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An electrochemical sensor for stripping analysis of Pb(II) based on multiwalled carbon nanotube functionalized with 5-Br-PADAP

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Abstract The present study serves to focus attention on the modification of multiwalled carbon nanotube with 2-(5-bromo-2-pyridylazo)-5-diethylaminophenol (5-Br-PADAP) and its application for the development of a new, simple, and selective modified electrode in order to determine Pb (II) in standard alloys and water samples. The electrochemical method was based on open circuit accumulation of lead ions onto a 5-Br-PADAP-modified multiwalled carbon nanotube electrode and then their anodic stripping voltammetric determination. The linear range for Pb(II) ions was from 0.9 to 114.6 $\mu g L^{-1}$. The detection limit and relative standard deviation were found to be 0.1 $\mu g L^{-1}$ and 1.6%, respectively. This method demonstrates the powerful application of carbon nanotubes in the field of mercury-free electrodes in voltammetric stripping analysis.

Keywords Multiwalled carbon nanotube · 5-Br-PADAP · Lead(II) · Stripping voltammetry

Introduction

Much attention has been paid to the monitoring of low levels of lead in the environment as its consequence is a serious cumulative body poison and is capable of entering through air, water, and food. This metal exhibits severe deleterious effects on the human body. In particular, it strongly affects the mental and physical growth of children.

Acute lead poisoning in human beings causes severe damage in the liver, brain, kidneys, reproductive system, and central nervous system; and even sometimes causes death. Mild lead poisoning causes anemia and headache. Moreover, the victim may feel fatigued and irritable. Chronic exposure to lead causes nephritis, scarring, and the shrinking of kidney tissues [1–3]. Therefore, several analytical methods are available for analyzing the lead concentration in real samples such as flame [4] and electrothermal atomic absorption spectrometry [5, 6], hydride generation atomic fluorescence spectrometry [7, 8], inductively coupled plasma spectrometry [9], and stripping voltammetry [10–30].

In recent years, adsorptive stripping voltammetry techniques are known to be very useful methods to determine trace elements. Compared to other techniques, these techniques accelerate the analysis, provide condition for good selectivity and sensitivity, excellent accuracy and precision, and low costs of instrumentation and maintenance [10–30]. Traditional working electrodes for voltammetric determination were generally based on a hanging drop mercury electrode or a mercury film electrode. However, the toxicity of the Hg and its precursors makes these materials undesirable for being disposable in situ sensing applications. Therefore, various chemically modified electrodes such as mercury-free electrodes are used for adsorptive stripping voltammetry of heavy metal ions [10–30]. Adsorptive stripping voltammetry is generally applied in an accumulation step prior to the voltammetric scan in order to develop analysis methods with higher levels of sensitivity. In this step, analyte (metal ion) is adsorbed from sample solution to electrode surface based on complexation between metal ion and ligand that is immobilized on the electrode surface. Owing to the strong sorption properties of carbon nanotubes and their advantages in

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electrochemical measurements in such a situation, the nano-structured material has lead to some novel methods of stripping analysis [10–16, 26].

In the present study, the multiwalled carbon nanotubes (MWCNTs) that were functionalized with a complex reagent, 2-(5-bromo-2-pyridylazo)-5-diethylaminophenol (5-Br-PADAP), was prepared and coated on a glassy carbon electrode. This modified electrode was successfully used for anodic stripping determination of lead(II) in some real samples.

Experimental

Reagents

The grade of all reagents used was analytical and they were used without further purification. 5-Br-PADAP was purchased from Sigma and MWCNTs (95% purity) with an average outer diameter of 3–20 nm, length of 1–10 μm, number of walls 3–15, and surface area of 350 $m^2 g^{-1}$ were prepared from Palasmachem GmbH (Berlin, Germany). The stock solution of 1,000 $mg L^{-1}$ Pb(II) was prepared from $Pb(NO_3)_2$ in doubly distilled water (Fater Electronic Co., Iran). Doubly distilled water was used for all experiments.

