International Journal of ELECTROCHEMICAL SCIENCE www.electrochemsci.org

CoO NPs/c-CNTs nanocomposite as electrochemical sensor for sensitive and selective determination of the carbofuran pesticide in fruits and vegetables

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Received: 24 January 2021 / Accepted: 22 March 2021 / Published: 30 April 2021

This study was carried out for preparation and characterization of nanocomposite of CoO nanoparticles and carboxylated CNTs (CoO@c-CNTs NC) as electrochemical sensor for determination of the carbofuran pesticide in fruits and vegetables. The CoO@c-CNTs NC was prepared by dropping the centrifuged suspension of c-CNTs and CoO nanoparticles (CoO NPs) on activated glassy carbon electrode (GCE) as substrate and drying at room temperature. Morphological and structural studies of prepared NC using SEM and XRD exhibited the anchoring of rock-salt cubic phase of CoO NPs on c-CNTs sheets which provided an effective electrical network for stabilization of well-distributed metal oxide NPs and higher effective surface area. Results of electrochemical studies with CV and DPV measurements showed that the high conductivity and large effective surface area of CNTs led to the higher electron transfer rate and higher current electro-oxidation of carbofuran in CoO@c-CNTs NC/GCE than that of CoO NPs/GCE. The linear range, detection limit and sensitivity values were obtained of 0-260 µM, 0.004 µM and 0.07275µA/µM for detection of carbofuran, respectively. The comparison of obtained results for prepared carbofuran sensors with the other reported sensors showed that the detection limit and linear range of the CoO@c-CNTs NC/GCE were comparable or better than that of values obtained in some of reported carbofuran sensors. The study of interference effects of CoO@c-CNTs NC/GCE showed the good selectivity and anti-interference ability of the sensor for determination of carbofuran. The practical application of sensor for determination of the carbofuran pesticide in cabbages and oranges samples revealed that the carbofuran contents in the cabbages and oranges samples were estimated 0.66 μ M and 0.18 μ M, respectively. Moreover, the obtained recovery and RSD values by the standard addition method indicated that the sensor had good accuracy for carbofuran detection in fruits and vegetables.

Keywords: Electrochemical sensor; Nanocomposite; CoO nanoparticles; Carboxylated CNTs; Pesticide; Carbofuran; Fruits; Vegetables

1. INTRODUCTION

Carbofuran ((2,2-Dimethyl-3H-1-benzofuran-7-yl) N-methylcarbamate) as a toxic carbamate pesticides is one of the most rapidly degraded insecticides in field crops, including potatoes, maize, corn, oat, rice, eggplant, soybeans, cotton, and cucurbits. Many researches have been shown that carbofuran has systemic activity in plants and was absorbed through the root of plants and quickly distributed to the leaves without accumulation in the roots [1-4]. In addition, over 80% of the carbofuran was converted to 3-hydroxycarbofuran and over 5% was converted to 3-oxocarbofuran [5].

This pesticide is widely applied as an effective insecticide in the agricultural industry as a major industry in the United States. Toxicity of carbofuran shows the same mechanism as notorious V-series nerve agents due to inhibiting cholinesterase and disruption of melatonin [6-8]. It is as a neurotoxic pesticide can effect on circadian rhythm balance and increase the diabetes risk [7, 9]. Furthermore, studies confirmed that carbofuran can disrupt the endocrine performance and leads to alterations in the concentration of hormones and serious reproductive problems. This pesticide not only presents risks to human health, but also is known to be particularly toxic to birds, coyotes, kites, golden eagles, buzzards, domestic dogs, raccoons, vultures, lions and other predators.

Therefore, carbofuran is classified as a very hazardous material in the United States, Canada, the European Union and Kenya [10]. On the other hand, many studies have been conducted on characterization and determination of carbofuran in field crops, fruits, vegetables and agriculture wastewaters through fluorimetry method, coupled-column liquid chromatography/mass spectrometry, high-performance liquid chromatography, colorimetry, fluoroimmunoassay, flow injection analysis, spectrophotometry, enzyme-linked immuno-sorbent assay and electrochemical techniques [11-19]. Thus, this study was carried out for determination of the carbofuran pesticide in fruits and vegetables by nanocomposite of CoO NPs and CNTs electrochemical sensor.

