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Electrosorption-enhanced solid-phase microextraction of trace anions using a platinum plate coated with single-walled carbon nanotubes

Quanlong Li*, Yujing Ding, Dongxing Yuan

State Key Laboratory of Marine Environmental Science, Environmental Science Research Center, Xiamen University, Xiamen 361005, China

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ABSTRACT

A platinum plate coated with single-walled carbon nanotubes (SWCNTs@Pt) was prepared by means of electrophoretic deposition. Using the SWCNTs@Pt plate, an electrosorption-enhanced solid-phase microextraction (EE-SPME) technique was proposed for the extraction of trace anions in water, described as follows: a positive potential was applied to the SWCNTs@Pt plate to extract F^-, Cl^-, Br^-, $NO_3{}^-$ and $SO_4{}^{2-}$ from water using electrosorption, and then a negative potential was applied to the plate placed in ultra-pure water for the desorption of the absorbed anions, and finally the desorbed anions were analyzed using ion chromatography (IC). The EE-SPME parameters, including extraction potential and time as well as desorption potential and time, were investigated. An analytical method based on the above procedures, i.e., EE-SPME-IC, was established and used for the analysis of trace anions in water. The results showed that the application of potential on the SWCNTs@Pt plate significantly enhanced the ion extraction efficiency, and an enrichment factor of 15–38 was achieved. The SWCNTs@Pt plate could be used more than 50 times without significant decay. The linear range, the limit of detection ($S/N = 3$), the limit of quantification ($S/N = 10$) and repeatability ($n = 7$) of our EE-SPME-IC method were 1.0–150.0 μg/L, 0.06–0.26 μg/L, 0.19–0.85 μg/L and 2.1–8.0%, respectively. The proposed method was successfully applied for the analysis of trace anions in deionized water, and acceptable recoveries between 65.3 and 121.1% were obtained for the spiked deionized water samples.

1. Introduction

As a simple and solvent-free sample preparation technique, solid phase microextraction (SPME) has been successfully applied in the analysis of environmental samples, food and pharmaceuticals [1–3]. However, direct application to ionic species has been limited since the neutral charge of conventional SPME coatings leads to a low coating/sample partition coefficient and poor analyte recoveries.

In the past decade, electrochemically controlled SPME (EC-SPME), in which SPME is combined with electrochemistry, has been extensively proposed by various research groups to extract anionic [4–9], cationic [5,8–11] and neutral analytes [12,13] from water samples. EC-SPME uses conducting polymers such as polypyrrole and its derivatives as the SPME stationary phase. If an anion of high mobility, e.g., Cl^-, $ClO_4{}^-$, $NO_3{}^-$, is incorporated into a conducting polymer, then the polymer becomes an anion exchanger. The incorporation of anions of low mobility into the polymer, typically polymeric counter anions themselves, causes it to function as a cation exchanger [8,14]. The charge on a polymer can be electrochemically controlled using either an oxidation or reduction procedure. Extraction and desorption in EC-SPME can be controlled simply by controlling the potential applied to the polymer. Although the application of EC-SPME for the direct extraction of ions from water is achieved, in some reports the SPME device is short lived and successive extraction is impossible [7,13] due to the oxidation and reduction of the stationary phase during every measurement. In addition, the extraction of analytes in EC-SPME is based on ion exchange, but the ion exchanging capacity of the polymer is limited due to its small volume. As a result, the extraction efficiency of EC-SPME is low [7,8,10,11,13] and extraction is needed several times for one sample [8].

