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Multi-walled Carbon Nanotubes Modified Screen-Printed Electrode Coated Bismuth Oxide Nanoparticle for Rapid Detection of Cd(II) and Pb(II)

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Paint has a variety of excellent properties containing anti-corrosion, waterproof, oil-proof, chemical-resistant, light-resistant, temperature-resistant. The wooden furniture covered with paint is commonly used to ensure their excellent quality. However, the soluble heavy metals in the paint exist on the surface of wooden furniture, endangering human health when they are enriched in human bodies. Therefore, an effective detection method is needed to analyze the contents of heavy metals in wooden furniture. Here, we designed and manufactured a disposable screen-printed electrode (SPE) for the Cd(II) and Pb(II) determination through square wave anodic stripping voltammetry, which used a special printed ink mixed multi-walled carbon nanotube and coated with bismuth oxide nanoparticle (BONP-MSPE). The characteristics of BONP-MSPE were studied using cyclic voltammetry, electrochemical impedance spectroscopy and square-wave stripping voltammetry, and then the detecting conditions were optimized. Under the optimal conditions, the values of peak currents of Cd(II) and Pb(II) increased linearly with the concentrations of Cd(II) and Pb(II). The linear ranges were from 0.5 to 40 μg/L for Cd(II) and 0.5 to 40 μg/L for Pb(II), with detection limits of 0.5 μg/L and 0.05 μg/L (S/N = 3), respectively, Lastly, the BONP-MSPE was further employed to measure the contents of Cd(II) and Pb(II) in pint samples analysis.

Keywords: Wooden furniture, Green furniture, Lead, Cadmium, Heavy mental, Screen-Printed Electrode, Bismuth oxide

1. INTRODUCTION

Green furniture[1], also known as environmentally friendly furniture, refers to its production and recycling process in line with the environmental requirements, which will not pullet the environment and endanger the consumers’ health. In everyday usage, the wooden furniture is sprayed with paraffin or paint to ensure the excellent quality[2]. However, paraffin or paint is added some additives[3, 4], such as antifouling agents, preservatives and flame retardants, which probably exist soluble heavy metals. Besides, some metallic compounds are important pigments to adjust the colors of paint[5]. It is considered as “the brighter color of paint, the more heavy metals in paint”. If wooden furniture is spayed with inferior paint or paint, the metallic compounds will slowly evaporate into the surrounding environment. Consumers are exposed to the environment existed excessive heavy metals for a long time, especially cadmium (Cd) and lead (Pb), which will be easy to enter the human’ bodies through the respiratory system and digestive system[6]. Because these two heavy metals are difficult to be degraded by the environment and metabolized by organisms[7]. Both Cd(II) and Pb(II) can bind with proteins in the human body, and then change the enzymes’ structure that cause the loss of biological activity[8]. These changes will further damage the human brain[9], nervous system[10], reproductive system[11], internal organs[12], and bones[13]. For example, children who have long-term exposure to Cd(II) and Pb(II) can cause physical and mental developmental disorders, and older people who have long-term exposure to Cd(II) and Pb(II) can cause a variety of diseases, especially Alzheimer's disease. According to the survey by the world health organization (WHO)[14, 15], about 600,000 new intellectual disabilities in children are caused by the lead exposure every year. Therefore, it is necessary to develop a convenient, rapid and inexpensive method to detect the Cd(II) and Pb(II) in the indoor environment, which can provide the foundation to evaluate whether the levels of Cd(II) and Pb(II) exceed the safety values.

There are many analytical techniques, such as UV-spectrophotometer[16,17], X-ray fluorescence spectrometer[18], atomic fluorescence spectrometry[19, 20], inductively coupled plasma mass spectrometry[21]. These spectroscopic methods have been extensively applied in specialized laboratory test that shows high reliability, stability and precision. But the detecting devices have large volume, high price and complex operation, which are not suitable for indoor and outdoor environments to measure heavy metals [22]. Compared with these methods mentioned above, electrochemical method is an effective technology [23, 24], which has some merits including simple to use, rapid detection and low cost. It can overcome the shortages of spectroscopic methods. Over the past decades, many references indicated that concentrated on the development of electrodes to improve the sensitivity and selectivity for heavy metal ions [25].

