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DEVELOPMENT OF A SiC/CDC-CS COMPOSITE MODIFIED SCREEN PRINTED ELECTROCHEMICAL SENSOR FOR THE SENSITIVE VOLTAMMETRIC DETERMINATION OF METHYL PARATHION

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ABSTRACT

An electrochemical sensor based on a screen-printed electrode modified with a SiC/CDC-CS composite was developed for the sensitive and rapid determination of methyl parathion. The composite was prepared using a solution-assisted dispersion method and deposited onto the SPE working surface by drop casting. Material morphology was investigated by scanning electron microscopy, while the electrochemical properties of the electrodes were evaluated using cyclic voltammetry and electrochemical impedance spectroscopy. SEM images confirmed the distribution of small CDC particles over spherical SiC particles and the formation of a heterogeneous composite structure. Compared with the bare SPE, the SiC/CDC-CS/SPE exhibited higher redox currents and lower charge-transfer resistance. Methyl parathion was quantified using square-wave voltammetry. Chitosan had no substantial influence on the analytical signal and primarily acted as a film-forming binder that stabilized the nanomaterials on the electrode surface. The maximum electrochemical response was obtained at pH 6.0 with an accumulation time of 6 min. The sensor provided a linear concentration range of $1 \times 10^{-9} \sim 1 \times 10^{-5}$ g mL⁻¹ and a detection limit of 5×10^{-9} g mL⁻¹. The electrode process was characterized as an adsorption-controlled, proton-coupled electron-transfer reaction. The sensor demonstrated good selectivity and stability and was successfully applied to real water samples, providing recoveries of 100.42-103.30%. These results highlight the potential of the SiC/CDC-CS/SPE platform for environmental monitoring.

Keywords: Methyl parathion; Electrochemical sensor; Silicon carbide; Coke-derived carbon; Chitosan; Screen-printed electrode.

1 INTRODUCTION

In recent decades, the quest to increase crop yields has led to a sharp rise in pesticide use worldwide, with organophosphate compounds accounting for a significant portion of these chemicals. Despite their effectiveness and low cost, studies show that less than 0.1% of the pesticides used actually reach the target pests, while the vast majority is dispersed into the soil, water, and air, where they persist and accumulate in the bodies of living organisms [1,2]. Methyl parathion (MP), a highly effective organophosphate insecticide is a prime example of this dilemma: its ability to irreversibly inhibit acetylcholinesterase makes it exceptionally lethal to insects, but it also poses a serious threat of neurotoxic effects and endocrine disruption to wildlife and humans, even in small quantities [3]. Although many countries have banned or strictly restricted its use, MP is still used in some regions due to its high effectiveness against insects and its affordable price. To protect public health, the U.S. Environmental Protection Agency (EPA) has set a lifetime recommended limit for MP in drinking water at 2 mg L⁻¹ and established maximum residue limits in food ranging from 0.1 to 1 mg L⁻¹ [4,5]. The widespread use of MP in the environment and the high neurotoxicity of its residual amounts underscore the urgent and immediate need for reliable analytical methods to monitor their levels in water and food products in order to protect public health and the integrity of the environment.

At present, methyl parathion (MP) is commonly quantified using advanced chromatographic and mass-spectrometric techniques, including gas chromatography–mass spectrometry (GC–MS) [6,7], high-performance liquid chromatography (HPLC) [8,9], and tandem mass spectrometry (MS) [10,11]. These conventional laboratory methods offer excellent accuracy, reproducibility, and

detection limits reaching the nanogram level. Nevertheless, their practical application is constrained by high resource requirements. Analytical procedures typically rely on bulky benchtop instruments, strictly controlled laboratory conditions, and extensive sample preparation, including solid-phase extraction, derivatization, and multistage purification. The consumption of organic solvents and the prolonged extraction and cleanup procedures may extend the overall analysis time to several hours, thereby limiting sample throughput. In addition, the acquisition, calibration, and routine maintenance of such instruments involve substantial financial costs and require highly trained personnel. Consequently, these techniques are poorly suited to decentralized and on-site monitoring applications, where rapid identification of pesticide contamination and immediate analytical feedback are essential [12].