2. MATERIALS AND METHOD

In order to prepared CoO@c-CNTs NC/GCE, GCE was polished using alumina powder (α -Al2O3, 0.3 µm, 99.9%, Anhui Elite Industrial Co., Ltd., China) on a polishing cloth and immersed in 0.1 M HCl for 2 minutes (37%, Qingdao HiseaChem Co., Ltd., China) and ethanol for 10 minutes (95%, Xilong Scientific Co., Ltd., China), respectively, and then ultrasonically rinsed with deionized water. To activate the surface of GCE, the GCE was sonicated in 1 mM nitric acid (68%, Qingdao HiseaChem Co., Ltd., China) for 5 minutes. The CNTs (99%, Xuzhou Jiechuang New Material Technology Co., Ltd., China) were ultrasonically purified by sonication in a 5 M HCl for 2 hours. Next, 10 mg of purified CNTs were carboxylated in a 20 ml mixture of 0.1 M H₂SO₄ (98%, Qingdao HiseaChem Co., Ltd., China) and 0.1 M HNO₃ in volume ratio of 3:1 for 2 hours. The carboxylated CNTs (c-CNTs) were ultrasonically washed with deionized water. In order to preparation the CoO@c-CNTs NC, 1g/l of c-CNTs and1g/l CoO nanoparticles (99.9%, 2 µm, Handan Yaxiang Chemicals Trading Co., Ltd., China) were ultrasonically dispersed in 1 ml of absolute anhydrous ethyl alcohol(Merck, Germany) for 5 minutes. Then, the mixture was centrifuged for 5 minutes at 1000 rpm, and the resulting suspension was dropped on GCE and dried at room temperature.

The morphology of c-CNTs/GCE, CoO NPs/GCE and NC/GCE CoO@c-CNTs NC/GCE was studied through scanning electron microscopy (Hitachi SEM S4800). X-ray diffraction (XRD) operated at 40 kV, current of 15 mA and wavelength radiation of CuK α (1.5418 Å) was used to study crystalline structures of prepared samples. Electrochemical studies with cycle voltammetry (CV) and differential pulse voltammetry (DPV) measurements were conducted on Autolab Potentiostatin a conventional three-electrode electrochemical cell which contained prepared electrode, Ag/AgCl and Pt electrochemical assessments were performed in 0.1 NaOH (99.9%, Shandong Kaiteda Chemical Co., Ltd., China) and phosphate buffer solution (PBS, 0.1M) which prepared from 0.1 MNa₂HPO₄ (99.0%,Shanghai Ruizheng Chemical Technology Co., Ltd., China) and 0.1 M H₃PO₄ (98%, Honghao Chemical Co., Ltd., China) in equal volume ratio. For preparation of the real samples of vegetables and fruits, the chopped cabbages and oranges samples were weighed, sprayed with 1µM concentration of carbofuran, and then transferred to centrifuge tubes (1500 rpm for 10 minutes). 1L of archived supernatants was mixed with 1L of 0.1 M PBS solution.

3. RESULTS AND DISCUSSION

FESEM images of c-CNTs and CoO@c-CNTs NC are shown in Figure 1. For c-CNTs, SEM image from Figure 1a shows c-CNTs with wide length distribution from 8 to 10 µm and diameters ranging from 30 to 55 nm. The SEM image of CoONPs@c-CNTs NC in Figure 1b exhibits the formation of a flower-like of CoO nanoparticles in diameter of about 25 nm which is distributed in c-CNTs texture. The c-CNTs in hybrid materials is providing an effective electrical network for stabilization of well-distributed metal oxide NPs [20].



Figure 1. SEM images of (a) c-CNTs, (b) CoO@c-CNTs NC.

The crystalline structure of the powders of prepared c-CNTs, CoO NPs and CoO@c-CNTs NC are displayed in XRD patterns of Figure 2. For c-CNTs, XRD pattern presents two diffraction peaks at 26.49° and 54.58°, which corresponded to the (002) and (004) planes of hexagonal graphite carbon of CNTs (JCPDS Card No. 75-1621). As shown in Figure 2b, XRD pattern of CoONPs@c-CNTs NC

exhibits three dominant diffraction peaks of rock-salt cubic phase of CoO at 36.48°, 42.38° and 61.73°, which attribute to the (111), (200) and (220) planes, respectively.XRD pattern of CoO@c-CNTs NC shows two diffraction peaks of c-CNTs and three diffraction peaks of CoO which indicated to anchoring of rock-salt cubic phase of CoO NPs on c-CNTs sheets.