Screen-printed electrode (SPE) [26-28] is a new structure sensor that has many advantages including small size, mass production and low price. It can avoid cross-contamination between the samples, which are much suitable to use in home environment with the portable electrochemical device. But the low sensitivity of bare SPE restricted its scope of use to measure the ultra-low contents of Cd(II) and Pb(II) in real samples. Many chemical reagents and nanomaterials[29] have been applied to modify SPE to improve the electrochemical detection performance. Multi-walled carbon nanotubes (MWCNTs) [30, 31] are commonly used in electrochemical sensors because they have special structural and characteristics, such as large surface area, chemically modifiable surface, high electrical conductivity, chemical stability and high mechanical strength, .

In this paper, we developed a new electrochemical electrode for the sensitive and selective determination of Cd(II) and Pb(II). Here, SPE was modified by MWCNTs and bismuth oxide nanoparticle (BONP-MSPE) to improve the sensitivity, of which the properties were examined by electrochemical methods. The detecting parameters of BONP-MSPE were optimized, which were served to establish the linear relationships between peak currents and different concentrations of Cd(II) and Pb(II).

2. EXPERIMENT

2.1 Chemicals and reagents

Standard solutions of Cd(II) and Pd(II) (1000 mg/L) were bought from the National Standard Reference Materials Center of China, and then diluted to the required concentrations. Multi-walled carbon nanotube (MWNT) was obtained from Xianfeng nanomaterials technology co., LTD (Nanjing, China). Bismuth oxide nanoparticle was offered by Sigma Aladdin (US). N-octylpyridinum hexafluorophosphate (OPFP, Purity>99%) was got from Chengjie Chemical co., LTD (Shanghai, China). Other reagents including cyclohexanone, acetone and N, N-dimethylformamide (DMF), were purchased from Beijing chemical factory, which were used for conductive ink. Sodium acetate-acetic acid buffer solution was selected as the supporting electrolyte for experiments. Metal compounds (potassium ferricyanide, potassium ferrocyanide, potassium chloride, and sodium hydroxide) were purchased from China Chemical Reagents Co., Ltd. (Shanghai, China). The rest of the chemicals and reagents not mentioned here were of analytical reagent grade and were used as received. Double distilled water was used throughout the experiments.

2.2 Instruments

A CHI660D electrochemical workstation was employed to collect the data of electrochemical measurements, which was produced by the CHI Instrument Company (Shanghai, China). The detecting system was consisted of the three electrodes. The BONP-MSPE was used as the working electrode, a bright platinum column was used as an auxiliary electrode and an Ag/AgCl (saturated KCl) electrode was used as a reference electrode. All electrochemical experiments were carried out at room temperature.

2.3 Principle

Square wave stripping voltammetry[32], which combines square wave and staircase potential, has a number of application in heavy metal ions detection. It needs two main steps: (i) a cathodic potential was applied on the working electrode to deposit and reduce the heavy metal ions on its surface; (ii) square wave stripping voltammetry was applied on the working electrode, and different heavy metals will give off electrons at different potentials that can be used to identify the type of heavy metals.

2.4 Fabrication of BONP-MSPE

The main steps in Figure 1 to fabricate the MSPE are similar to the previous work [33]. Briefly, 0.25 g OPPF and 1 g multi-walled carbon nanotubes are added in a solution that mixes 1.25 mL cyclohexanone and 1.25 mL acetone and dissolves the 0.025 g cellulose acetate. This mixed solution is used as ink to make the working electrode and counter electrode. The reference electrode is printed using conductive silver adhesives. Then, the MSPE need to anneal in ambient air at 80 °C for 30 min. 0.5 mg Bi_2O_3 nanoparticle is dispersing into 10 mL 0.2 wt% chitosan solution, which is prepared to modify the working electrode of MSPE and annealed at 50 °C for 30 min. Finally, MSPE coated with Bi_2O_3 nanoparticle is immersed into 0.1 mol/L KOH solution and holds on 1000 s at -1.4 V potential to reduce Bi_2O_3.