Electrochemical sensors have attracted significant attention due to their fast response, compact size, low power consumption, and the ability to perform analysis directly at the sampling site. They enable the detection of target compounds with high sensitivity and selectivity, without requiring complex or time-consuming sample preparation [13]. Among such analytical devices, electrodes manufactured using screen printing (SPE) occupy a special place. On a single miniature substrate, they combine a working electrode, a reference electrode, and a counter electrode. This scalable printing technology enables the mass production of disposable sensor platforms, helps reduce the cost of analysis and reagent consumption, and minimizes the risk of cross-contamination of samples. Furthermore, the ability to organize production using “roll-to-roll” technology allows for the mass production of ready-to-use sensors. Thanks to these combined advantages, SPE platforms are a promising tool for rapid analysis in field settings and at medical care facilities, where results must be obtained on-site and as quickly as possible [14]. To overcome these limitations, various improvement strategies have been developed, including the modification of the working electrode using nanomaterials and the application of electrochemical pretreatment to activate the carbon surface [15]. Currently, there is a trend toward using multifunctional nanocomposite coatings that simultaneously enhance conductivity, catalytic activity, and analyte adsorption, enabling the non-enzymatic determination of pesticides at concentrations below the nanomolar level.

Petroleum coke (PC), a carbon-rich by-product of petroleum refining, has attracted considerable interest as an inexpensive and widely available precursor for producing advanced carbon materials. Recent studies have demonstrated that PC can be converted into various nanostructured carbons, including graphene-like structures and reduced graphene oxide, which exhibit high electrical conductivity and a large specific surface area. These characteristics make petroleum coke-derived carbon materials promising for the fabrication of electrochemical electrodes and electrocatalyst supports [16,17]. However, the physicochemical properties, morphology, and structural organization of the resulting carbon materials are strongly influenced by the selected precursor and synthesis conditions. Moreover, different technological approaches can be employed for coke production depending on the desired material characteristics and its intended application [18].

Modifying the surfaces of screen-printed electrodes with a SiC/CDC-CS composite further enhances their analytical performance in the determination of methyl parathion (MP) at subnanomolar concentrations. In this composite, silicon carbide (SiC) provides high chemical stability and favorable charge transfer properties, while carbon derived from coke (CDC) imparts a porous structure, a large electroactive surface area, and numerous sites for MP adsorption. Chitosan (CS) acts as a biocompatible film-forming binder, promoting uniform immobilization of SiC and CDC on the electrode surface while simultaneously enhancing the mechanical stability of the sensor layer. The synergistic interaction between these components can accelerate electron transfer, increase analyte accumulation, and enhance the sensitivity and selectivity of the modified SPE. Furthermore, the non-enzymatic SiC/CDC-CS based sensor avoids the limited stability and sensitivity to external factors characteristic of enzymatic systems. Thus, SiC/CDC-CS modified SPEs represent a reliable, portable, and cost-effective platform for rapid on-site monitoring of pesticide residues in water and food samples.



Figure 1 - Schematic illustration of the preparation of a SiC/CDC-CS/SPE and voltammetric detection of the analyte

2 MATERIALS AND METHODS

Petroleum coke, potassium ferrocyanide ($K_4[Fe(CN)_6]$), potassium ferricyanide ($K_3[Fe(CN)_6]$), Silicon carbide (SiC) from Sinopharm Chemical Reagent Co. Ltd. (Shanghai, China). The electrochemical experiments were conducted in a solution of 5 mmol L^{-1} $K_3[Fe(CN)_6]/K_4[Fe(CN)_6]$ and 0.1 M KCl . This redox pair solution is well suited for studying the electrochemical redox behavior of the synthesized material. The pH of the solution was adjusted to the desired value using hydrochloric acid and sodium hydroxide solutions.

Electrochemical measurements, including cyclic voltammetry (CV), electrochemical impedance spectroscopy (EIS), and square-wave voltammetry (SWV), were performed using a CS Potentiostat/Galvanostat (Wuhan CorrTest Instruments Co., Ltd., Wuhan, China). The surface morphology of the fabricated films was studied using SEM images obtained on ZEISS Ultra 55 scanning electron microscope (ZEISS, Germany). EDS analyses were performed using an Energy Dispersive Spectrometry (EDS, Bruker Nano GmbH, Berlin, Germany).

