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A Simple, Low-Cost and Efficient β-CD/MWCNTs/CP-based Electrochemical Sensor for the Rapid and Sensitive Detection of Methyl Parathion

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Based on the β-cyclodextrin/multi-walled carbon nanotubes/carbon paper (β-CD/MWCNTs/CP) composite, a simple, low-cost and efficient electrochemical sensor is first designed in the work for the rapid and sensitive detection of methyl parathion (MP). The characteristic peaks of β-CD and MWCNTs are simultaneously observed in the XRD and FTIR results, and these two materials disperse homogeneously on the surface of CP electrode. The results of electrochemical measurements suggest that the obtained β-CD/MWCNTs/CP composite electrode exhibits remarkable synergetic effect, significantly enhancing the efficiency and sensitivity of electrochemical sensor for the selective adsorption property of β-CD, high electrical conductivity and large surface area of MWCNTs, as well as easy operation of CP electrode. Under the optimal conditions, the β-CD/MWCNTs/CP-based electrochemical sensor exhibits excellent MP determination performance with a low detection limit of 2.4 ng mL^{-1}(S/N=3) and a wide linear concentration range from 0.01 to 13 μg mL^{-1}. Given that the CP is not expensive and the construction of CP-based electrochemical sensor can avoid several complex working procedures, this work has a great significance on promoting the more practical electrochemical sensor for the rapid and sensitive detection of MP.

Keywords: β-CD/MWCNTs/CP composite electrode; Synergetic effect; Electrochemical sensor; Methyl parathion

1. INTRODUCTION

The methyl parathion (MP) is vital to contral plant diseases and insect pests, increase production and ensure food supply in the modern agricultural production[1-3]. However, the heavy use of MP always presents pesticide residue problem, adversely affecting human health, ecological balance

and agricultural production[4, 5]. Accordingly, the rapid, convenient and efficient pesticide residue analytical technique should be developed. At present, the conventional analysis methods of MP primarily include the gas chromatography, high performance liquid chromatography, gas or liquid chromatography-mass spectrometer[6-9], etc. These methods are of huge implications to the detection of MP in food and environmental, whereas they have many drawbacks such as the complicated sample pretreatment, time-consuming analysis, excessive professional operation, etc.

In recent years, scientific researchers have deepened the study on rapid detection technology of electrochemical sensors[10, 11]. With the construction of high-performance electrochemical sensors based on chemically modified electrodes, a new research route is created for the rapid, accurate and stable detection of MP. The modification materials mainly include carbon nanomaterials (carbon nanotubes[12], graphene[11, 13, 14], mesoporous carbon[15, 16], etc.), metal nanoparticles[17-19], conductive polymers[20-22], etc. Among these materials, carbon nanotubes have aroused increasing attention for their special microstructural morphology, mechanical performance, and electronic property. Especially, the multi-walled carbon nanotubes (MWCNTs) possess large surface area, high electrical conductivity and electrocatalytic activity, which can effectively optimize the property of electrochemical sensors. Many research works have demonstrated the excellent performance of MWCNTs as modification material for electrochemical sensors[12, 23].

It is generally known that β-cyclodextrin (β-CD) has been extensively considered the critical cyclodextrin with seven glucose units. This material is a type of cyclic oligosaccharide that possesses hydrophobic inner cavity and hydrophilic external cavity. The inner cavity with hydrophobic property exhibits excellent molecular recognition ability and accumulation ability, conducive to the selective recognition and adsorption of numerous organic and inorganic molecules[24, 25]. Moreover, the hydrophilic external cavity can take full advantage of good water solubility to achieve the uniform dispersion of water-resisting materials. According to the existing literature[23, 26, 27], β-CD can effectively disperse MWCNTs in aqueous solution due to the Van der Waals force and hydrogen-bonding interaction of β-CD molecules. In particular, the combination of MWCNTs and β-CD has played a significant role in the sensitivity detection of methyl parathion[28, 29].