$$Bi_2O_3 + 3H_2O + 6e^- \rightarrow 2Bi + 6\ OH^-$$

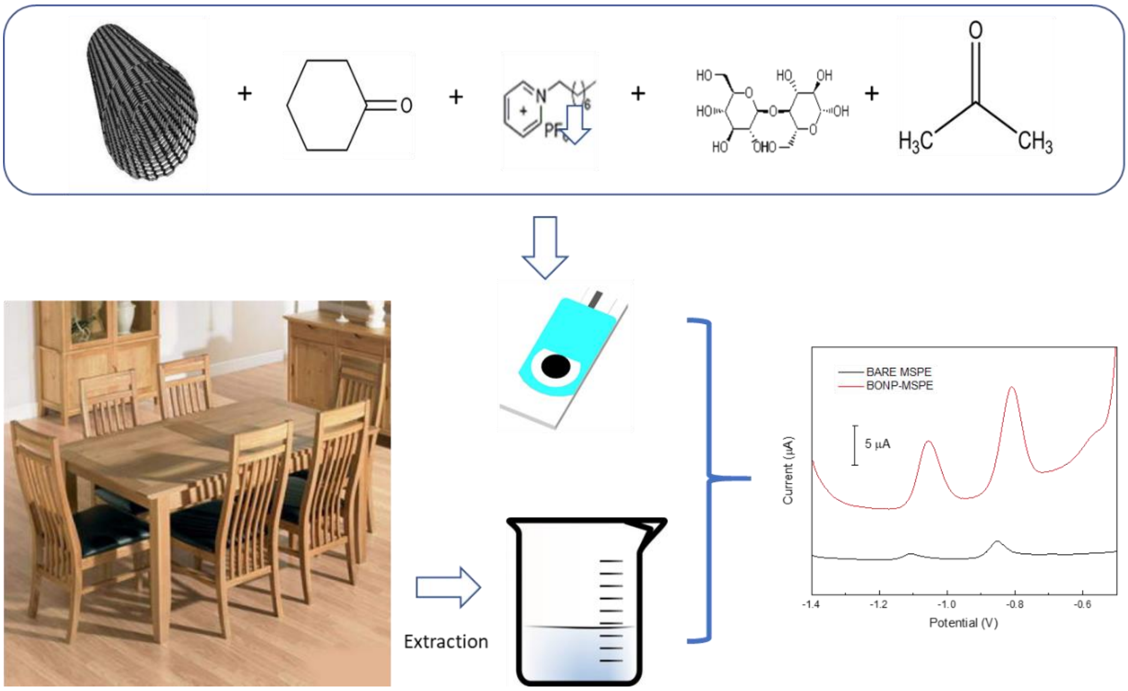

Figure 1. The structure of the detecting procedure

2.5 Measurement Procedures

The three electrodes were immersed in the 0.1 mol/L in the extracting solution of a pait sample, which the pH was adjusted by the sodium acetate buffer solution. Firstly, a negative potential of -1.4 V was performed on the working electrode for 360 s. After that, the agitator stops agitating the solution for 20 s equilibration period. Finally, square wave stripping voltammetry carried out on the working electrode and recorded the currents at different potentials.

3. RESULTS AND DISCUSSION

3.1 The characteristic of BONP-MSPE

Figure 2. shows the cyclic voltammetric signals of MSPE before and after modified BONP in a mixture solution of 5 mmol/L $[Fe(CN)_6]^{3-/4-}$ and 0.1 mol/L KCl. a redox peak (black) was observed At bare MSPE with peak potential separation as 200 mV and peak current as 80 μA, which can be ascribed to the large surface area of multi-walled carbon nanotube. After MSPE was modified with Bi_2O_3 nanoparticle and reduced to Bi nanoparticle, the redox peak current of BONP-MSPE (red) was increased a little and peak potential separation decreased in comparison with BARE MSPE, indicating that the

electron transfer at the interface was dramatically improved [34]. The reason was the conductive nanoparticle had high specific surface area [35] that can enhance the contact area between the BONP-MSPE' surface and the mixture solution of 5 mmol/L $[Fe(CN)_6]^{3-/4-}$ and 0.1 mol/L KCl.

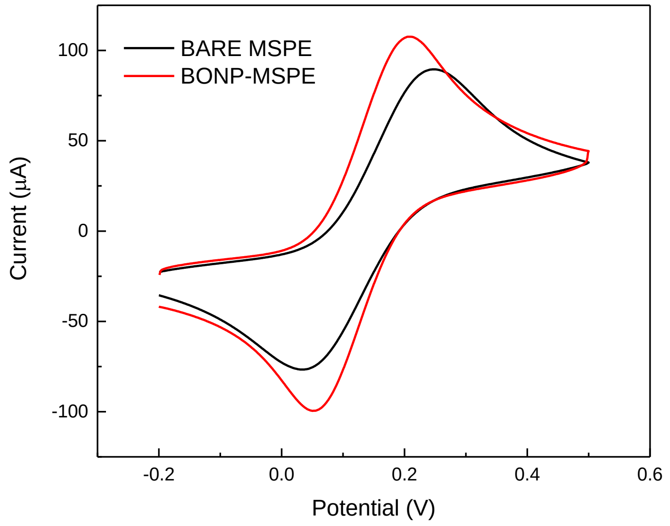

Figure 2. Cyclic voltammetric signals of BARE MSPE (black) and BONP- MSPE (red) in a mixture solution of 5 mmol/L $[Fe(CN)_6]^{3-/4-}$ and 0.1 mol/L KCl with scan rate 100 mV/s