2.1 Preparation of SiC/CDC-CS

The SiC/CDC-CS composite was prepared using a simple solution-assisted dispersion method. First, a 0.5% chitosan solution was prepared by dissolving 50 mg of chitosan in 10 mL of 1.0 % acetic acid solution. The mixture was magnetically stirred until a clear and homogeneous solution was obtained. Subsequently, 10 mg of SiC and 10 mg of CDC were added to the chitosan solution. The resulting suspension was ultrasonicated for 30-45 min to break up particle agglomerates and ensure the uniform distribution of SiC and CDC within the chitosan matrix. The mixture was then continuously stirred for 2 hours at room temperature to promote interactions between the carbon material, SiC particles, and functional groups of chitosan. The obtained homogeneous SiC/CDC-CS suspension was used directly as an electrode-modifying ink. A defined volume of the composite suspension was drop-cast onto the working area of the screen-printed electrode and dried at room temperature or at 40°C until a stable sensing layer was formed. Before electrochemical measurements, the modified electrode was gently rinsed with distilled water to remove loosely bound particles and dried under ambient conditions. The SiC mass ratio and chitosan concentration may be further optimized to achieve the highest electrochemical response toward the target analyte.

2.2 Preparation of the sensor

Before modification, the surface of the SPE was sequentially rinsed with deionized water and ethanol to remove possible contaminants and subsequently dried at room temperature. To prepare the modification ink, 2 mg of the previously synthesized SiC/CDC-CS composite was dispersed in 1 mL

of deionized water and ultrasonicated for 10 min to obtain a homogeneous and stable suspension. Subsequently, 5 μL of the resulting suspension was drop-cast onto the working electrode surface of the SPE. The modified electrode was allowed to dry under ambient conditions until a uniform sensing layer was formed. The prepared sensor, denoted as SiC/CDC-CS/SPE, was stored in a desiccator at room temperature until further electrochemical measurements.

3 RESULTS AND DISCUSSION

3.1 Surface morphology

The surface morphologies of SiC, CDC, and the SiC@CDC composite were investigated using SEM analysis. Figure 2 and 3 present SEM images of the materials obtained at different magnifications: (a, b) SiC, (c, d) CDC, and (e, f) the SiC@CDC composite. Figure 2(a), recorded at $\times 200$ magnification, shows numerous densely packed spherical SiC particles of various sizes. The close contact between the particles and the formation of individual agglomerates indicate the heterogeneous structure of the material. The image obtained at $\times 1000$ magnification in Figure 2(b) clearly demonstrates the spherical morphology of the SiC particles.