Thus far, scientific researchers primarily use the optimized glassy carbon electrode and carbon paste electrode to construct the electrochemical sensors. Especially, the glassy carbon electrode possesses good conductivity, high stability, sensitive electrochemical response, etc. However, it is noteworthy that the electrochemical sensor based on glassy carbon electrode has numerous unavoidable shortcomings such as high production cost and complex pretreatment process. In contrast, the carbon paper (CP) is not expensive, and the CP-based electrochemical sensor can avoid a range of working procedures such as polishing and ultrasonic cleaning. Yue et al. developed the carbon nanotubes/carbon paper composite electrochemical sensor, which has been successfully applied for the sensitive determination of catechol[30]. To the best of our knowledge, the application of the β-CD/MWCNTs/CP-based electrochemical sensor for the sensitive detection of MP has been rarely reported.

In this work, a simple, low cost and efficient β-CD/MWCNTs/CP-based electrochemical sensor is first designed, which is successfully developed for the rapid and sensitive detection of MP. The structure and morphology of the obtained composite were characterized by XRD, FTIR and SEM,

respectively. The electrochemical properties of β-CD/MWCNTs/CP composite electrochemical sensor are studied in depth.

2. EXPERIMENTAL

CP was purchased from Zhuzhou Chenxin Induction Equipment Company. All other reagents were provided from Sinopharm Chemical Reagent Co., LTD. CP was tailored to the suitable size (length: 5 cm, width: 0.5 cm) for use. All reagents were of analytical grade and they were used without being further purified. A certain amount of the mixed solution of NaH_2PO_4 and Na_2HPO_4 was prepared to be used as phosphate buffer solution (0.1 M PBS), and the pH was regulated with 0.1 M H_2SO_4 and 0.1 M KOH solutions. All solutions were prepared with ultrapure water.

The drop-coating method was employed to fabricate the β-CD/MWCNTs/CP-based electrochemical sensor. In a typical fabrication process, a certain amount of MWCNTs was homogeneously dispersed in dimethylformamide (DMF) solvent under ultrasonic dispersion. Subsequently, stoichiometry β-CD powder was added into the above MWCNTs suspension with further ultrasonic treatment to obtain the homogeneous mixture of β-CD/MWCNTs. Then, the mixed suspension (10 μL) was coated on CP surface. After drying with the infrared lamp, the β-CD/MWCNTs/CP-based electrochemical sensor was successfully developed. The MWCNTs/CP composite electrochemical sensor was obtained by using similar technique without β-CD.

The structure and morphology of the obtained composite were studied by X-Ray Diffraction (XRD), under a Fourier transform infra-red spectroscopy (FTIR) and a scanning electron microscopy (SEM), respectively. Cyclic voltammetry (CV), differential pulse voltammetry (DPV) and Electrochemical impedance spectroscopy (EIS) were carried out by using CHI660E electrochemical workstation at room temperature. The modified CP composite electrodes were adopted as working electrode with platinum wire and saturated calomel electrode (SCE) as counter electrode and reference electrode, respectively. CV was carried out under the potential from + 0.6 to - 0.8 V at a scan rate of 20 mV s^{-1}. DPV was carried out between + 0.6 and - 0.8 V with the parameters of pulse amplitude 50 mV, pulse width 0.05 s and pulse period 0.2 s. EIS was carried out with 5 mM $K_3[Fe(CN)_6]/K_4[Fe(CN)_6]$ solution containing 0.1 M KCl in the frequency range of 0.1 Hz-10^5 Hz under an AC applied potential of 0.18 V.

3. RESULTS AND DISCUSSION

Fig. 1a and b show the XRD patterns of MWCNTs and β-CD/MWCNTs. **Fig. 1**a shows that, MWCNTs present significant characteristic diffraction peaks and no impurity diffraction peaks can be observed, suggesting the high purity of MWCNTs[31]. After introducing a certain amount of β-CD, the β-CD/MWCNTs composite (**Fig. 1**b) shows significantly different diffraction peaks from that of MWCNTs. With the exception of the diffraction peaks of MWCNTs, some emerging sharp diffraction peaks can be indexed to β-CD[32], suggesting the co-existence of β-CD and MWCNTs in the

composite. **Fig. 1**c shows the FTIR spectra of β-CD/MWCNTs. It is obvious that this composite exhibits some obvious vibration absorption peaks and stretching vibration peaks. Among them, the characteristic peak at 1630 cm^{-1} belongs to the C=O stretching vibration of MWCNTs, and the characteristic peaks in the range of 2800-3300 cm^{-1} correspond to the C-H and O-H stretching vibration of MWCNTs. Moreover, the vibration absorption peak at 746 cm^{-1} and stretching vibration peaks at 1146, 1073, 988 cm^{-1} correspond to β-CD[33]. The results of the above analysis reveal that the mixture of β-CD and MWCNTs has no substantive effect on their own structures.