Figure 2. XRD patterns of the powders of prepared (a) c-CNTs, (b) CoO nanoparticles and (c) CoO@c-CNTs NC.

In order to study the electrochemical properties of GCE, c-CNTs/GCE, CoONPs/GCE and CoO@c-CNTs NC/GCE, the CV measurements were performed in 0.1 M NaOH pH 10 at scan rate of 20 mV/s. As seen from Figure 3, there aren't any redox peaks for the CVs of the GCE and c-CNTs/GCE in alkaline solution. The CVs of the CoO NPs/GCE and CoO@c-CNTs NC/GCE show the three peaks which contained two oxidation peaks (O₁and O₂) in the anodic sweep for CoOOH undergoes a change in the oxidation state of the Co atom to formation of $CoO_2(O_2)$ and $Co_3O_4(O_1)$ [21], and single reduction peak (R₁) in the cathodic sweep for reduction of Co₃O₄ to form CoOOH [21]. The peak currents of CoO@c-CNTs NC/GCE show the larger values than that of CoO NPs/GCE. It can be due to the uniform distribution of CoO NPs and less rigidity of CNTs nanostructure. Moreover, according to the SEM images it is observed that the CoO NPs formed flower-like on CNTs which provide great stability and higher electro-active surface area.

For study the electrocatalytic properties of the prepared electrode in presence of carbofuran, the DPV measurements were performed in presence of carbofuran 0.1 M PBS pH 10 at scan rate of 20 mV/s. The obtained DPVs in Figure 4 illustrate that the electro-oxidation of carbofuran occurs at the CoO NPs/GCE and CoO@c-CNTs NC/GCE. It seems that the electron transfer kinetics is efficiently

promoted at CoO@c-CNTs NC/GCE. The great electrocatalytic response is recorded in CoO@c-CNTs NC/GCE due to the synergistic effects CNTs nanosheets and of CoONPs which consisted of presence of high catalytic active site for the carbofuran oxidation on well-distributed, high amounts of loaded nanoparticles on nanocomposite, fast electron-transfer kinetics between the electrode and the redox-active centers through anchored CoO NPs on the CNTs nanosheets sides, and CNTs high electrical conductivity and high chemical stability [20, 22, 23]. Thus, the high conductivity and large effective surface area of CNTs leads to the higher electron transfer rate and higher current electro-oxidation of carbofuran in CoO@c-CNTs NC/GCE than that of CoONPs/GCE [24]. Therefore, the next experiments to study the catalytic activity of carbofuran were conducted on CoO@c-CNTs NC/GCE.



Figure 3. The CVsof (a) GCE, (b) c-CNTs/GCE, (c) CoO NPs/GCE and (d) CoO@c-CNTs NC/GCE in 0.1NaOH pH 10 at scan rate of 20 mV/s.

Figure 5 displays the DPVs measurements of CoO@c-CNTs NC/GCE in different pH (2-11) of 0.1 M PBS containing 100 μ M carbofuran at scan rate of 20 mV/s. As shown, there are the oxidation peak for any DPVs which with increasing pH of the PBS containing 100 μ M carbofuran, peak potential tend to shift to negative potentials. There is a linear correlation between the anodic peak potential and pH value with a slope value of 0.058V/pH (Figure 5b). This value is very close to the theoretical value of 0.059V/pH that indicates an equal number of proton(s) and electron(s) transferred during the oxidation process [23, 25, 26]. In addition, variation of oxidation peak current with pH in Figure 5c reveals that the maximum peak current recorded at pH 7.0 and 6.0. Thus, the following studies for determination of carbofuran were carried out in 0.1 M PBS (pH 7.0).



Figure 4. The DPVs of (a) GCE, (b) c-CNTs/GCE, (c) CoO NPs/GCE and (d) CoO@c-CNTs NC/GCE in 0.1 M PBS pH 7 at scan rate of 20 mV/s in presence of 100 µM carbofuran.