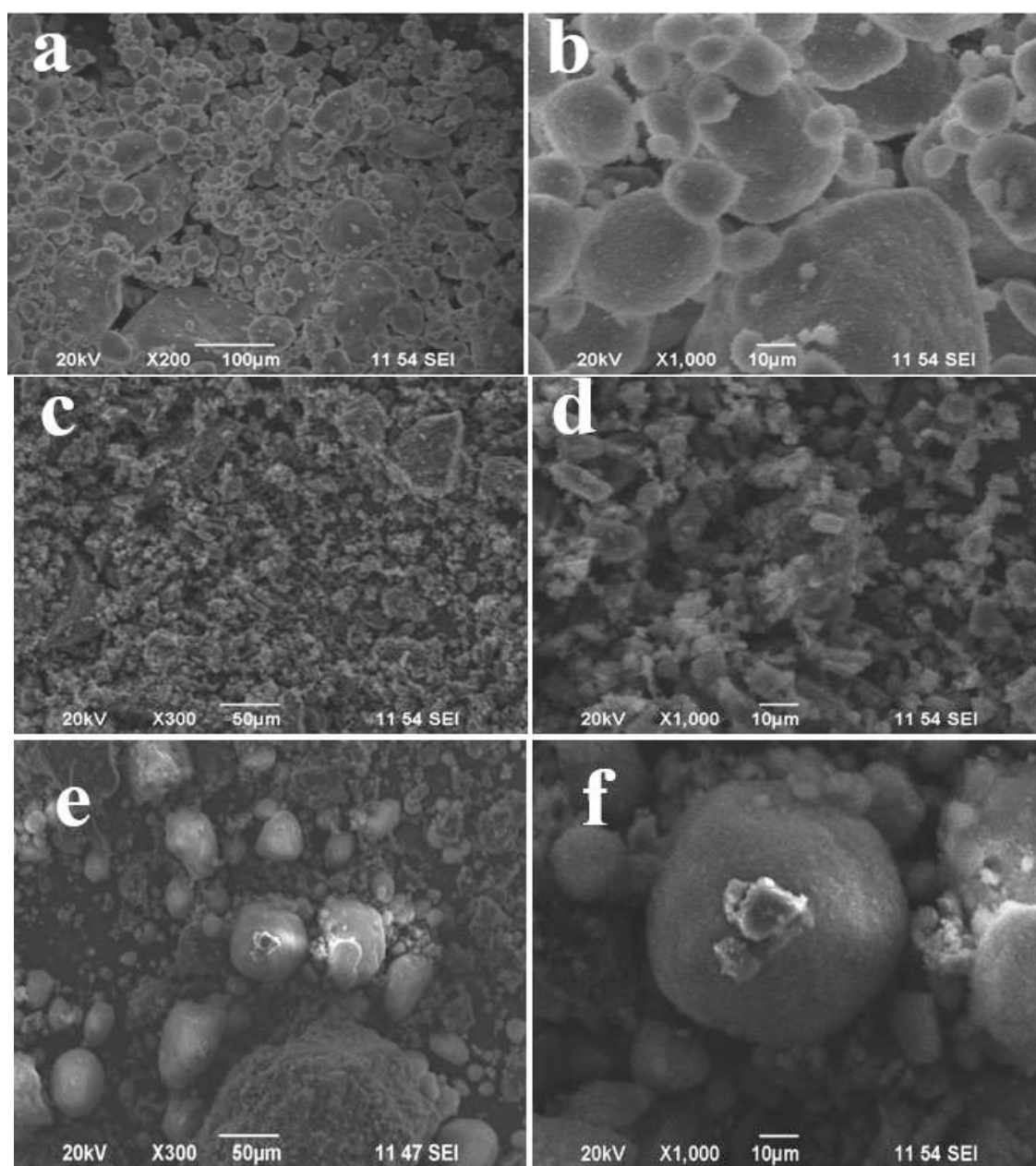


Figure 2 - SEM images of SiC-NPs (a,b), CDC (c, d), and SiC@CDC nanocomposites (e, f)

According to the scale bar, their sizes generally range from several to tens of micrometers. Although some particles possess relatively smooth surfaces, fine granular structures and rough regions can also be observed. Figures 2(c, d) show the morphology of the CDC material. The low-magnification SEM image indicates that CDC consists of small, irregularly shaped particles and their agglomerates. At $\times 1,000$ magnification, its granular, rough, and non-uniform surface structure becomes clearly visible. The CDC particles do not exhibit a well-defined geometrical shape, and their sizes vary within the micrometer range. Such heterogeneous morphology may increase the accessible surface area of the material and facilitate the formation of active sites suitable for the adsorption of analyte molecules. Figures 2(e, f) present the micromorphology of the SiC@CDC composite. The composite contains large spherical SiC particles surrounded by smaller CDC particles. At $\times 300$ magnification, spherical particles are observed to be distributed throughout the investigated region. At $\times 1,000$ magnification, fine granular structures and agglomerates corresponding to CDC are clearly visible on the surfaces of the SiC particles. This feature indicates that CDC particles are attached to the SiC surface and confirms the formation of a two-component composite structure.

The coating of the SiC surface with CDC particles increases the surface roughness and the number of electrochemically active sites compared with the pristine material. The carbon structure of CDC may facilitate electron transfer, whereas the SiC particles can provide structural and chemical stability to the modifying layer.

Therefore, the obtained SiC@CDC composite can be considered a promising material for modifying screen-printed electrodes and developing sensitive electrochemical sensors. The elemental composition of the CDC material is presented in Table 1.

Table 1. Elemental composition of the CDC material

Element	Weight % ($\pm$)	Atomic % ($\pm$)
C	89.37 $\pm$ 0.03	92.59 $\pm$ 0.03
O	8.18 $\pm$ 0.03	6.39 $\pm$ 0.03
Al	0.08 $\pm$ 0.00	0.04 $\pm$ 0.00
S	2.37 $\pm$ 0.01	0.98 $\pm$ 0.00
Total	100.00	100.00

3.2 Electrochemical Characterization of the SiC/CDC-CS/SPE

The electrochemical properties of the modified electrode were evaluated using CV (Figure 3A) and EIS (Figure 3B). The measurements were conducted in a 5 mmol L⁻¹ K₃[Fe(CN)₆]/K₄[Fe(CN)₆] redox-probe solution containing 0.1 M KCl at pH 6.5. The CV experiments were performed at a scan rate of 50 mV s⁻¹. In Figure 3, curve a corresponds to the SiC/CDC-CS/SPE, whereas curve b represents the bare SPE.