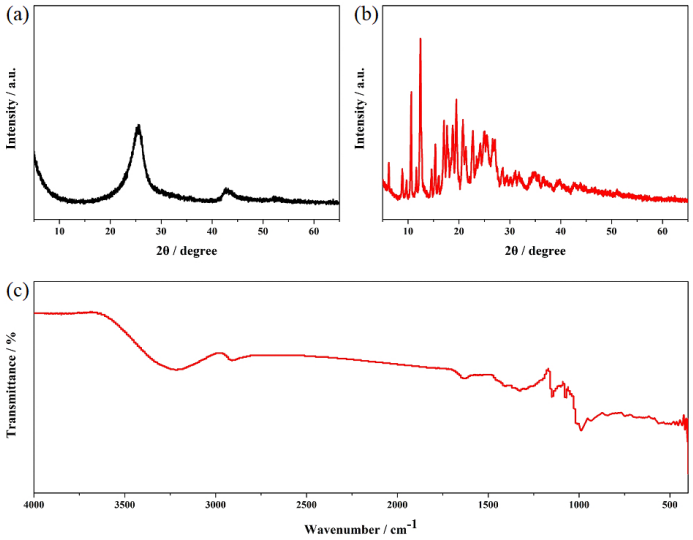

Figure 1. XRD patterns of (a) MWCNTs and (b) β-CD/MWCNTs, (c) FTIR spectras of β-CD/MWCNTs.

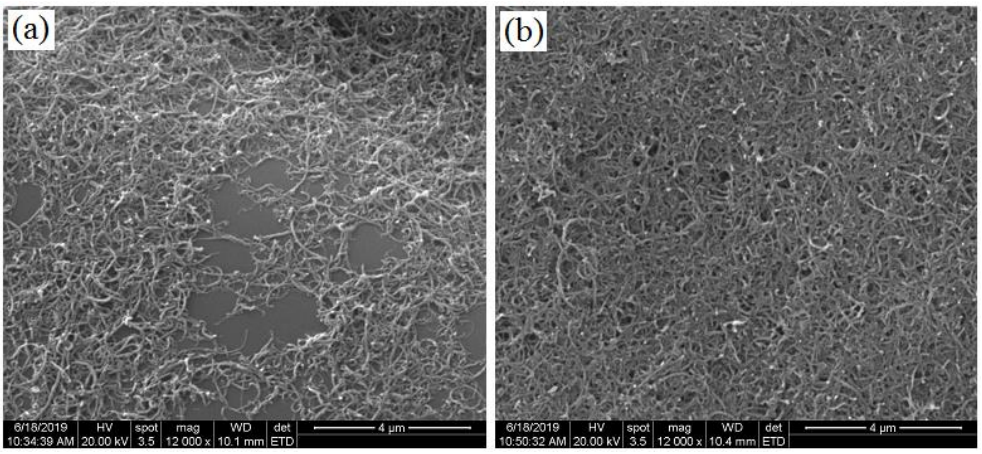

Figure 2. SEM images of (a) MWCNTs/CP and (b) β-CD/MWCNTs/CP electrodes.

The surface morphology of modification material usually significantly affects the electron transport rate and electrolytic participation, which affect the performance of electrochemical sensor. **Fig. 2** shows the SEM images of the MWCNTs/CP and β-CD/MWCNTs/CP electrodes. For the MWCNTs/CP electrode shown in **Fig. 2**a, the MWCNTs were inhomogeneously adhered to the

surface of CP electrode due to the effect of heat distribution and surface tensions associated with the droplet, which is consistent with the previous work[23]. In contrast, the β-CD/MWCNTs/CP composite electrode shown in **Fig. 2**b displays relatively homogeneous distribution of MWCNTs, which mainly benefits from the uniform dispersion effect of β-CD on the water-resisting materials because of the Van der Waals force and hydrogen-bonding interaction of β-CD molecules[28]. Furthermore, when considering the good selective recognition and adsorption effect of β-CD, the β-CD/MWCNTs/CP composite electrode may exhibit prominent electrochemical sensor performance.