Apparatus

Infrared spectrum of the sample was recorded with Brucker FTIR spectrophotometer model TENSOR 27 using a sample on the KBr plate. All voltammograms were recorded with a three-electrode system consisting of an Ag/AgCl as the reference electrode, a platinum wire as the counter electrode, and a glassy carbon electrode (GCE; 2 mm diameter, Azar Electrode Co., Iran), modified with presented procedure as the working electrode. Voltammetric experiments were performed using a Metrohm electro-analyzer (Model 757 VA Computrace). The measurements were recorded using VA Computrace version 2.0 (Metrohm) that run under Windows XP. A Metrohm 710 pH meter was used for pH adjustments. A stirrer (Jenway, Germany) was used to stir solutions in the accumulation step. All measurements were carried out at room temperature (20±1 °C).

Functionalization of MWCNTs with 5-Br-PADAP

Before being used, the MWCNTs were purified and pretreated in order to remove graphitic nanoparticles, amorphous carbon, and catalyst impurities. After that, they were functionalized with carboxylic acid groups [26, 31], in the way that raw MWCNTs were heated at 300 °C for 1 h to remove amorphous carbon and then were refluxed in conc. nitric acid for 5 h. The black MWCNTs suspension was also filtered and then was washed with doubly distilled water until neutral pH was achieved. The treated MWCNTs were dried at 80 °C and stored for further use. After these procedures, the carboxylic acid groups were brought in the crosssections of the MWCNTs. In order to prepare 5-Br-PADAP-functionalized MWCNTs, the 5-Br-PADAP dissolved in ethanol was added to 1 g of pretreated MWCNTs drop-wise, and the solution was stirred for 12 h till it became colorless. After that, the modified MWCNTs were filtered and were washed with doubly distilled water. Then, they were dried at room temperature. After these steps, the 5-Br-PADAP was physically adsorbed onto the MWCNTs surface.

Preparation of 5-Br-PADAP/MWCNT/GCE

Ultrasonication agitation for 30 min was applied to disperse 5.0 mg 5-Br-PADAP/MWCNT into 10 ml of double distilled water in order to give 0.5 mg/ml suspension. Before coating, the GCE was polished with a nano-Al_2O_3 powder (Fluka) on a polishing pad (Metrohm) that was rinsed thoroughly with doubly distilled water, and then was sonicated in acetone for 2 min. Finally, the GCE was coated with 5 μL of 0.5 mg/ml 5-Br-PADAP/MCNT suspension, and the water was allowed to evaporate at room temperature in the air. The blank electrode was prepared using the same procedure with the exception of using unfunctionalized MWCNTs.

### Stripping determination of Pb(II) on 5-Br-PADAP/MWCNT/GCE

For differential pulse anodic (DPA) stripping voltammetric experiments, the 5-Br-PADAP/MWCNT/GCE was immersed in 25 ml of sample solution (0.1 M acetate buffer, pH 5.6) that contained a known amount of Pb(II). The solution was stirred for 9 min. This was followed by medium exchange to a 0.1-M stripping solution (0.1 M KNO_3, 0.1 M acetate buffer, pH 4.2) where the DPA voltammogram was recorded from −1.0 to −0.2 V (with 12.5 $mV s^{-1}$ scan rate, 50 mV pulse amplitude, and 4 ms pulse period).

Results and discussion

Spectral characteristics of the 5-Br-PADAP/MWCNT

In Fig. 1, typical FT-IR spectral patterns were shown for unmodified MWCNTs, 5-Br-PADAP, and 5-Br-PADAP/

