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Use of nickel implanted boron-doped diamond thin film electrode coupled to HPLC system for the determination of tetracyclines☆

Surudee Treetepvijit[a], Anchana Preechaworapun[a], Narong Praphairaksit[a], Suchada Chuanuwatanakul[a], Yasuaki Einaga[b], Orawon Chailapakul[a,*]

[a] *Materials Chemistry and Catalysis Research Unit, Department of Chemistry, Faculty of Science, Chulalongkorn University, Patumwan, Bangkok 10330, Thailand*
[b] *Department of Chemistry, Faculty of Science and Technology, Keio University, 3-14-1 Hiyoshi, Yokohama, Kanagawa 223-8522, Japan*

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Abstract

The electrochemical analysis of tetracyclines was investigated using nickel-implanted boron-doped diamond thin film electrode (Ni-DIA) by cyclic voltammetry and high performance liquid chromatographic with amperometry. Cyclic voltammetry was used to study the electrochemical oxidation of tetracyclines. Comparison experiments were carried out utilizing as-deposited BDD and glassy carbon electrodes. Ni-DIA electrode provided well-resolved oxidative irreversible cyclic voltammograms and the highest current signals among the electrode studied. High performance liquid chromatography (HPLC) with amperometric detection was also studied. The chromatography was performed using a commercially available Inertsil C18 column, with the mobile phase being: 80% phosphate buffer (pH 2.5)–20% acetonitrile and detected at 1.55 V. The methods were validated over the concentration range 0.05–100 ppm with the overall average recoveries from 83.3 to 102.5% and R.S.D. of less than 10%. The proposed method was further applied to analyse shrimp samples.

Keywords: Tetracyclines; Nickel-implanted boron-doped diamond; Cyclic voltammetry; Flow injection system; HPLC; Amperometric detection

1. Introduction

Conductive boron-doped diamond electrodes (BDD) have attracted tremendous interest for various electrochemical applications, including electroanalysis [1], electrosynthesis [2] and electrochemical treatment of wastewater [3]. BDD electrodes have recently attracted a great deal of attention due to their superior properties, which are significantly different from those of other conventional electrodes, e.g. glassy carbon or platinum electrode materials [4,5]. The growing popularity of this boron-doped diamond electrode in analytical applications over its conventional counterparts lies mainly on its various attractive features, such as very low and stable voltammetric background currents, wide working potential window in aqueous solutions, high resistance to deactivation via fouling, insensitivity to dissolved oxygen and long-term response stability [6–8].

While some metals, such as platinum is known to oxidize hydrogen peroxide and methanol, as well as nickel is conventionally used for carbohydrates electrochemical detection in alkali solution [9–11]. BDD electrode is found completely inactive for those kinds of catalytic reactions. However, it has been reported that the dispersion of metallic particles within an organic polymer or an inert surface resulted in drastic increase of the catalytic activity and sensitivity of the electrode because the dispersed particles behave like microelectrode arrays [10]. The chemically modified electrodes (CMEs) which are capable of lowering the operational potential required to oxidize scarcely electroactive organic compounds has caught a great deal of interest. Reduced electrode fouling has also been reported. CMEs based on the modification of glassy carbon or graphite rods with various metals (e.g.

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* Corresponding author. Tel.: +662 2187615; fax: +662 2541309.
E-mail address: corawon@chula.ac.th (O. Chailapakul).

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copper [12–14], cobalt [15], and nickel [10,11,16–18]) have shown catalytic activities towards polyhydroxy compounds. These electrodes have been successfully applied to detect carbohydrate [10–12,15,16], amino acids [16,18], sugars [13] and aliphatic alcohol compounds [17] using amperometric detection. Unfortunately, glassy carbon electrode has a major drawback of yielding high background current. These metal-modified diamond electrodes appear to be well suited to overcome such problems. BDD film would be the best choice for the deposition of metal electrocatalysts.

The preparation of some metal-modified BDD electrodes for electrochemical analysis by using chemical precipitation and electrochemical deposition method had been reported [9].