Figure 3 shows EIS is employed to investigate the modified electrodes. The electron-transfer resistance (R_{et}) associates with the electrical conductivity of the electrode/electrolyte interface. As shown in Figure 4, the R_{et} of BARE MSPE was about 300 Ω. After Bi_2O_3 nanoparticle was coated and reduced on MSPE, the dielectric between BONP- MSPE and electrolyte was enhanced slightly [36] that R_{et} decreased to 220 Ω. This phenomenon was consistent with the result attained from the cyclic voltammetry.

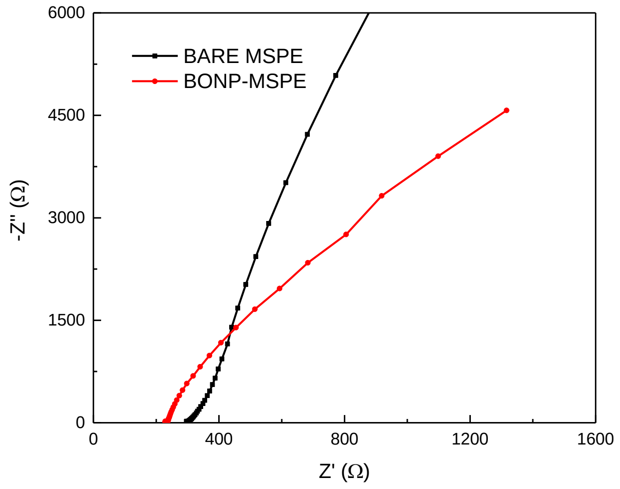

Figure 3. Electrochemical impedance spectra of BARE MSPE (black) and BONP-MSPE (red) with the frequencies from 1 to 10^5 Hz in 5 mmol/L $[Fe(CN)_6]^{3-/4-}$ and 0.1 mol/L KCl

Figure 4 shows the potential window of MPCE before and after modified with BONP in 0.1 mol/L acetate buffer solution. The hydrogen overvoltage potential of BARE MPCE (black) without reduced bismuth nanoparticles displayed relative positive, which located at -1.2 V. After MPCE modified and reduced Bi_2O_3 nanoparticle, the hydrogen evolution potential of BONP-MSPE was more negative than BARE MPCE due to the formation of a bismuth nanometer film, which was about -1.4 V. The reason was that the special crystal plane structure of bismuth on the MSPE's surface was less prone to hydrogen evolution [37]. These results demonstrated that the BONP-MSPE can be used to stripping analysis.

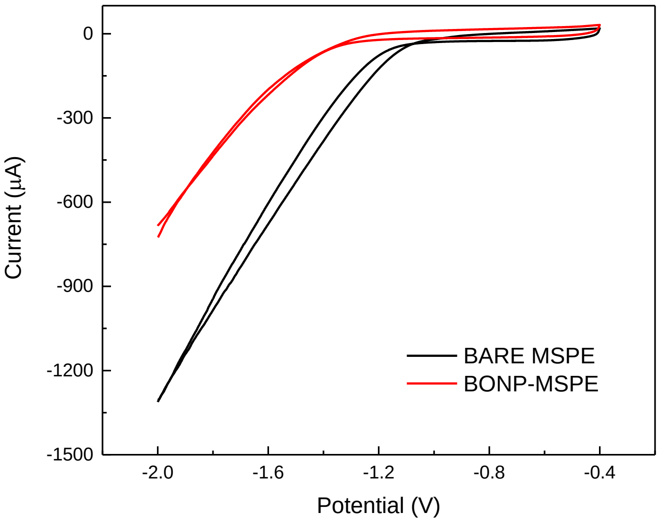

Figure 4. Cyclic voltammograms of BARE MPCE (black) and BONP-MSPE (red) in acetate buffer solution(0.1 mol/L, pH 4.5) with scan rate 50 mV/s