Figure 5. (a) The DPVs of CoO@c-CNTs NC/GCE in different pH (2-11) of 0.1 M PBS containing 100 μ M carbofuran at 20 mV/s scan rate. Variation of oxidation peak (b) potential and (c) current *vs.* pH.

Figure 6a exhibits the DPVs measurements of CoO@c-CNTs NC/GCE in different concentrations of carbofuran in 0.1 M PBS (pH 7.0) at scan rate of 20 mV/s.As observed, the oxidation peak current is increased linearly with increasing concentrations of carbofuran in electrochemical cell. The calibration curve in Figure 6b shows that sensitivity and detection limit values are obtained of 0.07275 μ A/ μ M and 0.004 μ M for detection of carbofuran, respectively. The linear range value of the carbofuran sensor is obtained 0-260 μ M for CoO@c-CNTs NC/GCE as a carbofuran sensor. The obtained results are compared with the other reported carbofuran sensor in Table 1. The detection limit and linear range of the CoO@c-CNTs NC/GCE are comparable or better than that of values obtained in some of reported carbofuran sensors [11-13, 15, 27, 28]. Thus, the CoO@c-CNTs NC/GCE can be applied to expand a potentially beneficial stable electrochemical sensor for determination of the carbofuran.



Figure 6. (a) DPVs of CoO@c-CNTs NC/GCE in different concentrations of carbofuran in 0.1M PBS (pH 7.0) at 20 mV/s scan rate and (b) its calibration curve.

Table	1.	Comparison	of	CoO@c-CNTs	NC/GCE	for	carbofuran	determination	with	other	reported
	se	nsors.									

detector	Technique	Detection	Linear	Ref.
		limit	range	
		(µM)	(µM)	
CoO@c-CNTs NC/GCE	DPV	0.004	0-260	This
				work
Au NPs@reducedgraphene	DPV	0.02	0.05-20	[11]
oxide/GCE				

diallyldimethyl ammonium @	DPV	0.407	-	[27]
graphene oxide/ indium tin oxide				
CoO decorated reduced	DPV	0. 019	0.5–200	[28]
graphene oxide				
acetylcholinesterase/dendrimerspo	DPV	0.004	0.0048-	[13]
lyamidoamine -Au/ CNTs			0.07	
Heated screen-printed carbon	DPV	0.05	0.4–400	[29]
electrode				
Acetylcholinesterase/Fe ₃ O ₄ -	SWV ^a	0.0036	0.005-	[15]
chitosan /GCE			0.09	
poly(3,4-	LSV ^b	0.1	1–90	[12]
ethylenedioxythiophene)/polystyre				
ne sulphoanate/graphene oxide				

^a SWV: Square wave voltammetry ^bLSV: Linear

^bLSV: Linear sweep voltammetry

In order to study the interference effect of CoO@c-CNTs NC/GCE to determination of carbofuran, the recorded peak current of DPVs measurements in 0.1 M PBS pH 7 at -0.15 V to addition 10 μ M of carbofuran and 25 μ M of the pesticides as possible interfering compounds which contained aldicarb, nitrophenol, pirimicarb, ethyl parathion, isoprocarb, methomyl and metholcarb. Table 2 shows the recorded peak current of DPVs measurements for additions of carbofuran and other substances which indicated the insignificant signal for interference. Therefore, the results illustrate the good selectivity and anti-interference ability of the CoO@c-CNTs NC/GCE for determination of carbofuran.

Table 2	2. The recorded peak current	of DPVs measurements in 0.1 M PBS pH 7 at -0.15 V to addition
	$10\ \mu M$ of carbofuran and 25	μ M of the other pesticides as possible interfering compounds

Substances	Concentration (µM)	Recorded peak current	RSD ^a (%)
		(µA)	
carbofuran	10	0.701	± 0.088
aldicard	25	0.081	±0.009
nitrophenol	25	0.089	± 0.007
pirimicarb	25	0.098	±0.006
ethyl parathion	25	0.101	±0.011
isoprocarb	25	0.077	±0.005
methomyl	25	0.107	±0.006
metholcarb	25	0.095	±0.004