Ion implantation into a material can be used to form near surface composites. The method is used to modify the structure of a target-near-surface by bombardment with heavy ions. This method is popular for the preparation of doping semiconductors, such as silicon and gallium arsenide, so it has been of particular interest for the fabrication of ion-implanted diamond. At present, some applications for electrochemical use by metal-implanted conductive BDD electrodes have been reported [19]. Therefore, we are interested in developing a method for the determination of tetracyclines by using the Ni-implanted diamond electrode.

Many antibiotics are widely used in veterinary for preventing and treating diseases as well as for promoting growth in food producing animals. These antibiotics are, for example, aminoglycosides, β-lactams, chloramphenicol, tetracyclines, macrolides, sulphonamides, quinolines, and nitrofurans. Tetracycline is one of the most important antibiotics utilizing in the industry and thus it is of particular interested. Tetracycline is a broad-spectrum antibiotic, such as tetracycline (TC), chlortetracycline (CTC), doxycycline (DC) and oxytetracycline (OTC). These compounds are commonly used in human pathologies as well as in veterinary medicine, animal nutrition and feed additives for cattle growth. It is used to treat many different infections, such as respiratory tract infections, urethritis and severe acne. It also plays a major role in the treatment of multidrug resistant malaria. Adverse effects in these subtances include gastrointestinal disturbances, renal dysfunction, hepatotoxicity, raised intracranial pressure and skin infections, such as rosacea and perioral dermatitis. Tetracyclines are widely used in diary cattle, poultry and shrimp.

The percentage of total worldwide shrimp consumption produced by farming increased from less than 2% in 1980 to more than 26% in 1989 [20]. In recognition of the steadily increasing amount of shrimp produced by aquaculture (rising from∼100 million lbs worldwide in 1979 to 1600 million lbs in 1999), oxytetracycline (OTC) is widely known to be one of the most common antibiotics used in shrimp aquaculture, particularly in countries other than the US. Therefore, shrimps were chosen as the test subject in this study.

Numerous methods have been reported for the determination of tetracyclines in various samples, such as milk [21], shrimp [22], animal feed [23,24], animal tissues [25,26] and pharmaceutical formulations [27,28] based on thin-layer chromatography, capillary electrophoresis [29], and high performance liquid chromatography (HPLC). HPLC is a common method that separates tetracyclines in the reverse-phase mode with a variety of detection methods, such as spectrophotometry [25,26], fluorometry [30,31], mass spectrometry [32,33] and electrochemistry [27,28].

Among these, the electrochemical method is distinctly attractive owing to its simplicity, no need for derivatization, fast analysis, low cost and high sensitivity. There are several reports utilizing polarography, potentiometry [34] and amperometry [27,28,35] for the analysis of tetracycline. In 2005, Charoenraks et al. reported the use of high performance liquid chromatography with pulsed amperometric detection at anodized boron-doped diamond thin film electrode for the detection of tetracyclines.

In this present work, we report the use of Ni-implanted boron-doped diamond thin film electrodes (Ni-DIA) to study the electrochemical oxidation of tetracyclines using cyclic voltammetry. Focus is placed on comparing the results with as-deposited BDD and glassy carbon electrodes. In addition, the performance of the Ni-DIA electrode for the detection of tetracycline was examined by HPLC with amperometric detection for determination of tetracyclines in shrimp.

2. Experimental

2.1. Chemicals and reagents

Tetracycline-HCl (TC), oxytetracycline-HCl (OTC), chlorteteacycline-HCl (CTC), and doxycycline-HCl (DTC) were available from Sigma–Aldrich. Acetonitrile and methanol (Merck) were of HPLC grade. Disodium hydrogenphosphate (BDH), citric acid monohydrate (J.T. Baker), ethylenediaminetetraacetic acid disodium salt dehydrate (Fluka), and phosphoric acid (Merck) were of analytical grade. Distilled water was purified in a Milli-Q system (Millipore, Bedford, MA, USA). Soild-phase extraction (SPE) C-18E cartridges (500 mg, 6 mL) were obtained from Phenomenex (USA).