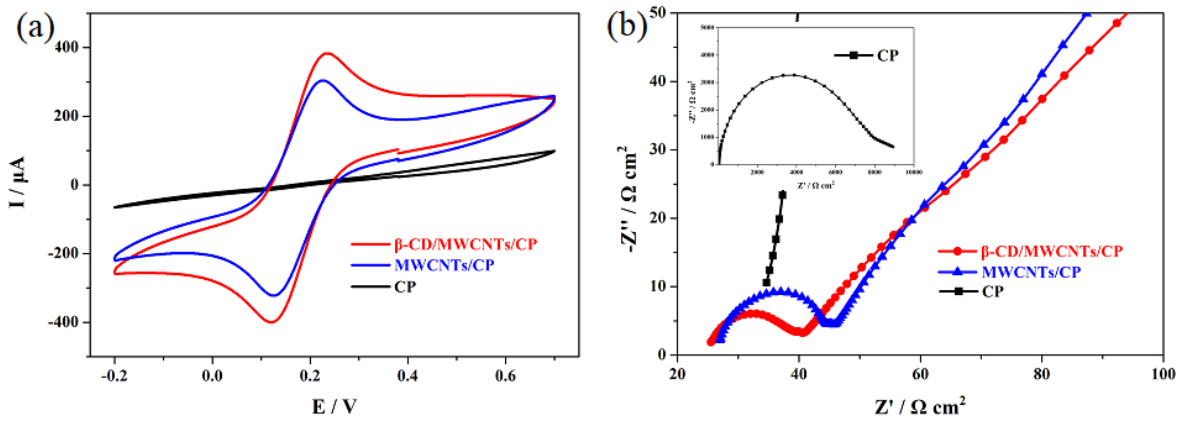

Figure 3. (a) CV curves of CP, MWCNTs/CP, and β-CD/MWCNTs/CP in 5 mM $K_3[Fe(CN)_6]/K_4[Fe(CN)_6]$ solution containing 0.1 M KCl at a scan rate of 20 mV s^{-1}. (b) Nyquist plots of CP, MWCNTs/CP, and β-CD/MWCNTs/CP in 5 mM $K_3[Fe(CN)_6]/K_4[Fe(CN)_6]$ solution containing 0.1 M KCl.

To explore the electrochemical property, the electrochemical sensors based on the undecorated CP, MWCNTs/CP, and β-CD/MWCNTs/CP were studied by cyclic voltammetry (CV) test, and the corresponding CV results are shown in **Fig. 3**a. It is suggested that the undecorated CP-based electrochemical sensor does not present obvious reversible redox peaks in the CV curve. After decorating with MWCNTs, the reversible redox peaks appear with obvious current response, which is closely associated with high electric conductivity and large surface area of MWCNTs[28]. For the β-CD/MWCNTs/CP-based electrochemical sensor, the satisfactory redox peaks can still be observed. In particular, the β-CD/MWCNTs/CP-based electrochemical sensor can present slightly higher peak current response with prominent electrochemical sensor performance, which can be attributed to the larger effective specific surface area of the β-CD/MWCNTs/CP composite electrode[29]. For the β-CD/MWCNTs/CP composite, the uniform dispersion effect of β-CD on the water-resisting materials can facilitate the relatively homogeneous distribution of MWCNTs when the electrode was dried with the help of infrared lamp[28].

Fig. 3b shows the Nyquist plots of these three electrochemical sensors. It is suggested that, the high-frequency region corresponds to the charge transfer resistance (R_{ct}), which has a strong association with the electronic conductivity of the probe at the electrode surface[34]. It can be seen that the decoration materials significantly affect the R_{ct} value. The undecorated CP-based electrochemical

sensor presents relatively high R_{ct} value, and the introduction of a certain amount of MWCNTs significantly reduces the Rct value, suggesting the high electronic conductivity of MWCNTs[30]. Although the β-CD is nonconductive, the β-CD/MWCNTs/CP-based electrochemical sensor still shows relatively satisfactory R_{ct} value due to the larger effective specific surface area of the β-CD/MWCNTs/CP composite electrode, suggesting the high conducting property.