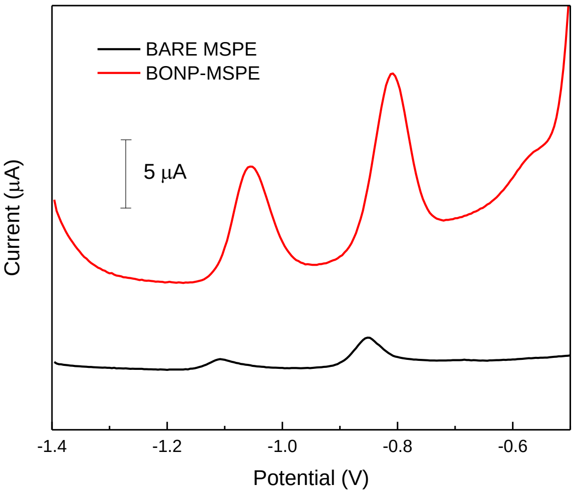

Figure 5. SWV responses of BARE MSPE (black) and BONP- MSPE (red) in 0.1 mol/L acetate buffer solution with 10 ug/L Cd(II) and 10 ug/L Pb(II)

Figure 5 shows the SWV responses of 10 μg/L Cd(II) and Pb(II) in 0.1 mol/L acetate buffer solution at BARE MPCE and BONP-MSPE, respectively. There were two unobvious current peaks located at -1.1 V and -0.85 V on the MSPE, which presented the reductive peak currents of Cd(II) and Pb(II). However, the SWV responses of BONP-MSPE was remarkably enhanced toward the Cd(II) and Pb(II) determination in compression with BARE MSPE. One reason was that the particular structure of bismuth nanoparticles enhanced contact interface with Cd(II) and Pb(II) in the solution[38]. On the other, bismuth was easy to form alloys with heavy metals that made metal ions reduced easily [39].

3.2 Optimization of experimental parameters

The detecting paramters using the BONP-MSPE were optimized in 0.1 mol/L acetate buffer solution containing 10 μg/L Cd(II) and 10 μg/L Pb(II).

Figure 6(A) shows the stripping responses of Cd(II) and Pb(II) were related with the volume of Bi_2O_3 nanoparticle solution. It was clear that the stripping responses of Cd(II) and Pb(II) were increased with the volume of Bi_2O_3 solution ranging from 1 to 3 μL, which was ascribed to the formation of bismuth nanoparticle on the electrode surface. While the volume exceed 3 μL, the stripping responses decreased because of the poor conductivity that Bi_2O_3 thick film was hard to reduce to Bi absolutely [40].

Figure 6(B) shows the relationship between the stripping responses of Cd(II) and Pb(II) and the pH of the buffer solution. BONP-MSPE was used to test 10 μg/L Cd(II) and 10 μg/L in different buffer solutions, which the pH was ranging from 3.0 to 6.0. When the pH was much lower than 4.5, the stripping responses of Cd(II) and Pb(II) were weak. The reason was that hydrogen was generated at excessively acidic circumstances on BONP-MSPE' surface that can decrease the effective contact area [40]. On the contrary, the stripping responses of Cd(II) and Pb(II) were low at higher pH due to the hydrolysis of the Bi nanoparticle [41]. Therefore, pH 4.5 was chosen for further study.

Figure 6(C) exhibits the stripping responses of Cd(II) and Pb(II) were affected by the deposition time. The stripping responses of Cd(II) and Pb(II) increased along with the deposition time from 1 to 9 min. However, the peak currents increased slowly after the deposition time exceeded 6 min. In order to improve the detection efficiency, 6 min was nominated as the optimized deposition time.

Figure 6(D) shows the stripping responses of Cd(II) and Pb(II) associated with the deposition potentials. When the deposition potential was higher than -1.4 V, the stripping responses of Cd (II) and Pb (II) were increased remarkably, which was ascribed to the weak abilities of adsorption. But the stripping responses decreased gradually when the deposition potential became more negative due to the hydrogen evolution. Thus, -1.4 V was chosen as the deposition potential.