Phosphate buffer solution of pH 2.5 were prepared from 0.01 M H_3PO_4 and adjusted to 2.5 by adding drop-wise 0.1 M Na_2HPO_4. The mobile phase for the HPLC condition consisted of 20% acetonitrile in 0.01 M phosphate buffer (pH 2.5).

Na_2EDTA–McIlvaine buffer solution (pH 4) was prepared by dissolving 15 g of disodium hydrogen phosphate dihydrate, 13 g of citric acid monohydrate and 3.72 g of EDTA in water and diluting to 1 L.

Stock standard solutions of tetracycline, oxytetracycline, chlortetracycline, and doxycycline were prepared by dissolving 10 mg of each compound in 10 mL of mobile phase to obtain a final concentration of 1000 μg/mL. Working standard solutions were prepared by diluting the stock solution

with the mobile phase. All of the solutions were protected from exposure to light and stored in a refrigerator.

2.2. Sample preparation procedure

Sea and farming shrimp were purchased locally. The shells and tails of shrimp were removed and ground in a conventional meat grinder. The homogenate was degassed overnight and kept in frozen until use. A 2.50 g shrimp samples were placed in 15 mL capped centrifuge tubes, 12.5 mL of Na_2EDTA–McIlvaine buffer (pH 4) was added to each tube portion and blended for 30 s with a homogenizer. The resulting homogenates were shaken for 10 min on a flatbed shaker at high speed. The tube was removed from the shaker and centrifuged for 30 min at 3500 radian per second. The supernatant was loaded into a SPE cartridge, previously activated with 10 mL of methanol and 10 mL of Milli-Q water. After sample loading, the SPE cartridge was washed with 10 mL of Milli-Q water, and finally tetracyclines were eluted by 10 mL of methanol. The solvent was removed under room temperature. The residues were filtered with a 0.45 μm PTFE filter. The solutions were analysed by HPLC.

2.3. Electrode

Highly boron-doped diamond electrode was deposited on Si (100) wafers in microwave plasma-assisted chemical vapor deposition (MPCVD) system (ASTeX Corp., Woburn, MA). A mixture of acetone and methanol in the ratio of 9:1 (v/v) was used as the carbon source. B_2O_3, used as the boron source, was dissolved in the acetone–methanol solution at B/C atomic ratio of 1:100. These films were implanted with 750 keV Ni^{2+} with a dose of 5×10^{14} cm^{-2} (Tandetron 4117-HC, HVEE). Annealing process was performed at 850 °C for 10 min in an H_2 ambient (80 Torr). It was reported that surface morphology and color change after implantation were not observed, a SEM image of the BDD surface after implantation showed the presence of small holes [19]. The presence of metal particles could not be seen, because the particle size is very small as well as the metal position is deeply inside the holes due to the high energy of the bombardment in implantation process.

The nickel-implanted boron-doped diamond electrodes have been prepared in Associate Professor Yasuaki Einaga's laboratory. The Ni-DIA electrodes were rinsed with ultrapure water prior to use.

2.4. Voltammetry

Electrochemical measurements were recorded using an Autolab Potentiostat 30 (Metrohm, Switzerland) with a standard three-electrode configuration. The planar working Ni-DIA or BDD electrode was pressed against a smooth ground joint at the bottom of the cell, isolated by an o-ring (area 0.07 cm^2). Placing the backside of the Si substrate on a brass plate made ohmic contact. For comparison, the as-deposited BDD electrode was used. A platinum wire was used as the auxiliary electrode and Ag/AgCl in KCl (sat'd) was used as the reference. Cyclic voltammetry was used to study the electrochemical reaction. The electrochemical measurements were housed in a Faradaic cage to reduce electronic noise. All experiments were performed at room temperature.