In recent years, electrosorption-enhanced SPME (EE-SPME), which is the combination of electrosorption and SPME, is proposed to directly extract charged and uncharged species from water [15–17]. Instead of using a conductive polymer as the stationary phase in EC-SPME, carbonaceous materials, such as pencil lead [15], activated carbon fiber [16] and carbon nanotubes [17], are used, due to their conductivity, as coatings to prepare the SPME fiber. When an electric field is applied, the charged ions move towards the fibers with an opposite charge, and an electrical double layer is formed on the carbonaceous fiber surface. Charged species are therefore concentrated and kept on the double layer. When the electric field is removed or reversed, the ions in the fiber

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* Corresponding author. Fax: +86 592 218 3137.
E-mail address: liql@xmu.edu.cn (Q. Li).

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are released back to the bulk solution. No reduction or oxidation of coating occurs during EE-SPME. Negative potential on a home-made SPME fiber made from pencil lead is used to extract ionized methamphetamine before analysis using GC–MS [15]. A positive potential on an activated carbon fiber is also used to trap aniline in aqueous solution [16]. An SPME fiber coated with multi-walled carbon nanotubes/Nafion composite is prepared [17] and used for the extraction of cationic (protonated amines) or anionic compounds (deprotonated carboxylic acids) in aqueous solutions by applying suitable negative or positive potentials. These studies show that EE-SPME can enhance the extraction efficiency and selectivity as well as reduce the adsorption time. However, SPME devices suitable for EE-SPME are very limited, and the application of EE-SPME in the extraction of inorganic ions has not yet been reported.

The application of a new carbon-based nano-material, carbon nanotubes (CNTs), as an SPME coating is reported [17–21]. An electrophoretic deposition (EPD) method for the preparation of SPME platinum (Pt) fiber coated with single-walled CNTs (SWCNTs) is also proposed [18]. In the above report, SWCNTs were attached to Pt wire without the use of any adhesive, and the obtained SPME fiber was found to be highly conductive. The conductivity of SWCNTs coating was estimated to be 4.0 S/m [18]. With high specific surface area and conductivity, this type of fiber or a similar device may find potential use in EE-SPME.

In our study, a Pt plate coated with SWCNTs (SWCNTs@Pt) was prepared with EPD and EE-SPME techniques and the SWCNTs@Pt plate was proposed in order to extract trace anions, i.e., F^-, Cl^-, Br^-, NO_3^- and SO_4^{2-}, from water. The EE-SPME conditions, the extraction efficiency and the life time of the SWCNTs@Pt plate were investigated. The analytical method based on EE-SPME-ion chromatography (IC) was established and applied for the analysis of the trace anions in the deionized water samples.

2. Experimental

2.1. Chemicals and standard solutions

Sodium fluoride, sodium chloride, sodium bromide, potassium nitrate, sodium sulfate, sodium carbonate, and sodium bicarbonate of analytical grade were purchased from Sinopharm Chemical Reagent Co. (Shanghai, China). Ultra pure water (18.2 MΩ cm) was obtained from a Milli-Q ultra-pure water system (Millipore, Billerica, USA) and used throughout the experiments. Commercially available SWCNTs with a specific surface area of 380 m^2/g were purchased from the Chengdu Organic Chemistry Co. (Chengdu, China). Single-anion stock standards of F^-, Cl^-, Br^-, NO_3^- and SO_4^{2-} each at 1000 mg/L were prepared by adding a certain amount of each to a 100 mL volumetric flask and diluting this with ultra-pure water. The multi-anion standard (each anion at 10 mg/L) was prepared from the single-anion stock standards.

2.2. Ion chromatography analysis

A Dionex ICS-2500 System (Sunnyvale, CA, USA) with a GP50 gradient pump and an ED50 electrochemical detector was used for all analyses. Separation was performed on a Dionex IonPac AS14A-7 μm (4 mm × 250 mm) column with an IonPac AG14A (4 mm × 50 mm) guard column. Post-column eluent suppression was achieved using a Dionex ASRS300 Anion Self-Regenerating Suppressor with a suppressor current of 200 mA. The columns and suppressor were housed in an LC25 chromatography oven with a six-port Rheodyne valve. The eluent was 8.0 mM Na_2CO_3/1.0 mM $NaHCO_3$ at a flow rate of 1.0 mL/min. Instrument control and data collection were accomplished using a personal computer and Chromeleon software (Dionex).