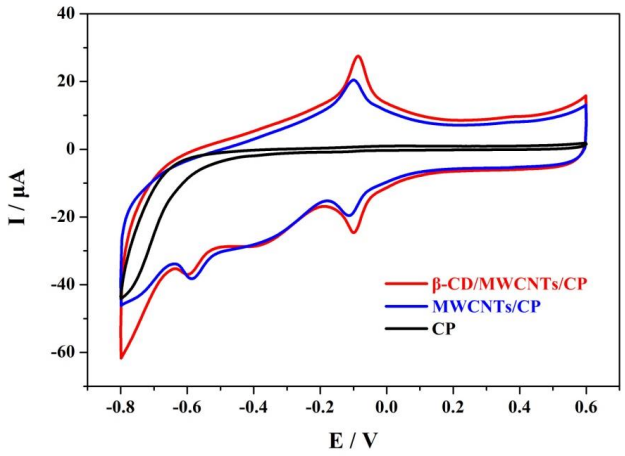

Figure 4. CV curves of CP, MWCNTs/CP, and β-CD/MWCNTs/CP in the presence of 3 μg mL^{-1} MP in 0.1 M PBS at pH 7.0 at a scan rate of 20 mV s^{-1}.

Fig. 4 shows the CV curves of 3 μg mL^{-1} MP in 0.1 M PBS (pH=7.0) at the electrochemical sensors based on the undecorated CP, MWCNTs/CP, and β-CD/MWCNTs/CP. This figure suggests the CV curve of the undecorated CP-based electrochemical sensor does not exhibit obvious current response. In contrast, the MWCNTs/CP-based electrochemical sensor can present irreversible reduction peaks, highly associated with the irreversible reduction of nitro group to hydroxylamine group[29]. Besides, there is a pair of redox peaks (ΔE=12 mV) , primarily due to the reversible redox reaction of hydroxylamine group[35]. For the β-CD/MWCNTs/CP-based electrochemical sensor, similar research results are shown in the CV curves. It is noteworthy that the redox peaks of the β-CD/MWCNTs/CP-based electrochemical sensor are quite sharp with the maximum current response and minimum redox potential difference (ΔE=3 mV), thus revealing the optimum detection performance. Such high performance is primarily attributed to the larger effective specific surface area of the β-CD/MWCNTs/CP composite electrode[28]. The MWCNTs exhibit large surface area and high electric conductivity, while the β-CD exhibits excellent molecular recognition ability and accumulation ability, significantly enhancing the selective adsorption property. The result above analysis indicates the combination of β-CD and MWCNTs can have more excellent effect on the MP detection performance.

To enhance the MP detection performance, the effects of MWCNTs concentration, β-CD concentration, accumulation time, and pH value on the current response were studied by CV or DPV methods, respectively, as shown in **Fig. 5**. **Fig. 5**a shows the effect of β-CD concentration on the oxidation peak current response by CV method, the MWCNTs concentration is 2 mg mL^{-1}. When

introducing a small amount of β-CD, the peak current response increases gradually with the increase of β-CD concentration due to the excellent molecular recognition ability and accumulation ability of β-CD. However, the excessive concentration can exert serious negative effect because of the increased electron transfer resistance. **Fig. 5**b shows the effect of MWCNTs concentration on the current response by CV method, the β-CD concentration is 9 mg mL^{-1}. The peak current response first increases then decreases with the increase of the MWCNTs concentration, and the optimal value is 2 mg mL^{-1}. When the MWCNTs concentration rises, the electrochemical active substance will be up-regulated. However, the higher concentration can lead to the thicker film on the electrode surface, which is not conducive to the electron transfer. **Fig. 5**c shows the effects of accumulation time on the oxidation peak current response by using DPV method. As the accumulation time prolonged, the peak current response is tending towards stability, and the accumulation time corresponding to saturation value is 90 s. Furthermore, it can be found from **Fig. 5**d that the peak current response first increases then decreases with the increase of the pH value, and the optimal value is 7, which may be primarily attributed to the degradation effect of alkaline medium on MP and close relationship between proton and redox reaction[11].