Fig. 1 FT-IR spectra for
a unmodified MWCNTs,
b 5-Br-PADAP, and
c 5-Br-PADAP/MWCNTs
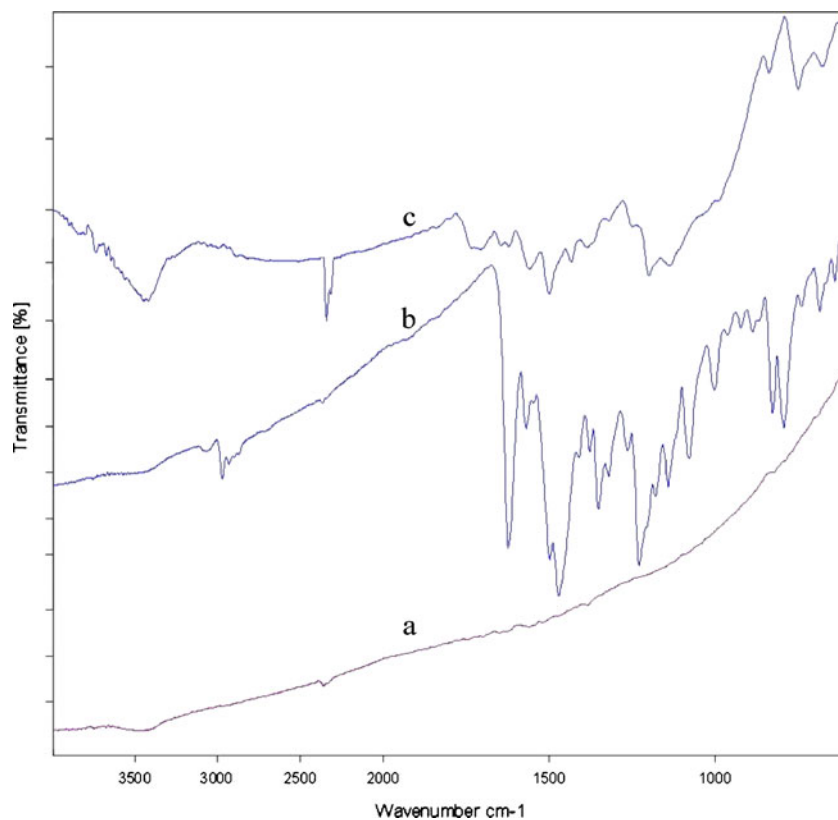
MWCNTs. The characteristic bands of 5-Br-PADAP (curve b) can be observed in spectrum of MWCNT modified with 5-Br-PADAP (curve c). It was also observed from the spectra that there was a slight change in the band positions of Fig. 1c, suggesting a weak interaction between the 5-Br-PADAP and MWCNT.

Electrochemical behavior of Pb(II) at 5-Br-PADAP/MCNT/GC electrodes

Figure 2a shows typical cyclic voltammogram for the detection of Pb^{2+} following open-circuit accumulation onto
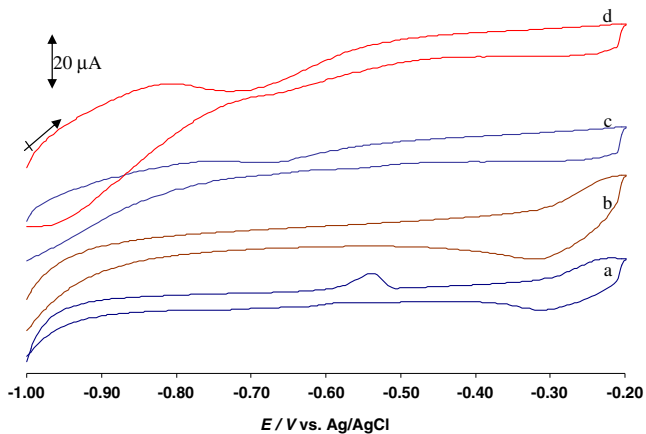

Fig. 2 Cyclic (50 mV/s) voltammograms in acetate buffer pH 4.2 after open circuit accumulation in acetate buffer pH 5.6: **a** 5-Br-PADAP/MWCNT/GCE,with 100 $\mu g L^{-1}$ Pb(II) in accumulation medium, **b** 5-Br-PADAP/MWCNT/GCE, no Pb(II) in accumulation medium, **c** MWCNT/GCE, with 100 $\mu g L^{-1}$ Pb(II) in accumulation medium, and **d** MWCNT/GCE, no Pb(II) in accumulation medium. Preconcentration time was 9 min and a reduction time of 15 s in −1.0 V was applied before CVs
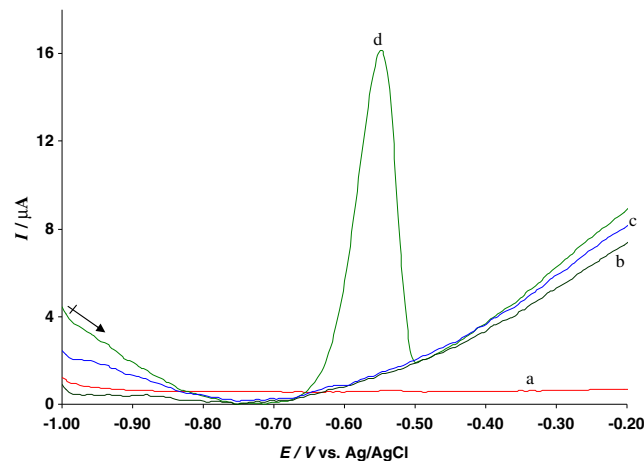