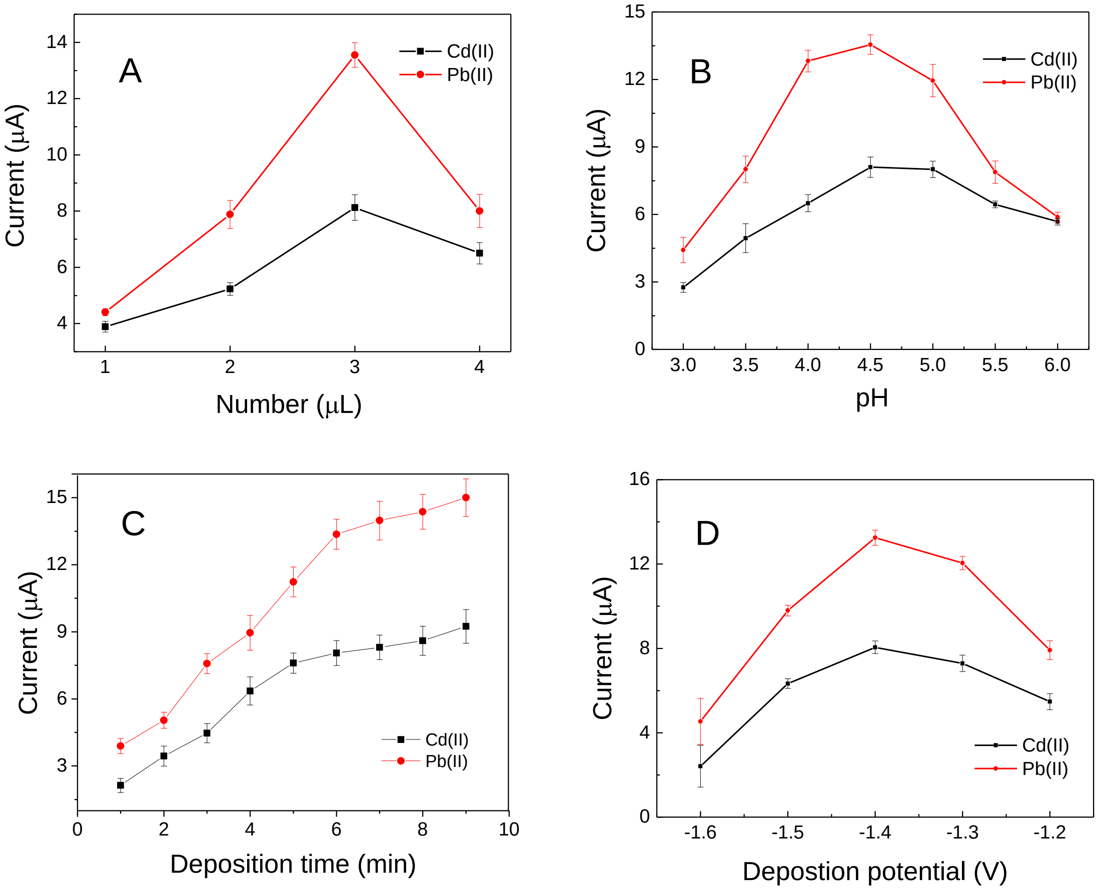

Figure 6. Effect of bismuth concentration(a), pH(b), deposition time (c) and deposition potential (don the stripping peak current of 10 μg/L Cd(II) and 10 μg/L Pb(II) on BONP-MSPE.

The square wave anodic stripping voltammetry was applied to measure the different concentrations of Cd(II) and Pb(II). Under the conditions optimized above, Figure 7 shows a series of stripping responses of Pb(II) and Cd(II) with the increasing the concentrations of Pb(II) and Cd(II). It was obvious that the peak currents of Pb(II) and Cd(II) were linear with the concentration of Cd(II) and Pb(II) in the range from 0.5 to 40 μg/L and 0.5 to 40 μg/L, which were revealed in the two insert figures. The linear regression equations were: I(μA) =0.7359+0.6027C (C: μg/L) and I(μA) =1.0682+1.5782C (C: μg/L) with the linear correlation coefficients of 0.998 and 0.997, respectively. The limits of detection were 0.22 μg/L for Cd (II) and 0.05 μg/L for Pb(II) based on three times the standard deviation of the baseline (S/N = 3).

