

Fabrication of a PtNPs Modified Platinum Electrode for Voltammetry Detection of Fenamiphos

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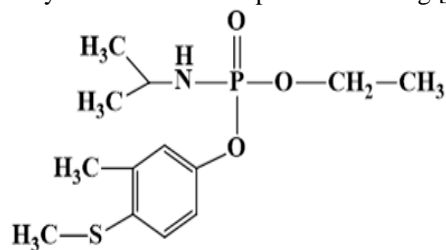
Abstract—Organophosphorus pesticides are ubiquitous environmental contaminants that cause adverse effects on human health and ecosystems, underscoring the critical need for efficient detection and remediation strategies. Safety monitoring intended to regulate these emerging hazards demands on-site sensing devices with easy fabrication and low detection limits. Electrochemical techniques meet these requirements owing to their inherent advantages, including rapid response, high analytical sensitivity, operational simplicity, cost-effectiveness, and compatibility with miniaturized and portable platforms. Therefore, an electrochemical sensor modified with platinum nanoparticles was developed for the determination of fenamiphos. Electrochemical deposition of platinum nanoparticles on a platinum electrode was performed via Chronoamperometry. The modified electrode was employed for cyclic voltammetric and electrochemical impedance spectroscopic analysis of fenamiphos.

Index Terms—Fenamiphos, Organophosphorus Pesticides, Insecticide, Sensor, Electrochemical Impedance Spectroscopy, Platinum nanoparticles

I. INTRODUCTION

Agrochemicals, namely organophosphorus pesticides and synthetic fertilizers, have played a significant role in enhancing agricultural productivity over the past few decades [1]. Due to their effectiveness in controlling crop pests, organophosphorus pesticides act as a key factor in maximizing crop yield and minimizing manual labour requirements [2]. In contrast, its misuse can lead to the accumulation of organophosphorus pesticide residues in agricultural products, posing a risk to food safety and potentially endangering human health [3]. Fenamiphos (FEN) is a sulphur-containing organophosphorus insecticide (figure 1) recognized for its high toxicity and systemic insecticidal activity, exerting its effects through direct contact with target pests [4]. Fenamiphos belongs to

the phosphoramidate class of organophosphate compounds [5]. It is commonly applied to crops such as bananas, citrus fruits, vegetables, potatoes, and ornamental plants to protect roots from nematode infestation and improve crop yield [6]. This insecticide exerts its toxic effect by blocking the activity of acetylcholinesterase, an enzyme pivotal for efficient neural communication in insects and nematodes [7]. Obstructing this enzyme results in the elevated levels of acetylcholine within the nervous system, hinders nerve function, and eventually resulting in paralysis and subsequent death of the target organism [8]. Humans are similarly exposed by FNP via inhalation, dermal contact, and dietary intake of contaminated food [9]. Because of its toxic nature and ecological concerns, a sensitive, rapid analytical method for fenamiphos detection is essential [10]. Multiple methodologies are available for the detection of fenamiphos, including chromatography [11] and spectrophotometric [12]. However, these methods often require expensive instrumentation, extensive sample preparation, and trained personnel [13]. As a result, electrochemical sensing approaches have emerged as promising alternatives due to their simplicity, rapid response, cost-effectiveness, and high sensitivity for on-site fenamiphos monitoring [14].