Fig. 3 DPA voltammograms of 5-Br-PADAP/MWCNT/GCE in acetate buffer solution after lead accumulation in different media with pH 5.6: **a** 0.1 M citrate, **b** 0.1 M oxalate, **c** 0.1 M phosphate, and **d** 0.1 M acetate. Other conditions were the same as in Fig. 2
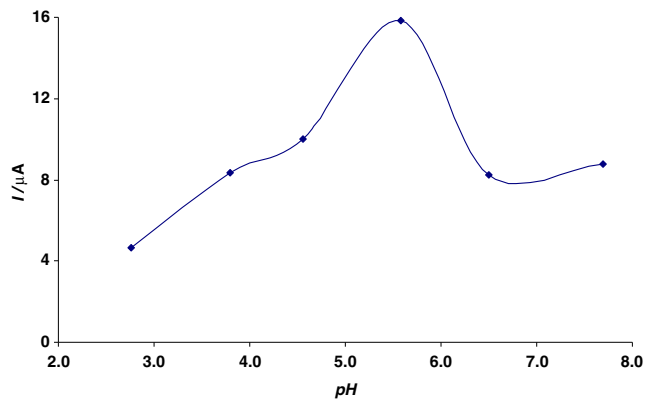
Fig. 4 The pH effect of accumulation media on the DPA currents. Pb(II) concentration was 100 μg L^{-1} and other conditions were the same as in Fig. 2

5-Br-PADAP/MWCNT film. Figure 2b is also under the same condition but in the absence of Pb^{2+} in accumulation medium.

As can be seen in Fig. 2a, there is an anodic peak at −0.55 V, which is related to the oxidation of lead(0). To prove the importance of 5-Br-PADAP as a chelator reagent in the electrode surface, GC electrode that was modified with MWCNTs without 5-Br-PADAP was prepared and was used for the accumulation and stripping of Pb^{2+}. Figs. 1d and 2c show no significant peak in the case of unmodified MWCNTs both in the presence and absence of Pb(II), respectively. These results show the importance of modification of MWCNTs with 5-Br-PADAP for the preconcentration of Pb^{2+}.

Based on the above observations and under the conditions of the experiments, the possible pathways for the analysis cycle from the modification of MWCNTs to the stripping voltammetry are postulated below ("sol" and "surf" subscripts mean that compound is in solution or surface):

Modification of pretreated-MWCNTs with 5-Br-PADAP:

$$MWCNT + 5-Br-PADAP_{(sol)} \xrightarrow{stir} 5-Br-PADAP/MWCNT \tag{1}$$

Accumulation of Pb^{2+} from acetate buffer solution containing Pb^{2+}:

$$Pb^{2+}_{(sol)} + [5-Br-PADAP/MWCNT]_{(surf)} \xrightarrow{open\ circuit} [Pb^{2+}/5-Br-PADAP/MWCNT]_{(surf)} \tag{2}$$
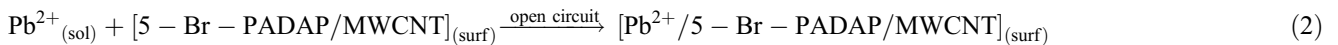
Fig. 5 DPA voltammograms of accumulated lead(II) at 5-Br-PADAP/MWCNT/GCEs with different start potentials. *Inset* the relationship between DPA currents of Pb(II) and start potential of voltammograms. Other conditions were the same as in Fig. 2
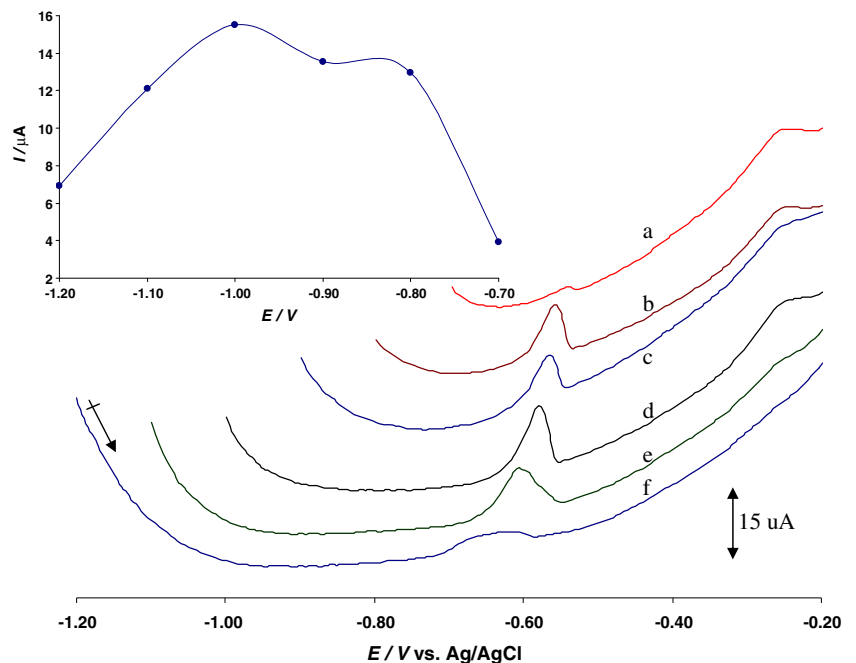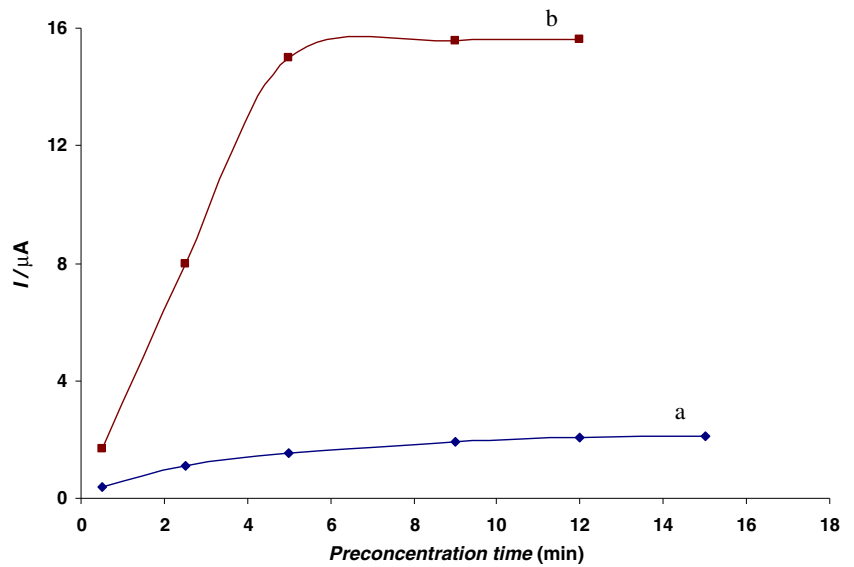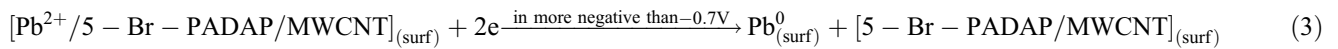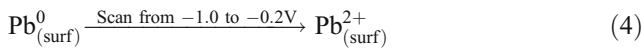
Fig. 6 Plot of DPA peak currents vs. accumulation time. Pb(II) concentrations were **a** 10 and **b** 100 $\mu g L^{-1}$ and other conditions were the same as in Fig. 2
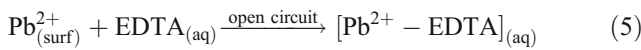
Reduction of accumulated Pb^{2+} in clean acetate buffer:

$$[Pb^{2+}/5 - Br - PADAP/MWCNT]_{(surf)} + 2e \xrightarrow{\text{in more negative than} -0.7V} Pb^0_{(surf)} + [5 - Br - PADAP/MWCNT]_{(surf)} \quad (3)$$

Stripping of reduced lead in clean acetate buffer:

$$Pb^0_{(surf)} \xrightarrow{\text{Scan from} -1.0 \text{ to } -0.2V} Pb^{2+}_{(surf)} \quad (4)$$

Regeneration of electrode in EDTA solution:

$$Pb^{2+}_{(surf)} + EDTA_{(aq)} \xrightarrow{\text{open circuit}} [Pb^{2+} - EDTA]_{(aq)} \quad (5)$$

Effective parameters on the electrode signal

The effects of different experimental parameters on the stripping voltammetry of the lead (stripping media, kind, and pH of accumulation media, start potential, reduction time, accumulation time, and amount of MWCNTs) have been tested by DPA. A 0.1-M potassium nitrate and 0.1-M