As shown in Figure 3 (A), the SiC/CDC-CS/SPE (curve a) exhibited a well-defined pair of quasi-reversible oxidation and reduction peaks at 0.26 and 0.17 V, respectively. The corresponding anodic and cathodic peak-current magnitudes were 581.4 and 513.7 μ A. In contrast, the bare SPE (curve b) produced considerably lower and less-defined redox peak currents. The enhanced current response observed after electrode modification indicates that the SiC/CDC-CS composite increased the electroactive surface area and facilitated electron transfer between the K₃[Fe(CN)₆]/K₄[Fe(CN)₆] redox probe and the electrode surface. This improvement can be attributed to the synergistic effects of the composite components. The conductive CDC network provides efficient pathways for electron transport, SiC contributes to the structural and chemical stability of the sensing layer, and chitosan promotes the homogeneous immobilization of SiC and CDC on the SPE surface. The interfacial charge-transfer behavior of the electrodes was further investigated using EIS. In a Nyquist plot, the diameter of the semicircle in the high-frequency region is associated with the charge-transfer resistance at the electrode-electrolyte interface. As shown in Figure 3 (B), the bare SPE (curve b) displayed a pronounced semicircle with an (R_{ct}) value of approximately 600 Ω , indicating relatively slow electron-transfer kinetics. By comparison, the SiC/CDC-CS/SPE (curve a) exhibited a markedly smaller semicircle followed by an almost linear low-frequency region. The substantial decrease in

(R_{ct}) confirms that the SiC/CDC-CS layer reduced the interfacial resistance and promoted faster electron transfer. The CV and EIS results are in good agreement. The increased redox peak currents and decreased charge-transfer resistance demonstrate that the SiC/CDC-CS composite was successfully immobilized on the SPE surface and significantly improved its electrochemical performance. These findings confirm that the SiC/CDC-CS/SPE provides a favorable interface for rapid electron transfer and can serve as a promising platform for the sensitive electrochemical detection of target analytes.

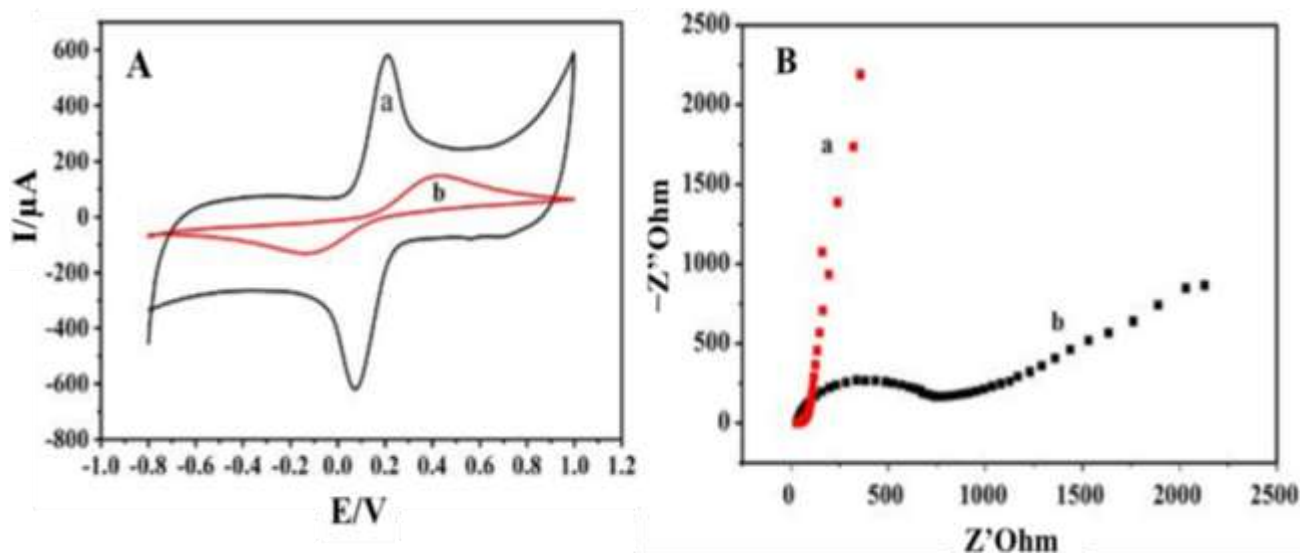


Figure 3 - (A) CV of the SiC/CDC-CS/SPE (curve a) and bare SPE (curve b) recorded at a scan rate of 50 mV s^{-1} . (B) Nyquist plots of the SiC/CDC-CS/SPE (curve a) and bare SPE (curve b) in a $5 \text{ mmol L}^{-1} \text{ K}_3[\text{Fe}(\text{CN})_6]/\text{K}_4[\text{Fe}(\text{CN})_6]$ solution containing 0.1 M KCl at pH 6.5