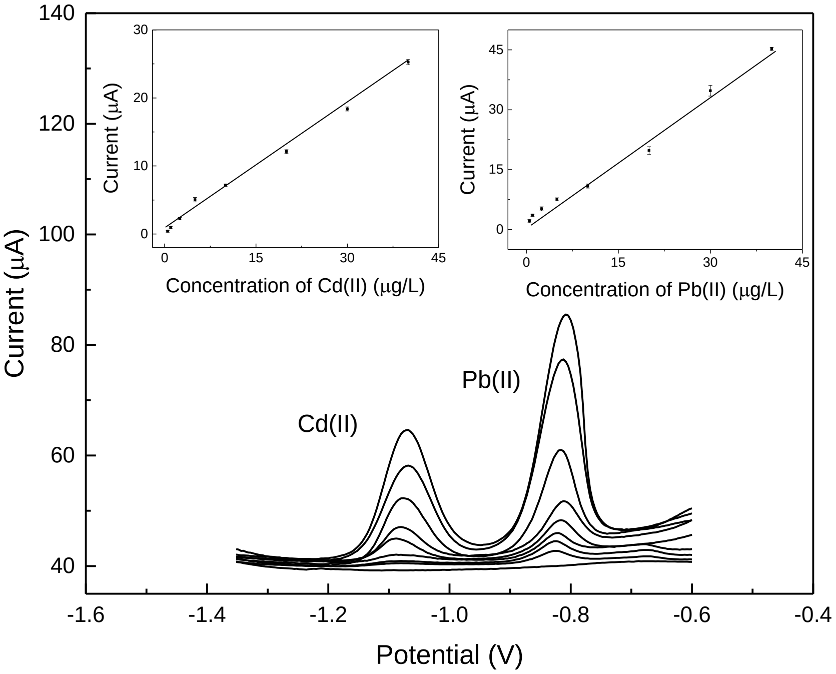

Figure 7. The square wave anodic stripping voltammetry for different concentrations of Cd(II) and Pb(II)

Compared with other reported electrodes, the detection performances for Cd(II) and Pb(II) were listed in Table 1. It was obvious that the linear range of BONP-MSPE was not particularly wide, but the limit of detection was much lower than most electrodes, which was much better than parts of the modified glassy carbon electrode. The proposed electrode can meet the requirements of the Cd(II) and Pb(II) detection.

Table 1. Comparison of performances of different electrodes for the Cd(II) and Pb(II) detection

Electrodes	Liner rang (μg/L)		Limit of detection (μg/L)		References
	Cd(II)	Pb(II)	Cd(II)	Pb(II)	
BI/MWNT-IL/SPCE	1-60	1-60	0.5	0.12	[42]
Bi/LGR/ GCE	7–120	5–120	0.47	0.41	[43]
ZnO@G /GCE	10–200	10–200	0.6	0.8	[44]
Bi_2Te_3/GO/GCE	0.2-20	0.5-20	0.1	0.2	[45]
MWCNT/SPCE	8.6–100.3	14.7–100.9	2,6	4.4	[46]
NF/G/PANI	1-300	1-300	0.1	0.1	[47]
PA/PPy/GO	5–150	5–150	2.13	0.41	[48]
Al_4SiC_4/RGO/GCE	50-2700	50-2700	2.15	1.30	[49]
MGF/GCE	2-70	0.5-110	0.5	0.1	[50]
BONP-MSPE	0.5–40	0.5–40	0.22	0.05	This work

3.3 Interference effects

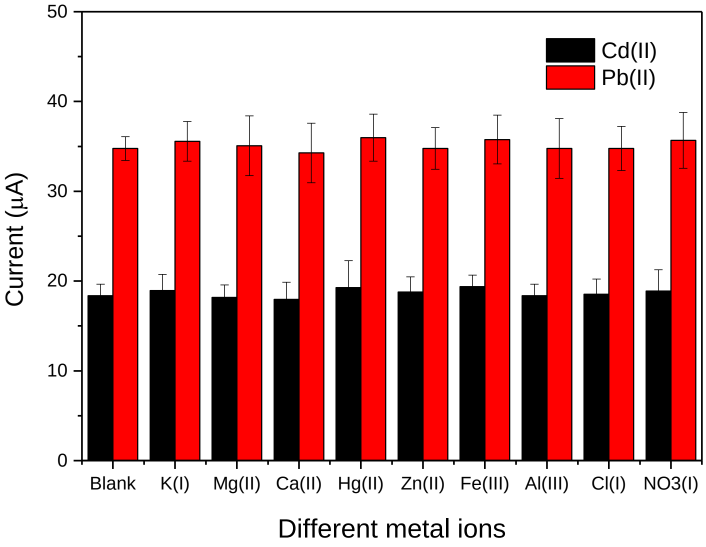

Figure 8. Selectivity of BONP-MSPE measured several coexisting ions

In paint samples, Cd(II) and Pb(II) coexist with many other ions. The interference of BONP-MSPE was important for electrochemical detection, which was explored against a standard solution containing 30 µg/L Cd(II) and 30 µg/L Pb(II) adding by adding different coexisting ions. The ions used to study the interfering effect were K(I), Na(I), Mg(II), Ca(II), Zn(II), Fe(III), Al(III), Cl(I) and NO_3(I) with all concentrations 100 folds that of Cd(II) and Pb(II). All of results were shown in Figure 8, which confirmed that there was no interference from any of the mentioned ions in the detection of Cd(II) and Pb(II) using BONP-MSPE.