Fig. 7 DPA stripping voltammograms of 5-Br-PADAP/MWCNT/GCEs after accumulation of lead from different lead (II) concentrations of (*a–j*): 0.93, 4.60, 10.40, 22.2, 32.3, 46.90, 62.20, 80.90, 96.90, and 114.6 $\mu g L^{-1}$ Pb^{2+} in accumulation medium. *Inset* the related calibration graph. Other conditions were the same as in Fig. 2
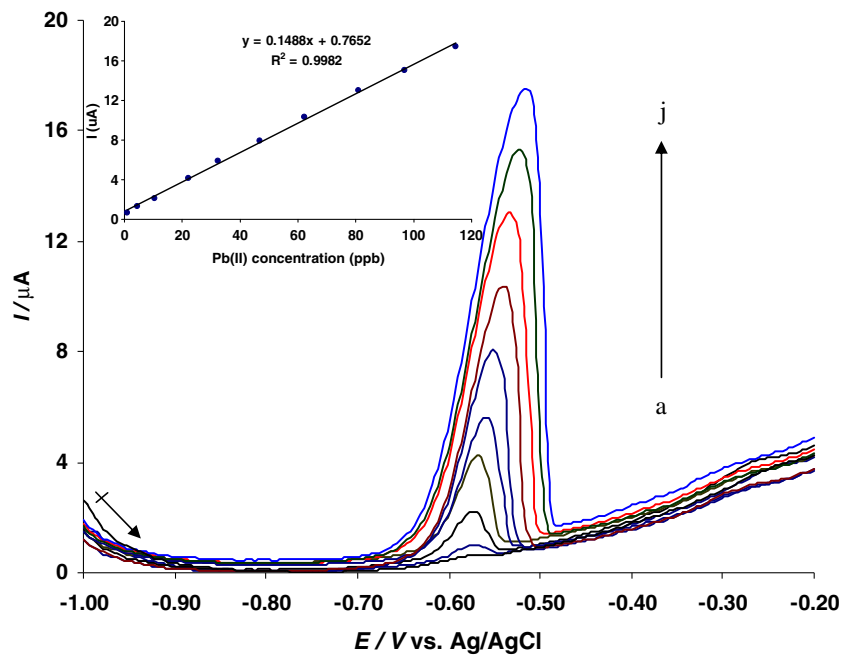
Table 1 Effect of diver ions on the lead determination

Salt or ion	Recovery (%)
KNO$_3$, NaNO$_3$, Cd(II)	100
NaCl	99
Na$_2$EDTA	26
Na$_2$S$_2$O$_3$	39
Bi(III), Cu(II)	98
Zn(II)	101
Al(III)	102
Fe(II), Hg(II), Co(II), Mn(II)	99
Fe(III), Ni(II)	97

Lead and interference concentrations are 50 and 5,000 μg L^{-1}, respectively, and other conditions are the same as in Fig. 2

acetate buffer solution adjusted to pH 4.2 were used as they give best peak shape and sensitivity.

As the accumulation of lead ions at the electrode surface was based on the complex formation with 5-Br-PADAP, the amount of coated 5-Br-PADAP in the surface was expected to have a significant influence on the voltammetric response. This amount can be controlled by changing the concentration of 5-Br-PADAP/MWCNT in the suspension used for modification of GCE. The anodic peak currents increased as the concentration of 5-Br-PADAP/MWCNT increased from 0.25 to 0.50 mg/ml. The increase in the currents may be related to the increase in surface area of the complex agent (5-Br-PADAP). The peak currents decreased when the concentration exceeded because the mass transport and charge transfer rate may have decreased when the 5-Br-PADAP/MWCNT film was too thick. Thus, the optimized amount of MWCNTs suspension was chosen as 5 μL of 0.50 mg/ml.

As can be seen in curves a to c in Fig. 3, no voltammetric peaks were observed when oxalate, citrate, or phosphate buffers were used to accumulate Pb(II). However, a good anodic peak can be obtained when acetate buffer was used as accumulation medium (Fig. 3d).

The chelating properties of 5-Br-PADAP with metal cations have been demonstrated to be pH-dependent [5, 32–36]. Therefore, the effect of pH on stripping peak current was studied in the range of 2.8–7.7 as shown in Fig. 4. The stripping peak currents were found to increase as the pH of the accumulation solution increased up to pH 5.6. In this case, peak potential of the voltammograms was constant because the pH of the stripping media was constant. Similar behaviors have been reported previously using 5-Br-PADAP as a complex agent for determination of lead [5, 32]. A pH of 5.6 was selected for the accumulation of lead.