2.5. LC system and conditions

The LC system consisted of a thin layer flow cell (GL Science), an injection port (Rheodyne No. 7125) with a 20 μL injection loop and a pump (Water Model 510 solvent delivery system, Waters Associates Inc., Milford, MA, USA). The column was ODS-3 Inertsil C18, 5 μM 4.6 mm × 250 mm i.d. (GL Science Inc.). The electrochemical detector was applied using a computer controlled potentiostat (Autolab PGSTAT 30, Metrohm, Switzerland). Separations were carried out under isocratic conditions using a mobile phase of 0.01 M phosphate buffer (pH 2.5)–acetonitrile (80:20, v/v). The flow rate was 1 mL/min. The thin layer flow cell consisted of a silicone gasket as a spacer, the Ag/AgCl in 3 M in NaCl as the reference electrode, and a stainless steel tube as the auxiliary electrode and outlet. The experiments were performed in a copper faradaic cage to reduce electrical noise.

3. Results and discussion

3.1. Cyclic voltammetry

Fig. 1(a–d) shows the cyclic voltammograms obtained for 1 mM tetracycline hydrochloride, 1 mM chlortetracycline, 1 mM doxycycline and 1 mM oxytetracycline + 0.1 M phosphate buffer (pH 2) at Ni-DIA electrode, as-deposited diamond electrode and glassy carbon electrode. The corresponding backgrounds are also shown. A well-defined irreversible cyclic voltammograms were obtained at the Ni-DIA electrode and diamond electrode while an ill-defined irreversible cyclic voltammograms was obtained at the glassy carbon electrode for all analytes. The electrochemical data obtained from cyclic voltammograms of these solutions at the mentioned electrodes is shown in Table 1. It was found that the Ni-DIA electrode provided the highest S/B ratios for tetracycline, chlortetracycline, doxycycline and oxytetracycline among the three electrodes studied. We have also carried out the experiments using pure nickel electrode for the comparison with the Ni-DIA electrode. It was found that no any response obtained for the determination of tetracycline antibiotics when using pure nickel electrode.

3.2. Liquid chromatography with amperometric detection

In general, because tetracyclines are analyzed by reversed phase HPLC, the seperation in this experiment was then per-