^a RSD: relative standard deviation

To study the practical application of CoO@c-CNTs NC/GCE to determination of the carbofuran pesticide in fruits and vegetables, the concentrations of carbofuran were detected in the cabbages and oranges samples trough record of DPVs measurements in prepared real sample in 0.1 M PBS (pH 7.0) at scan rate of 20 mV/s with addition of carbofuran solutions. Figure 7 and 8 show the DPV measurements and related calibration curves of prepared cabbages and oranges samples, respectively. As shown in the calibration plots in Figs. 7 and 8, the carbofuran content in electrochemical cells of prepared samples of the cabbages and oranges in 0.1M PBS are 0.83 μ M and 0.509 μ M respectively, which implied to presence of 1.66 μ M and 1.18 μ M in pure real samples of cabbages and oranges, respectively. This value is very close to initial concentration of sprayed carbofuran solution on samples. Therefore, the carbofuran contents in the cabbages and oranges samples are estimated 0.66 μ M and 0.18 μ M, respectively. Moreover, the obtained recovery (93.66 to 98.50% for cabbages and 95.00 to 97.00% for oranges samples) and RSD (2.14 to 4.11% for cabbages and 1.24 to 4.15% for oranges samples) values by the standard addition method in Table 3 indicated that the CoO@c-CNTs NC/GCE has good accuracy for carbofuran detection in fruits and vegetables.



Figure 7. (a) DPVs and (b) calibration plots of CoO@c-CNTs NC/GCE to addition of carbofuran solution at scan rate of 20 mV/s in 0.1 M PBS (pH 7.0) prepared of real samples of cabbage.



Figure 8. (a) DPVs and (b) calibration plots of CoO@c-CNTs NC/GCE to addition of carbofuran solution at scan rate of 20 mV/s in 0.1 M PBS (pH 7.0) prepared of real samples of oranges

Samples	added(µM)	Found(µM)	Recovery(%)	RSD(%)
cabbage	1.00	0.98	98.00	2.14
	2.00	1.88	94.00	2.31
	3.00	2.81	93.66	3.81
	4.00	3.94	98.50	4.11
orange	1.00	0.95	95.00	1.24
	2.00	1.78	89.00	2.22
	3.00	2.91	97.00	3.32
	4.00	3.84	96.00	4.15

Table 3. The analytical results of simultaneous determination of carbofuran in the cabbages and oranges samples (n = 4).

4. CONCLUSION

This work was conducted on preparation and characterization of nanocomposite of CoO and CNTs as electrochemical sensor for determination of the carbofuran pesticide in fruits and vegetables. To prepare nanocomposite, the centrifuged suspension of carboxylated CNTs and CoO nanoparticles was dropped on GCE and dried at room temperature. Structural studies of prepared nanocomposite using SEM and XRD exhibited the anchoring of rock-salt cubic phase of CoO NPs on c-CNTs sheets which provide an effective electrical network for stabilization of well-distributed metal oxide NPs and

higher electroactive surface area. Results of electrochemical characterizations of CoO@c-CNTs NC/GCE with CV and DPV measurements showed that the high conductivity and large effective surface area of CNTs led to the higher electron transfer rate and higher current electro-oxidation of carbofuran in CoO@c-CNTs NC/GCE than that of CoONPs/GCE. Linear range, detection limit and sensitivity values were obtained of 0-260 µM, 0.004 µM and 0.07275µA/µM for detection of carbofuran, respectively. The comparison of obtained results with the other reported carbofuran sensor showed that the detection limit and linear range of the CoO@c-CNTs NC/GCE were comparable or better than that of values obtained in some of reported carbofuran sensors. The study of interference effect of CoO@c-CNTs NC/GCE displayed the good selectivity and anti-interference ability of the CoO@c-CNTs NC/GCE for determination of carbofuran. The practical application of CoO@c-CNTs NC/GCE to determination of the carbofuran pesticide in cabbages and oranges samples revealed that the carbofuran contents in the cabbages and oranges samples were estimated 0.66 µM and 0.18 µM, respectively. Moreover, the obtained recovery (93.66 to 98.50% for cabbages and 95.00 to 97.00% for oranges samples) and RSD (2.14 to 4.11% for cabbages and 1.24 to 4.15% for oranges samples) values by the standard addition method indicated that the CoO@c-CNTs NC/GCE had good accuracy for carbofuran detection in fruits and vegetables.

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