Ethyl (3-methyl-4-(methylthio)phenyl) isopropylphosphoramidate
or
Fenamiphos

Figure 1 Structure of Fenamiphos

Contemporary research has demonstrated the determination using electroanalytical techniques. R.F. Franca et al. [15] reported an electroanalytical detection of fenamiphos in natural waters using a boron-doped diamond electrode. T. Lima et al. [16] also designed a sensor based on a boron-doped diamond electrode for simultaneous detection of fenamiphos with carbendazin. B. Qader [17] and a co-worker in 2019 employed a glassy carbon electrode for detecting the organophosphate insecticide fenamiphos (FNP) and its primary metabolite, fenamiphos sulfoxide (FNX). P. Hashemi et al. [18] developed a sensor utilizing a reduced graphene oxide-Cu/CuO-Ag nanocomposite modified glassy carbon electrode (rGO/Cu/CuO-Ag/GCE) for simultaneous determination of fenamiphos and carbaryl. A. Gevaerd and co-worker [19] also employed reduced graphene oxide for electrode modification. Graphene oxide was electrochemically reduced to fabricate screen printed electrode for detection of fenamiphos in tomato samples. B. Qader et al. [20] designed a molecularly imprinted sensor employing computational approaches, including Density Functional Theory (DFT) and semi-empirical PM3 calculations for fenamiphos sulfoxide. H. Karimi-Maleh [21] and co-worker similarly fabricated a molecularly imprinted electrode with Co_3O_4 nanowires and $\text{Co}_3\text{O}_4@\text{MOF-74}$ core-shell nanocomposite materials for fenamiphos analysis. L. Codogno et al. [22] developed a boron-doped diamond electrode (BDD) combined with factorial design for the simultaneous and selective detection of bentazon (BTZ) and fenamiphos (FNP). Yamuna [9] and co-worker developed an electrochemical sensor based on a composite material consisting of samarium stannate ($\text{Sm}_2\text{Sn}_2\text{O}_7$) combined with laser-induced graphene (LIG) for fenamiphos analysis in environmental and food samples. H. S. Granja et al. [23] designed a Carbon paste electrode incorporated with grape seed biochar (E-bSU) for detection of fenamiphos.

According to recent research, metal nanoparticle modified electrodes have attracted considerable attention in recent years because of their exceptional properties and wide-ranging applications in biosensor development, drug delivery systems, and energy storage technologies [24-25]. Herein, in this work, we have developed an electrode modified with platinum nanoparticles for the voltammetry detection of fenamiphos.

II. CHEMICALS AND INSTRUMENTATION

Chemicals

Fenamiphos used in the experiment was obtained from Sigma-Aldrich. Additional reagents employed in this study included phosphate-buffer saline (PBS), sulphuric acid, potassium chloride, and sodium borohydride.

Instrumentation

Cyclic Voltammetry and Electrochemical impedance spectroscopy analysis was performed using a Multi Autolab M204 electrochemical workstation operated through NOVA 2.1 software. The electrochemical evaluations were carried out in a typical three-electrode arrangement consisting of the modified working electrode, a saturated calomel electrode (SCE) as the reference electrode, and a platinum sheet as the counter electrode. Surface characterization of the electrode was conducted with a ZEISS EVO 18 scanning electron microscope (SEM).

III. EXPERIMENTAL

Fabrication of Platinum nanoparticle

A platinum wire embedded in a soft glass served as the working electrode for all the electrochemical experiments. Before experimental measurements, the platinum electrode surface was manually polished via emery paper and then soaked in ethanol for 48 hr. The abraded electrode was then ultrasonicated in ethanol and subsequently subjected to sonication in triple-distilled water. Afterwards, the electrode was boiled in nitric acid and again ultrasonicated with triple-distilled water for cleaning. After electrochemical cleaning, platinum nanoparticles were deposited onto the electrode surface utilizing a solution containing 5 mM $\text{H}_2[\text{Pt}(\text{OH})_6]$ and 0.5 M H_2SO_4 . The nanoparticle deposition was carried out at a constant potential of 170 mV for 60 min.

IV. SURFACE MORPHOLOGY INVESTIGATION

The surface morphology of the PtNPs-modified platinum electrode was examined using scanning electron microscopy (SEM), and the corresponding image is presented below in Figure 2.

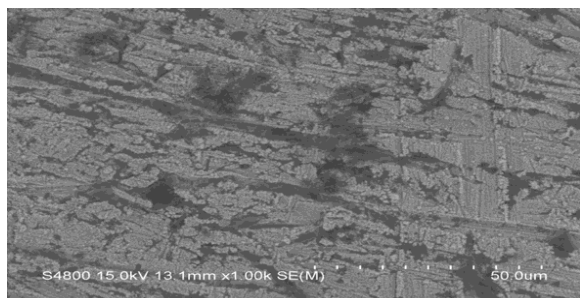


Figure 2 SEM image of Platinum nanoparticle deposited platinum electrode

V. RESULTS AND DISCUSSION

Electrochemical investigation through Cyclic Voltammetry

The effect of electrode modification on the electrochemical behaviour of fenamiphos (FNP) was systematically investigated using cyclic voltammetry (CV). CV studies were performed in phosphate-buffered saline (PBS, pH 6) containing 10^{-6} M fenamiphos. Electrochemical measurements were conducted using a bare platinum electrode and a PtNPs-modified platinum electrode. The voltammogram revealed a distinct oxidation peak for fenamiphos at 1.20 V vs. SCE on a PtNPs-modified platinum electrode. In comparison, the bare platinum electrode displayed a weak and less defined oxidation signal, suggesting sluggish electron transfer kinetics at the unmodified surface.