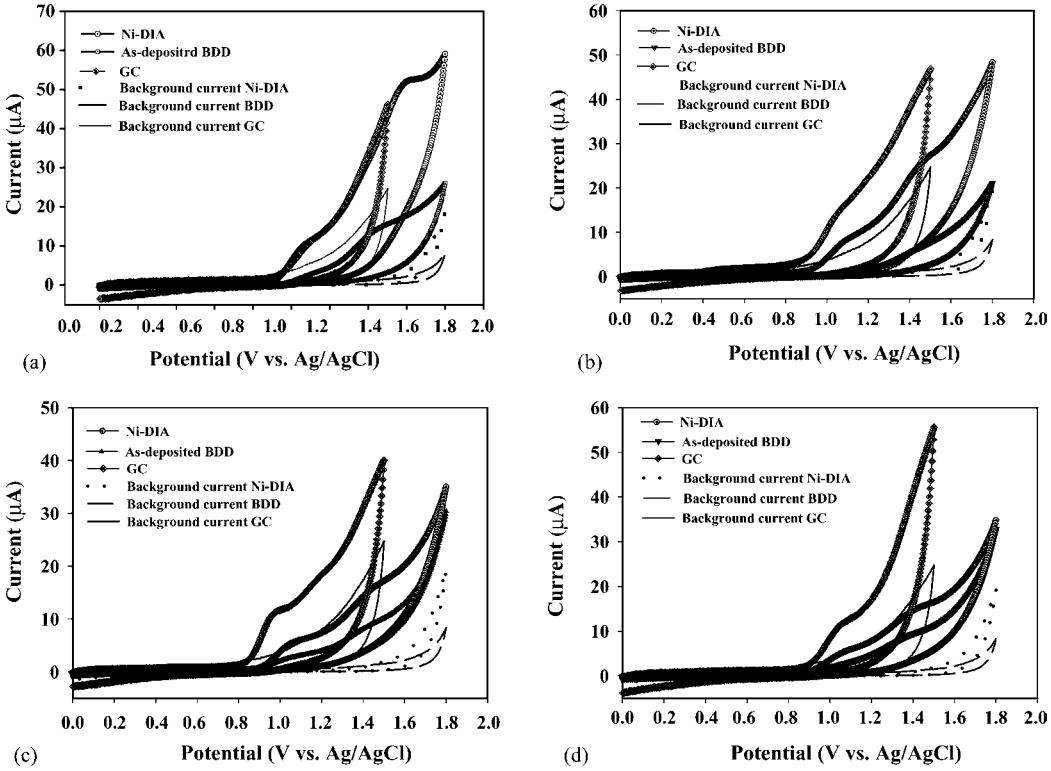

Fig. 1. Cyclic voltammograms for (a) tetracycline, (b) oxytetracycline, (c) chlortetracycline, and (d) doxycycline in 0.1 M phosphate buffer (pH 2) at Ni-DIA, as-deposited diamond and glassy carbon electrodes. The scan rate was 50 mVs^{-1}. Background voltammogram (0.1 M phosphate buffer, pH 2) is also shown in this figure.

formed using a C-18 column. The pH of the mobile phase was selected to be 2.5 so as to reduce the formation of isometric analogues. This pH also gave a well-defined and high signal of cyclic voltammograms of tetracycline oxidation. In this experiment, the phosphate buffer was chosen because it provided low background currents. No reaction between buffer and tetracyclines was observed over the potential range of interest. Therefore, phosphate buffer (0.01 M, pH 2.5) was used to separate tetracyclines, and significantly prolonged the retention time in the presence of 20% acetonitrile. The chromatograms of a standard solution of tetracyclines are presented in Fig. 2. The orders of elution were oxytetracycline,

Table 1
The electrochemical data of 1 mM tetracycline, 1 mM chlortetracycline, 1 mM doxycycline and 1 mM oxytetracycline at Ni-DIA electrode, as-deposited diamond electrode and glassy carbon electrode

Analytes	Electrode	E_p^{oxa}(V)	I_p^{oxb}(μA)	S/B^c
Tetracycline	Ni-DIA	1.501	20.90	12.06
	BDD	1.501	17.00	11.56
	GC	1.178	7.10	1.42
Chlortetracycline	Ni-DIA	1.501	17.30	9.61
	BDD	1.438	9.00	6.12
	GC	0.975	7.80	1.56
Doxycycline	Ni-DIA	1.501	16.60	9.22
	BDD	1.477	10.60	7.21
	GC	1.059	7.00	1.40
Oxytetracycline	Ni-DIA	1.511	27.90	15.50
	BDD	1.506	7.80	5.31
	GC	1.064	9.90	1.98

[a] Oxidation peak potential.
[b] Oxidation peak current.
[c] calculated from I_p^{ox}/background current.

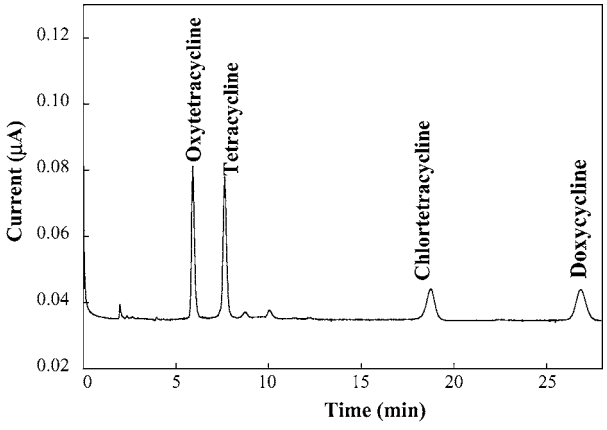

Fig. 2. Chromatogram of 1 ppm a standard mixture seperated on ODS Inertsil C18 column (5 μM 4.6 × 250 mm i.d.) using a mobile phase of phosphate buffer (0.01 M, pH 2.5)–acetonitrile (80:20). The injection volume was 20 μL, and the flow rate was 1 mL/min.