3.3 Electrochemical behavior of MP on SiC/CDC-CS/SPE

To evaluate the effect of electrode-surface modification on the electrochemical detection of MP, the SWV responses of the bare SPE (curve a), CS/SPE (curve b), SiC/CDC-CS/SPE (curve c), and SiC/CDC/SPE (curve d) were compared. As shown in Figure 4, the bare SPE exhibited a peak current of $4.405 \mu\text{A}$ at a peak potential of -0.039 V , whereas the CS/SPE produced a peak current of $4.928 \mu\text{A}$ at -0.133 V .

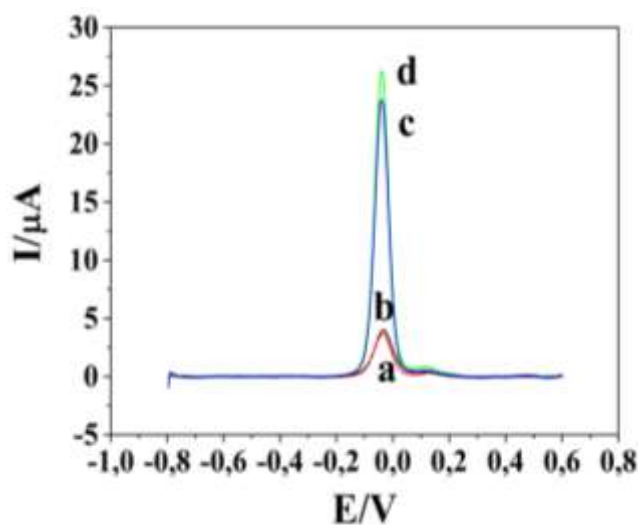


Figure 4 - Square-wave voltammograms of $10 \mu\text{g mL}^{-1}$ methyl parathion obtained using the bare SPE (curve a), CS/SPE (curve b), SiC-CDC-CS/SPE (curve c), and SiC-CDC/SPE (curve d) in 0.2 mol L^{-1} phosphate buffer solution at pH (6.0)

The comparable current responses obtained for the bare SPE and CS/SPE indicate that chitosan does not provide a substantial electrocatalytic contribution to parathion detection. Instead, it primarily serves as a film-forming binder that promotes the uniform and stable immobilization of the nanomaterials on the electrode surface. The SiC/CDC-CS/SPE and SiC/CDC/SPE exhibited significantly enhanced responses toward parathion. The SiC/CDC-CS/SPE produced a peak current of 23.928 μA at -0.028 V, while the SiC/CDC/SPE showed a peak current of 25.998 μA at 0.0209 V. Compared with the bare SPE, the peak currents of the SiC/CDC-CS/SPE and SiC/CDC/SPE increased by approximately 5.4 and 5.9-fold, respectively. This pronounced signal enhancement can be attributed to the increased electroactive surface area of the SiC/CDC composite, the presence of additional adsorption and reaction sites, and the improved electron-transfer properties of the modified electrode. Although the SiC/CDC/SPE generated a slightly higher peak current than the SiC/CDC-CS/SPE, its modifying layer exhibited lower mechanical and storage stability. The film-forming properties of chitosan improve the adhesion and homogeneous distribution of SiC and CDC particles on the SPE surface, thereby reducing material detachment and increasing the stability of the sensing layer. Therefore, considering its high analytical response, improved stability, and suitability for reproducible measurements, the SiC/CDC-CS/SPE was selected as the optimal sensing platform for subsequent

The electrochemical passivation reaction in a PB buffer solution with a concentration of 0.2 mol L⁻¹ was analyzed at various pH values. As the pH of the buffer solution increases, the peak oxidation current first increases and then decreases. At an electrolyte pH of 6.0, the peak oxidation current reaches its maximum, as shown in Figure 5 (A). Therefore, a pH of 6.0 was selected as the optimal electrolyte condition. As the pH increases, the oxidation potential of parathion shifts toward the negative side, and a good linear relationship is observed between the oxidation peak potential and the pH value. As shown in Figure 5 (B), the equation for the linear relationship is $E_{\text{pa}} (\text{V}) = -0.0676\text{pH} + 0.3552$ ($R^2 = 0.9883$), and the slope of the equation is 67.6 mV/pH. According to the Nernst equation, the electrochemical reaction of parathion at room temperature is a reaction with an equal exchange of protons and electrons.