3.4 The stability of BONP-MSPE

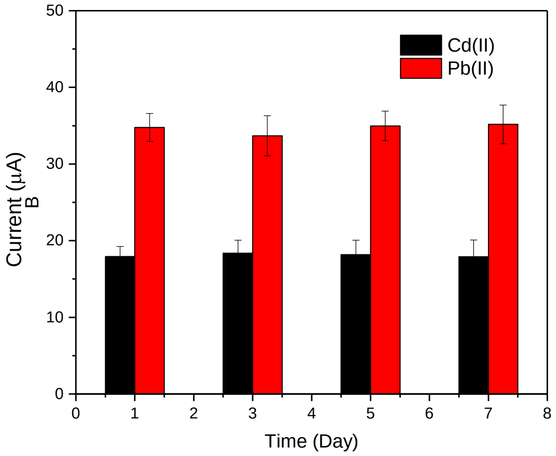

Figure 9. BONP-MSPE for the 30 µg/L Cd(II) and 30 µg/L Pb(II) detection every two days

To study the stability, three BONP-MSPE were fabricated following the step in 2.4, which were stored at room temperature. BONP-MSPE was measured the 30 µg/L Cd(II) and 30 µg/L Pb(II) solution every two days, and results shown in Figure 9. We found that the stripping responses were almost no change at first seven days, indicating BONP-MSPE had good stability.

3.5 Application for the sample analysis

To evaluate the applicability and feasibility, Cd(II) and Pb(II) in various paint samples was determined by the proposed electrode.

For paint samples, (a) scraping the paint from the floor surface was ground and sieved using 0.5 mm sifter; (b) weighing 1 g paint mixed with 100 mL of a hydrochloric acid solution; (c) the mixture was sonicated at 37 °C for 1 h; (d)the supernatant was filtered, and then adjusted the pH by 0.1 mol/L acetate buffer solution and 0.1 mol/L sodium hydroxide solution. These extracting solutions were detected by flame atomic absorption spectrometry (FAAS) and the BONP-MSPE, which were listed in Tab. 2 and Tab. 3. It can be seen that the recoveries of Cd(II) and Pb(II) in extracting solutions was ranging from 92.39 to 107.87%, indicating that these two methods were no significant difference for the Cd(II) and Pb(II) detection.

Table 2. Results for the Pb(II) determination in various paint samples using the proposed electrode (N = 3).

Sample	Add (mg/kg)	Found [a] (mg/kg)	FAAS [a] (mg/kg)	Recovery (%)
1	-	3.38±0.34 [b]	3.58	94.43
	5	8.84±0.41		103.03
2	-	1.14±0.27	1.23	92.68
	5	6.26±0.29		100.48
3	-	2.65±0.21	2.59	102.31
	5	7.21±0.33		95.37
4	-	2.34±0.28	2.21	105.58
	5	7.55±0.30		104.71
5	-	1.32±0.11	1.23	107.31
	5	6.25±0.45		100.32

a. SWASV and AAS measurements were repeated three times (n=3)

b. Mean value±standard deviation

Table 3. Results for the Cd(II) determination in various paint samples using the proposed electrode (N = 3).

Sample	Add (mg/kg)	Found [a] (mg/kg)	FAAS [a] (mg/kg)	Recovery (%)
1	-	0.85±0.11[b]	0.92	92.39
	5	6.08±0.36		102.70
2	-	1.37±0.15	1. 27	107.87
	5	6.49±0.57		103.51
3	-	-	0.13	-
	5	5.28±0.35		102.92
4	-	1.27±0.09	1.35	94.07
	5	6.55±0.49		103.15
5	-	0.71±0.05	0.76	93.42
	5	5.78±0.31		100.34

a. SWASV and AAS measurements were repeated three times (n=3)

b. Mean value±standard deviation

4. CONCLUSION

A disposable screen-printed electrode for the trace level detection of Cd(II) and Pb(II) was developed using the SWV technique. The proposed electrode exhibited good selectivity, high sensitivity, low detection limit, high repeatability and stability in the view of the good conductivity and high chemical stability of OPPF and MWNT. In addition, the proposed BONP-MSPE can be successfully applied in simultaneous detection of Cd(II) and Pb(II) in real samples, which had a good application foreground.

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