Table 2
Calibration characteristics of oxyteracycline, tetracycline, chlortetracycline and doxycycline for the proposed method at the Ni-DIA electrode

Analytes	Linear dynamic range ($\mu g\,mL^{-1}$)	Slope (peak areas units/mg/kg)	Intercept (μA)	R^2	Limit of detection ($\mu g\,mL^{-1}$)
Oxytetracycline	0.05–100	0.0473	0.0562	0.9975	0.01
Tetracycline	0.05–100	0.0395	0.0453	0.9977	0.01
Chlortetracycline	0.1–100	0.0110	0.0045	0.9990	0.05
Doxycycline	0.1–100	0.0111	0.0066	0.9996	0.05

Table 3
Recoveries of tetracyclines in shrimp farming samples using the HPLC with amperometry method at the Ni-implanted electrode ($n = 3$)

Analyte	Mean of %recovery ($x \pm$ S.D.)			
	Spiking level of 0.5 mg kg^{-1}	Spiking level of 1 mg kg^{-1}	Spiking level of 5 mg kg^{-1}	Spiking level of 10 mg kg^{-1}
Oxytetracycline	84.8 ± 3.0	96.8 ± 2.7	102.5 ± 3.4	99.6 ± 1.8
Tetracycline	93.3 ± 5.5	85.9 ± 7.7	96.6 ± 2.4	97.0 ± 5.5
Chlortetracycline	91.48 ± 5.3	94.8 ± 5.9	91.6 ± 5.2	97.9 ± 3.8
Doxycycline	89.2 ± 6.7	88.4 ± 3.0	97.7 ± 5.4	103.7 ± 7.4

Table 4
Recoveries of tetracyclines in shrimp sea samples using the HPLC with amperometry method at the Ni-DIA electrode ($n = 3$)

Analyte	Mean of %recovery ($x \pm$ SD)			
	Spiking level of 0.5 mg/kg	Spiking level of 1 mg/kg	Spiking level of 5 mg/kg	Spiking level of 10 mg/kg
Oxytetracycline	94.9 ± 1.6	83.3 ± 4.3	86.8 ± 5.0	96.5 ± 2.4
Tetracycline	92.0 ± 1.1	88.4 ± 4.4	89.2 ± 1.2	96.9 ± 4.6
Chlortetracycline	91.8 ± 8.6	91.9 ± 3.0	86.0 ± 8.0	93.3 ± 5.1
Doxycycline	102.0 ± 9.0	96.2 ± 1.7	90.6 ± 0.1	99.4 ± 2.4

tetracycline, chlortetracycline and doxycycline, respectively. To complete the separation of tetracyclines, approximately 27 min are required.

3.3. Optimum potential for HPLC

Fig. 3 depicts the optimum potential *i–E* curve obtained at the Ni-DIA electrode for a 20 μl injection of 100 μM of tetracyclines mixture standard solution. 0.01 M of phosphate buffer (pH 2.5) was used as the carrier solution. Each datum represents an average of two injections. The magnitude of the background current at each potential is also shown for comparison. The optimum potential of tetracyclines mixture standard solution at the Ni-DIA electrode exhibited a well-defined sigmodal shape with a half peak potential at about 1.55 V versus Ag/AgCl. Therefore, this potential was fixed for the amperometric potential detection in HPLC system analysis experiments.

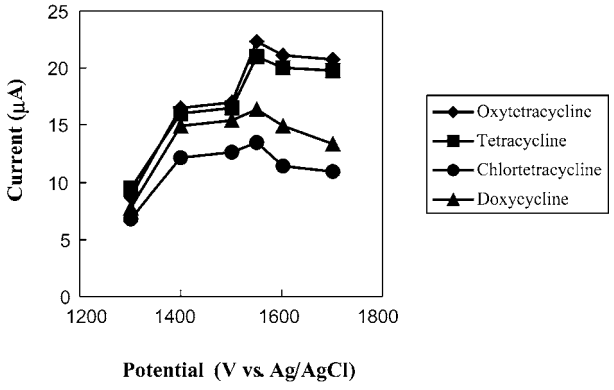

Fig. 3. Optimum potential of 10 ppm of tetracyclines in 0.01 M phosphate buffer (pH 2.5). 0.1 M phosphate buffer (pH 2.5) was used as a carrier solution, flow rate 1 ml min^{-1}.