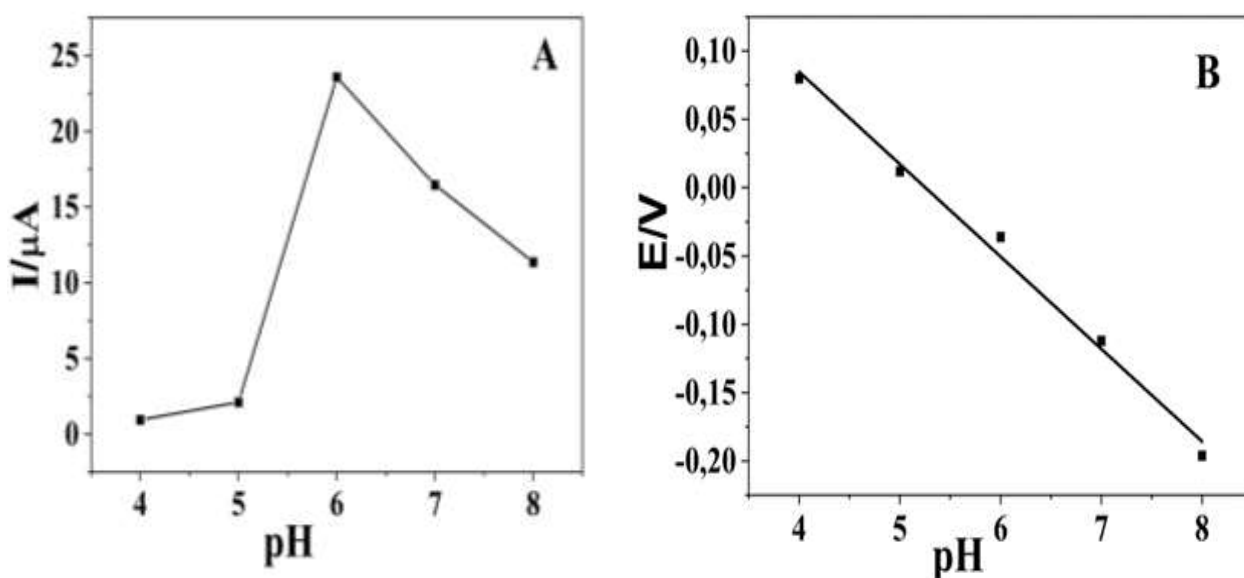


Figure 5 - Investigation of 10 $\mu\text{g mL}^{-1}$ parathion using the SiC/CDC-CS/SPE in 0.2 M phosphate buffer solutions at different pH values (4.0–8.0): (A) relationship between the oxidation peak current and pH; (B) linear relationship between the oxidation peak potential and pH

Under optimized experimental conditions, the quantitative determination of parathion by the SiC/CDC-CS/SPE method using the highly sensitive SWV technique was investigated. As shown in Figure 6 (A), in the concentration range from $1 \times 10^{-9} \sim 1 \times 10^{-5}$ g/mL of SiC NPs-CS/SPE, the current

of the GCE oxidation peak also gradually increases, and, as shown in Figure 6 (B), a good linear relationship is observed between the oxidation peak current and concentration. The corresponding linear equation is: $I_{pa} = 0.00246C \text{ (ng/mL)} + 8.5588$ ($R^2 = 0.9933$). According to the calculations, the sensor's detection limit was $5 \times 10^{-9} \text{ g/mL}$ ($S/N = 3$). These experimental results demonstrated the high sensitivity of SiC NPs-CS/GCE to parathion.