The influence of start (or reduction) potential on the anodic peak current of lead(II) was studied by varying the start potential from −0.7 to −1.2 V. As shown in Fig. 5, when the potential was increased to −1.0 V, a well-defined peak that had the highest peak current was obtained. Hence, −1.0 V was employed as an optimum reduction potential for further studies.

Also, the effect of reduction time was examined. By increasing the reduction time, the peak current of lead(II) was found to be constant after 15 s. Therefore, voltammograms were recorded with applying the reduction time of 15 s.

When a 5-Br-PADAP/MWCNT/GCE is immersed in a solution containing lead(II), interaction occurs between the lead ions and 5-Br-PADAP. The longer the electrode is exposed to the solution containing the analyte, the more lead(II) is accumulated on the surface, which consequently yields a higher current. We studied the dependence of the anodic stripping peak current on the preconcentration time for two different Pb(II) concentrations. The peak current was found to increase with increasing preconcentration time, indicating an enhancement of Pb(II) uptake at the electrode surface. Normally, the increase in the response current continues until a maximum signal level (presumably corresponding to either saturation or an equilibrium surface coverage) is attained. As shown in Fig. 6, the attainment of a steady

Table 2 Determination of Pb(II) in standard alloys

Sample	Certified concentration of Pb(II) (mgg^{-1})	Found Pb(II) (mgg^{-1})	Recovery (%)
NKK no. 916, aluminum alloy	0.40	0.39±0.01	97.5
Composition%: Si, 0.41; C, 0.41; Fe, 0.54; Mg, 0.1; Cr, 0.05; Sn, 0.05; Zn, 0.3; Ti, 0.1; Pb, 0.04; Sb, 0.01; B, 0.0006; Zr, 0.05; Co, 0.03; Mn, 0.11; Bi, 0.03; Ni, 0.06; V, 0.02; Cu, 0.27			
NKK no. 1021, Al, Si, Cu, Zn alloys	1.80	1.74±0.04	96.6
Composition%: Pb, 0.18; Zn, 1.76; Si, 5.56; Sn, 0.10; Mg, 0.29; Ti, 0.04; Cr, 0.03; Fe, 0.99; Bi, 0.01; Zr, 0.01; Sb, 0.01; Ca, 0.004; Mn, 0.20; Ni, 0.14; V, 0.007; Cu, 2.72			

Table 3 Determination of Pb(II) in various waters

Sample	Present method ($\mu g L^{-1}$)[a]	Spiked Pb^{2+} ($\mu g L^{-1}$)	Found Pb^{2+} ($\mu g L^{-1}$)[b]	Recovery (%)
Spring water [b]	No detect	10.0	10.4±0.2	104.0
		100.0	101.8±1.9	101.8
Drinking water[c]	No detect	10.0	10.3±0.2	103.0
		100.0	101.3±1.8	101.3
Waste water[d]	9.2±0.3	10.0	18.9±0.5	97.0
		100.0	108.3±2.3	99.1

[a] Mean of three determinations±standard deviation

[b] Prepared from Hossein Abad spring, Sirjan, Iran

[c] Prepared from Shahid Bahonar University, Kerman, Iran

[d] Prepared from Golegohar factory, Sirjan, Iran; after dilution by 200 times

state accumulation level of Pb(II) at the electrode surface requires an exposure time of 5 min for 100 $\mu g L^{-1}$ and 9 min for 10 $\mu g L^{-1}$ Pb^{2+}. Hence, preconcentration time of 9 min was employed in all subsequent experiments.

Calibration curve, detection limit, reproducibility, repeatability, and selectivity

Standard solutions containing different concentrations of Pb(II) were prepared in pH-5.6 solutions and adjusted to the optimized anodic stripping voltammetric procedure. Voltammograms at these concentrations are shown in Fig. 7. A linear calibration graph was obtained in the concentration range of 0.9–114.6 $\mu g L^{-1}$ (inset, Fig. 7). The detection limit was found to be 0.1 $\mu g L^{-1}$ Pb(II) and for five successive determinations of 100 $\mu g L^{-1}$ Pb(II), the relative standard deviation was 1.6%.