An increase in current response was observed at the modified electrode, highlighting its improved electrocatalytic activity toward fenamiphos detection. The highest current response obtained with this modified electrode can be attributed to enhanced electron transfer efficiency and improved surface interactions at the electrode interface.

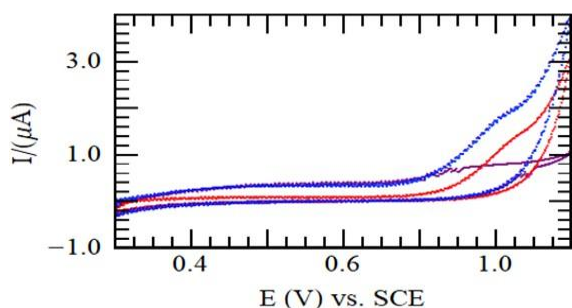


Figure 3 Cyclic voltammograms obtained in PBS (pH 6) showing the response in the absence of fenamiphos (purple) and in the presence of 10^{-6} M fenamiphos at

the bare electrode (red) and PtNPs-modified platinum electrode (blue).

VI. IMPEDANCE-BASED ELECTROCHEMICAL STUDY OF FENAMIPHOS

Electrochemical impedance spectroscopy (EIS) inspection was accomplished by applying a 1.2V DC perturbation relative to the reference electrode over a frequency range spanning 0.10 Hz-100 kHz. Experimental impedance results were analysed using Nova software and fitted with the widely used Randles equivalent circuit. This circuit includes resistive, capacitive, and diffusion-related components. In this model, R_s corresponds to the resistance of the electrolyte solution containing the redox species, while the constant phase element (CPE) reflects the capacitive behavior of the modified interface. The charge transfer resistance (R_{ct}) represents the resistance to electron movement across the electrode surface, indicating the ease or difficulty of electron exchange between the redox probe in solution and the electrode. Additionally, the Warburg impedance (Z_w) accounts for the diffusion process of electroactive species from the bulk solution to the electrode surface through the modified layer.

The Nyquist plot analysis revealed that the charge transfer resistance (R_{ct}) decreased from 650 Ω for the bare platinum electrode to 500 Ω for the platinum nanoparticle-modified platinum electrode. The lower R_{ct} value observed for the modified electrode indicates enhanced electron-transfer kinetics at the electrode-electrolyte interface, demonstrating the beneficial effect of platinum nanoparticle modification on electrochemical performance.

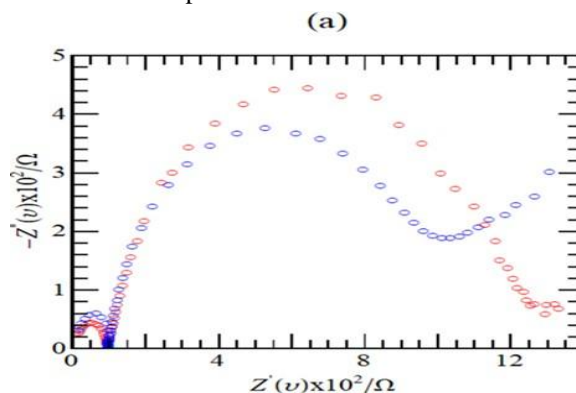


Figure 4 Nyquist plots recorded for the bare electrode (red) and PtNPs-modified electrode (blue) at 0.4 V over the frequency range of 0.10 Hz to 100 kHz.

VII. SUMMARY AND CONCLUSION

In this investigation, a sensor was fabricated to detect fenamiphos using cyclic voltammetry. Given the results of the study, platinum nanoparticles proved to increase the sensitivity of the electrode. The incorporation of nanoparticles significantly increased the effective surface area and conductivity of the electrode, resulting in amplified current responses and lower detection limits compared to the unmodified electrode. Owing to its simplicity, cost-effectiveness, and potential for miniaturization, the proposed nanoparticle-modified electrode system represents a promising platform for sensitive and reliable monitoring of fenamiphos in environmental and agricultural samples.

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