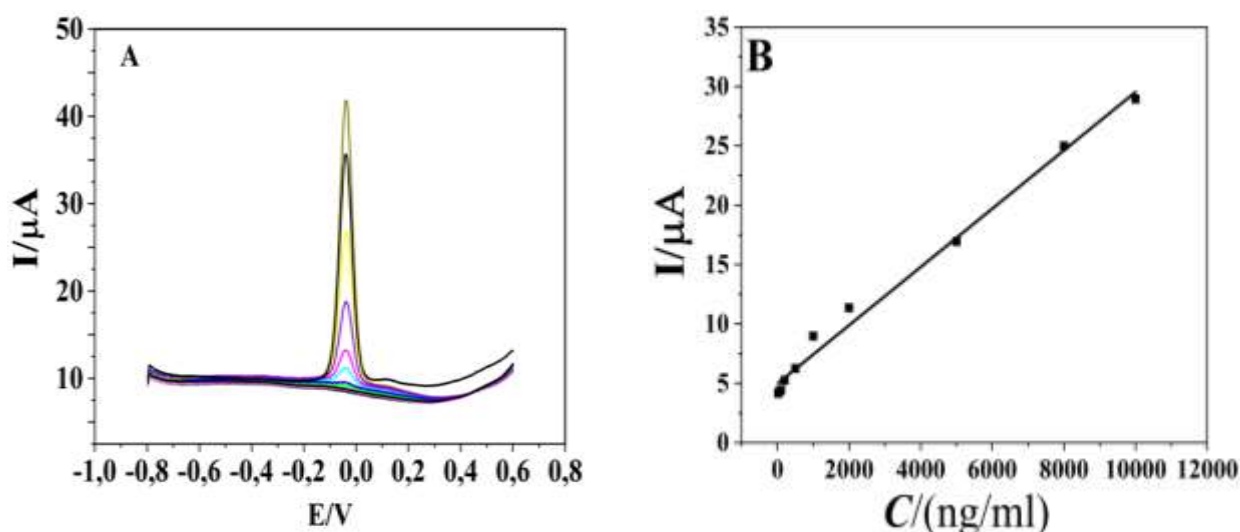


Figure 6 - (A) Rectangular voltamograms obtained on a SiC/CDC-CS/SPE electrode at various concentrations of parathion in a 0.2 M phosphate buffer solution at pH 6.0; (B) Calibration curve showing the dependence of the oxidation peak current on parathion concentration. The inset shows a linear relationship between the oxidation peak current and low methyl parathion concentrations

3.4 Real sample detection

To investigate the sensor's practical performance, we use the standard addition method to determine the methyl parathion concentration in real water samples (See Table 2). The test results are shown in Table 2, with a recovery rate between 100.42% and 103.3% and a relative standard deviation (RSD) of less than 2% for three parallel experiments, indicating that the sensor we developed meets the requirements. with specific analytical needs and promising application prospects.

Table 2. Determination of the methyl parathion content in actual samples

Sample	Added (ng/ml)	Found (ng/ml)	Recovery (%)	RSD (%)
1	300	309.09 ± 7.84	103.3	0.7
2	900	906.06 ± 10.17	100.7	0.6
3	5000	5021.21 ± 195.41	100.42	1.8

4 CONCLUSION

In conclusion, an electrochemical sensor was successfully fabricated by modifying the SPE surface with the SiC/CDC-CS composite using a simple drop-casting method and was subsequently applied to the determination of methyl parathion. The SiC/CDC composite provided favorable adsorption properties and excellent electrochemical performance toward methyl parathion, whereas chitosan effectively immobilized the nanomaterials without adversely affecting the analytical current response. The electrochemical reaction between methyl parathion and the SiC/CDC-CS/SPE was governed predominantly by an adsorption-controlled process. The maximum electrochemical response was obtained in phosphate buffer solution at pH 6.0 with an accumulation time of 6 min.

Under the optimized experimental conditions, the sensor exhibited a good linear relationship between the oxidation peak current and methyl parathion concentration over the range of 1×10^{-9} to $1 \times 10^{-5} \text{ g mL}^{-1}$. The limit of detection was calculated to be $5 \times 10^{-9} \text{ g/mL}$ ($S/N=3$). Moreover, the sensor fabricated using the straightforward and environmentally friendly drop-casting procedure demonstrated good selectivity and stability. Its practical applicability was successfully validated

through the determination of methyl parathion in real water samples, with recoveries ranging from 100.42% to 103.30%. These results demonstrate that the SiC/CDC-CS/SPE is a promising platform for the sensitive and reliable monitoring of methyl parathion in environmental samples.

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