Ability of electrode to regenerate was tested based on stable complex formation between EDTA and lead ions. Results showed that a used electrode can be completely regenerated after at least 1.5 min in 0.1 M EDTA solution. There is no significant difference in the response of a regenerated electrode compared to a new one. The response

of regenerated electrodes was found to decrease only 2.1% even after ten successive regeneration/measurement cycles.

The presence of other metal ions would interfere with Pb(II) determination if they compete for complexation with 5-Br-PADAP in accumulation step and also if their peak overlaps with lead(II) peak. Various salts and metal ions were added individually to a solution containing 50 $\mu g L^{-1}$ of lead and the general procedure was applied. Recoveries are given in Table 1. Among the salts and metal ions examined, most of them did not interfere at the high levels of concentration. As can be seen in Table 1, the presence of $S_2O_3^{2-}$ or EDTA caused high depressions of the Pb(II) peak. However, these compounds were not present in tested samples. Thus, the method is selective and useful for determination of Pb(II) in real samples.

Validity of method

Various standard reference materials were analyzed for checking the reliability of method. Standard reference materials including Nippon Keikinzoku Kogyo (NKK) CRM, no. 916 aluminum alloy, and no. 1021 Al, Si, Ca, Zn alloys were analyzed for lead. The 0.20-g standard alloy

Table 4 Comparison of some properties of the present work with other studies

No.	Electrode surface	t_a (min)	D. L. ($\mu g L^{-1}$)	L. R. ($\mu g L^{-1}$)	RSD (%)	Interferences	Ref.
1	MWCNT–Nafion	2	1.0	16.5–1,243	6.0	Cu(II), Hg(II)	[10]
2	Bismuth-modified MWCNT	5	1.3	2–100	5.3	Cu(II), Benzene, Xylene	[12]
3	Dihydroxyl hydrogen phosphate–MWCNT	5	0.8	4.1–2,070	4.5	Hg(II)	[13]
4	MWCNT–nafion/bismuth composite	2	0.025	0.05–100	5.0	SDS, Hg(II), TBAB, Triton X100	[14]
5	MWCNT paste	3	6.6	58.4–646.2	5.6	Ni(II), Sb(II), Cu(II), Co(II), Cr(II)	[16]
6	5-Br-PADAP/MWCNT	9	0.1	0.9–114.6	1.6	EDTA, $S_2O_3^{2-}$	This work

t_a accumulation time, *D.L.* detection limit, *L.R.* linear range

was dissolved completely in 10 mL hydrochloric acid (1+ 1) by heating it on a water bath and then 1 mL of 30% (*v/v*) hydrogen peroxide was added to it. The excess of peroxide was decomposed by heating the sample on a water bath. The solution was cooled, filtered, and diluted to 100 mL distilled water in a standard flask. An aliquot of this sample was added to 20 mL of 0.1 M acetate buffer with pH 5.6, and general procedure was used on resultant solution. The obtained results are given in Table 2. These results are in agreement with certified values and confirm that the proposed method has good accuracy.

Real samples

For the analysis of different kinds of water, such as drinking, spring, and waste water, their samples were exchanged with distilled water used for the preparation of acetate buffer (pH 5.6). The general procedure was used for these resultant solutions. The standard addition method was applied for the determination of lead in all real samples. In order to evaluate the validity of the proposed method for the determination of Pb^{2+}, recovery studies were carried out on samples to which a distinct amount of Pb^{2+} was added. The results are given in Table 3. As can be seen, the ability of the method for the analysis of lead in real matrix is satisfactory.

Comparison to other methods

We compared the results obtained in this work with results obtained using other MWCNT-modified electrodes [10, 12–14, 16]. In comparison with the results reported by other research groups, our results show that linear range in this modified electrode is wide and detection limit is good. Furthermore, reproducibility is better than other works and selectivity is good (see Table 4).

Conclusion

We have demonstrated that GCE modified with 5-Br-PADAP/MWCNT can be used for the stripping voltammetry of lead(II). The ability of the 5-Br-PADAP to extract lead(II) into the electrode surface leads to an electrode that is precise and accurate. The analyte ion may be preconcentrated by chemical interaction with the electrode at open circuit potential. This method showed a high degree of selectivity because of the complexation with 5-Br-PADAP and the exchange of the medium. The modified electrode

prepared in this study can be used for the determination of lead in standard alloys and water samples with good results.

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