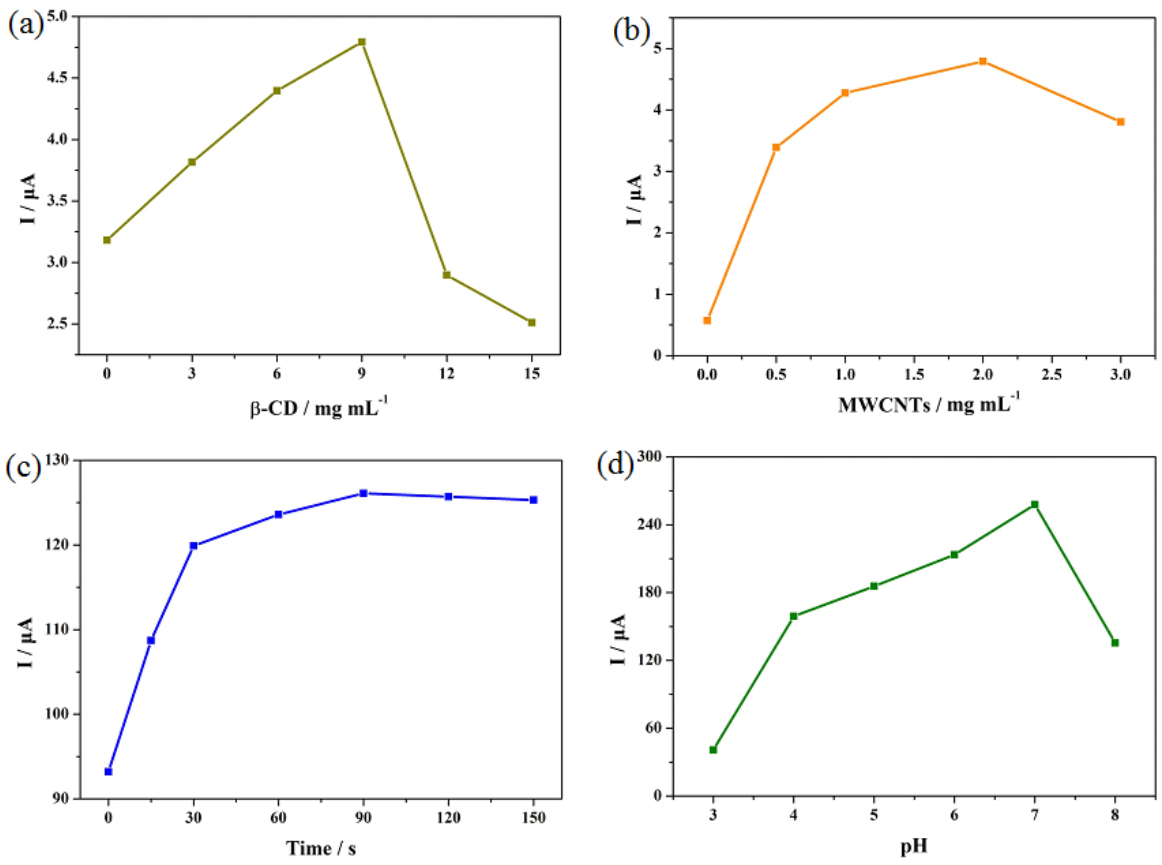

Figure 5. Effects of (a) β-CD concentration, (b) MWCNTs concentration, (c) accumulation time, and (d) pH value on the current response were studied by CV or DPV methods, respectively.

Fig. 6 shows the effect of scan rate on the CV curves of the β-CD/MWCNTs/CP-based electrochemical sensor. The corresponding scan rate is 20, 50, 100, 150, 200, 300 mV s^{-1}, respectively. According to the test results, all the current responses of the oxidation peaks, reversible reduction peaks present an upward tendency along with the increase of scan rate, suggesting that the scan rate can have an important impact on the electrochemical behavior of MP on the obtained electrochemical sensors.