3.4. Linearity and detection limit

The tetracycline mixture standard solutions covering the concentration range of 0.01–100 $\mu g\,mL^{-1}$ were analyzed and their peak areas were plotted versus concentration. The calibration characteristics of oxytetracycline, tetracycline, chlortetracycline and doxycycline at the Ni-DIA electrode are given in Table 2.

3.5. Recoveries

The average recoveries of tetracyclines from shrimp farming and shrimp sea sample at four different spiking levels (0.5, 1, 5 and 10 μg/mL each compound) are summarized in Tables 3 and 4. It can be observed from the chromatogram

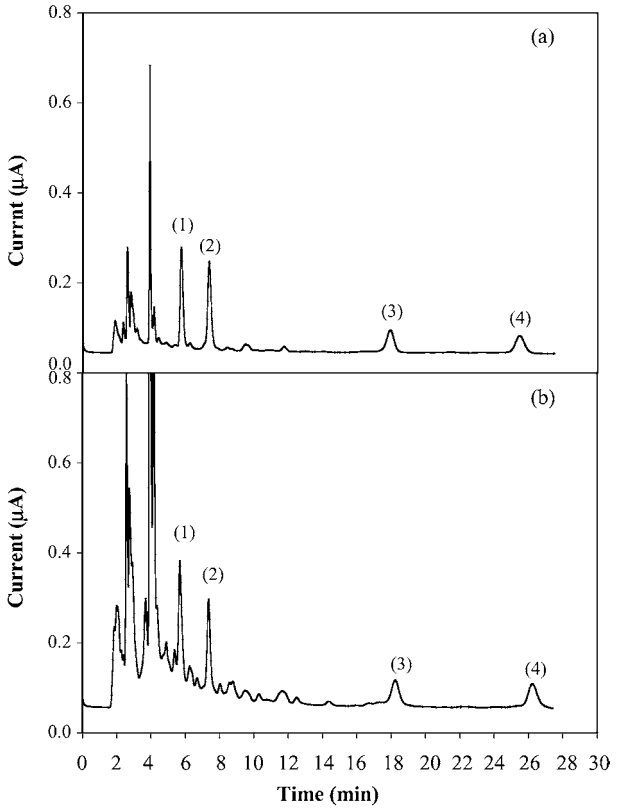

Fig. 4. HPLC chromatograms of (a) shrimp farming sample and (b) shrimp sea sample spiking with 5 μg/mL each of (1) oxytetracycline, (2) tetracycline, (3) chlortetracycline, and (4) doxycycline at the Ni-implanted diamond electrode. The other conditions are the same as in Fig. 2.

(Fig. 4) that the peaks due to the other components did not interfere with those of tetracyclines.

4. Conclusions

This is the first use of Ni-DIA electrodes for the electroanalysis of tetracyclines. It was found that Ni-DIA electrodes exhibited excellent performance for the oxidative detection of tetracycline. Well-defined voltammograms were obtained at the Ni-DIA electrode, which exhibited high sensitivity and demonstrated significant advantages over the BDD and glassy carbon electrode. The outstanding capabilities of the Ni-DIA electrode were demonstrated by coupling with HPLC. HPLC with amperometry at Ni-DIA electrode has been successfully applied to determine four types of tetracyclines (oxytetracycline, tetracycline, chlortetracycline and doxycycline) in shrimp samples. Experimental detection limit of 0.01–0.05 μg/mL were obtained for four tetracyclines studied. A linear dynamic range from 0.05 to 100 μg/mL was achieved. Application of the proposed method for the determination of tetracycline in shrimp sample shows that this method is precise, accurate and very sensitive.

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