensor performance. The findings pave the way for further advancements in polymer-based electrochemical sensors for pharmaceutical and clinical diagnostics.

4. CONCLUSION

In this study, we successfully developed and optimized a poly(1,4-diaminobenzene) (PDB) film for selective electrochemical detection of paracetamol. Through systematic evaluation of key polymerization parameters, we identified the optimal conditions for achieving a highly permselective membrane electrode. The findings demonstrated that a film thickness of 7 mC provided the best selectivity, effectively allowing paracetamol detection while preventing interference from ascorbic acid. Additionally, the ideal polymerization potential was determined to be 0.600 V, maximizing the oxidation peak current of paracetamol while maintaining membrane stability. The influence of monomer and electrolyte concentrations was also investigated, with the highest response achieved using 50 mM 1,4-diaminobenzene and 100 mM KCl. These conditions facilitated the formation of a uniform, adherent polymeric film, ensuring enhanced sensitivity and reproducibility in electrochemical measurements. Compared to conventional electrochemical sensors, the optimized PDB membrane electrode offers superior selectivity, stability, and cost-effectiveness without requiring noble metal modifications. This study underscores the potential of PDB-based membranes in pharmaceutical and biomedical applications, where accurate and interference-free paracetamol detection is crucial. The method's simplicity, reproducibility, and efficiency make it an attractive alternative to existing analytical techniques, such as high-performance liquid chromatography and spectrophotometry. Future research should explore the long-term stability of the PDB film, its applicability in real biological samples, and potential enhancements through nanomaterial incorporation. The optimized poly(1,4-diaminobenzene) film represents a promising step toward the development of reliable, high-performance electrochemical sensors for pharmaceutical and clinical diagnostics.

DECLARATIONS

Ethical Approval

We affirm that this manuscript is an original work, has not been previously published, and is not currently under consideration for publication in any other journal or conference proceedings. All authors have reviewed and approved the manuscript, and the order of authorship has been mutually agreed upon.

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Not applicable

Availability of data and material

All of the data obtained or analyzed during this study is included in the report that was submitted.

Conflicts of Interest

The authors declare that they have no financial or personal interests that could have influenced the research and findings presented in this paper. The authors alone are responsible for the content and writing of this article.

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