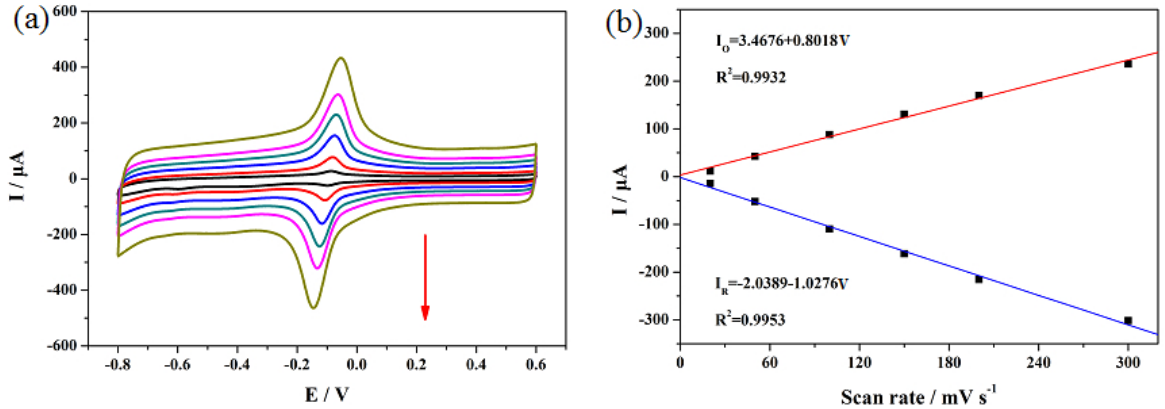

Figure 6. (a) CV curves of β-CD/MWCNTs/CP in 0.1 M PBS(PH=7.0) containing 3 μg mL^{-1} MP at a scan rate of 20-300 mV s^{-1}, (b) the plot of peak current vs. scan rate.

Furthermore, it is obvious that the peak current values are linearly related to the scan rate. The corresponding regression equations are I_O (μA) = 3.4676+0.8018v (R^2=0.9932) and I_R (μA) = -2.0389-1.0276v (R^2=0.9953), respectively. Accordingly to these results, the electrochemical reaction of MP on the β-CD/MWCNTs/CP composite electrode surface is a surface-confined diffusion controlled electrocatalytic process, which agrees with the previous results[4, 10, 14].

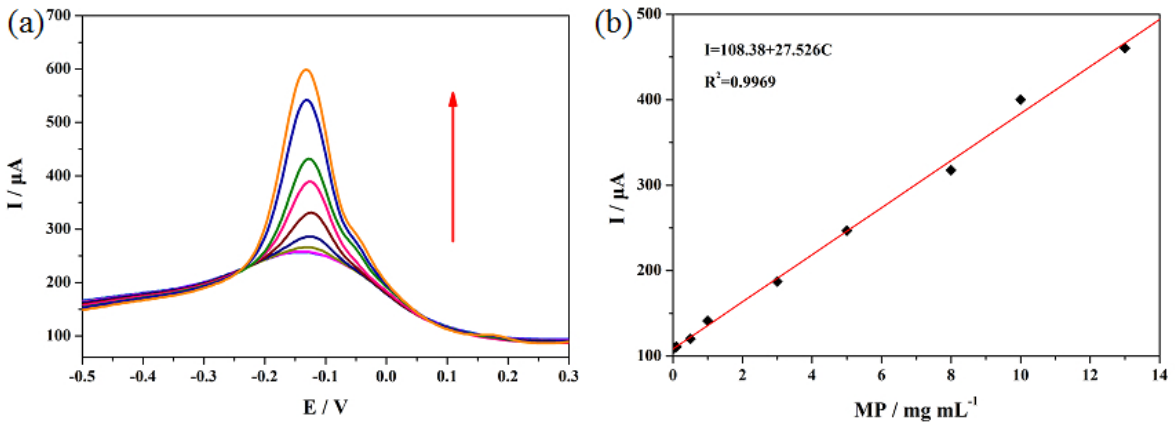

Figure 7. (a) DPVs for determination of MP in 0.1 M PBS(PH=7) with β-CD/MWCNTs/CP at MP concentrations ranging from 0.01 to 13 μg mL^{-1} (0.01, 0.05, 0.1, 0.5, 1, 3, 5, 8, 10 ,13 μg mL^{-1}). (b) Linear relationship between oxidation peak current and MP concentration.

DPV measurement has a higher sensitivity to the trace analysis of MP compared to CV. **Fig. 7**a shows the DPV of the β-CD/MWCNTs/CP-based electrochemical sensor was developed for the determination of MP at concentrations of 0.01-13 μg mL^{-1} in 0.1M PBS with the following optimized parameters. The DPV Potential window from + 0.6 V to- 0.8 V, the pulse amplitude 50 mV, the pulse width 0.05 s, the pulse period 0.2 s and accumulation time of 90 s. It is shown that, with the increase of MP concentration, the oxidation peak current is up-regulated. Furthermore, **Fig. 7**b suggests that the peak current values are linearly related to the MP concentration ranging from 0.01 to 13 μg mL^{-1}. The corresponding regression equation is I (μA) = 108.38+27.526C (R^2=0.9969). The detection limit of the β-CD/MWCNTs/CP-based electrochemical sensor can reach up to 2.4 ng mL^{-1} (S/N=3). The research results of the existing reports and this work are listed in **Table 1**. It can be found that the β-CD/MWCNTs/CP-based electrochemical sensor can exhibit excellent detection performance of MP, which are primarily contributed by the synergistic effect of β-CD and MWCNTs on the CP electrode surface[28, 29]. Given that the carbon paper (CP) is not expensive and the CP-based electrochemical sensor can avoid a range of complex working procedures such as polishing and ultrasonic cleaning, this work can significantly promote the research and development of high-performance electrochemical sensor for the rapid and sensitive detection of MP.

Table 1. Comparison of the performance the existing reports and this work.

Electrode	Analytical method	Detection limit (ng mL^{-1})	Linear range (μg mL^{-1})	Reference
CuO-TiO_2/GCE	DPV	1.21	0-2	[36]
AuNPs/Nafion/GCE	SWV	26.3	0.13-31.59	[37]
ZrO_2/Au electrode	SWV	3	0.005-0.1	[38]
Pd/MWCNTs	DPV	50	0.1-14	[39]
Au- ZrO_2-GNs/GCE	SWV	1	0.001-2.4	[13]
BCL@MOF/nanofibers/chitosan/GCE	DPV	17.64	0.026-10	[40]
β-CD/MWCNTs/CP	DPV	2.4	0.01-14	This work

Table 2. Analytical results of MP in the real water samples using the proposed method (n = 3)

Sample	MP added (μg mL^{-1})	MP found (μg mL^{-1})	Recovery (%)	RSD (%)
River water 1	0.12	0.13	105.55	4.6
River water 2	4.50	4.41	97.90	3.3
River water 3	6.50	6.48	99.63	0.3
Tap water 1	0.12	0.12	99.99	8.3
Tap water 2	4.50	4.45	98.97	4.5
Tap water 3	6.50	6.45	99.20	0.5

To demonstrate the applicability of the proposed sensor for MP determination in two real water samples containing tap water and river water. The two real water samples were first filtered using a standard 0.22 μm filter and then spiked with MP standard solutions at three concentrations[41]. The

recovery amount of each sample is the average of three measured concentration using the standard addition method. As listed in **Table 2**, the recoveries of the river water and tap water samples were varied from 97.90 % to 105.55 % and 98.97 % to 99.99 %, respectively. Therefore, this result showed that the proposed sensor possesses an excellent practicability and accuracy for the determination of MP in the real water sample.

4. CONCLUSIONS

In this work, a simple, low cost and efficient β-CD/MWCNTs/CP-based electrochemical sensor is developed for the rapid and sensitive detection of MP. According to the research results, the β-CD/MWCNTs/CP composite electrode has a remarkable synergistic effect, which can make full use of the selective adsorption property of β-CD, high electrical conductivity and large surface area of MWCNTs. Especially, the CP is not expensive and the CP-based electrochemical sensor does not involve the polishing and ultrasonic cleaning procedures. Most importantly, the β-CD/MWCNTs/CP-based electrochemical sensor has a linear relation with the MP concentration in range of 0.01-13μg mL^{-1} with a low detection limit of 2.4 ng mL^{-1}. This work provides a pretty valuable strategy to effectively detect the methyl parathion.

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