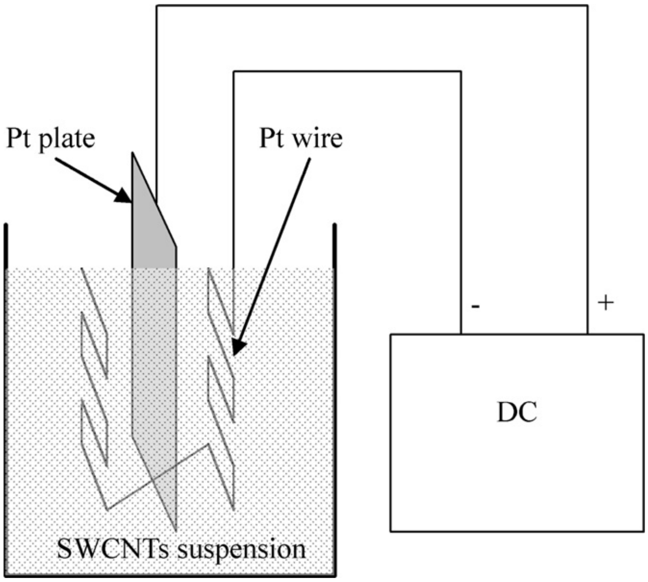

Fig. 1. Schematic of EPD for the preparation of a SWCNTs@Pt plate.

2.3. Preparation and thermal treatment of SWCNTs@Pt plates

The method used to prepare SWCNTs@Pt plates with EPD was similar to that described in our previous work [18], in which a Pt fiber was coated with SWCNTs. In brief, the SWCNTs were oxidized with a mixture of concentrated nitric and sulfuric acid and ultrasonically dispersed in dimethylformamide to form a stable suspension containing 2 mg/mL of SWCNTs. One Pt plate, 3.5 cm in length, 0.8 cm in width and 0.01 cm in thickness, was washed with acetone before use. As shown in Fig. 1, the plate and a Pt wire were immersed into the suspension, and then a DC voltage of 40 V was applied between the Pt plate and the Pt wire for 10 s, and a SWCNTs deposit was formed on both sides of the Pt plate, which served as the anode. The plate with its deposit was removed from the suspension and dried at 120 °C to remove the solvent, and thus a SWCNTs@Pt plate was obtained. Plates with different coating thicknesses could be produced by repeating the EPD procedure various times. In this study, a SWCNTs@Pt plate 1.6 cm × 0.8 cm and about 50 μm in thickness was prepared. The amount of SWCNTs coated on the plate was about 10 mg.

The SWCNTs@Pt plate was placed in a quartz tube and fixed with quartz wool. The quartz tube was then placed in a home-made tube furnace, and H_2 was introduced at a flow rate of 40 mL/min into the tube. The SWCNTs@Pt plate was treated at 500 °C for 30 min. This thermal annealing treatment can remove the polar groups on the SWCNTs surface [22], which were introduced during the oxidation procedure with the concentrated nitric and sulfuric acid mixture. As a result, the possibility of ion-exchange due to the polar groups was greatly reduced. The morphology of the surface of SWCNTs@Pt plate was observed with Scanning Electron Microscope (SEM, Hitachi S-4800).

2.4. Solid-phase microextraction and desorption procedure

The schematic for the EE-SPME of anions in water sample using an SWCNTs@Pt plate is shown in Fig. 2(a). A 25 mL water sample and a stirring bar were placed in a 25 mL beaker. The plate and a Pt wire were immersed in the sample and a constant positive potential from the electrochemistry analyzer (CHI800b, Shanghai Chenhua Apparatus Co. Ltd., Shanghai, China) was applied. The plate was used as the anode while the Pt wire was the counter electrode.

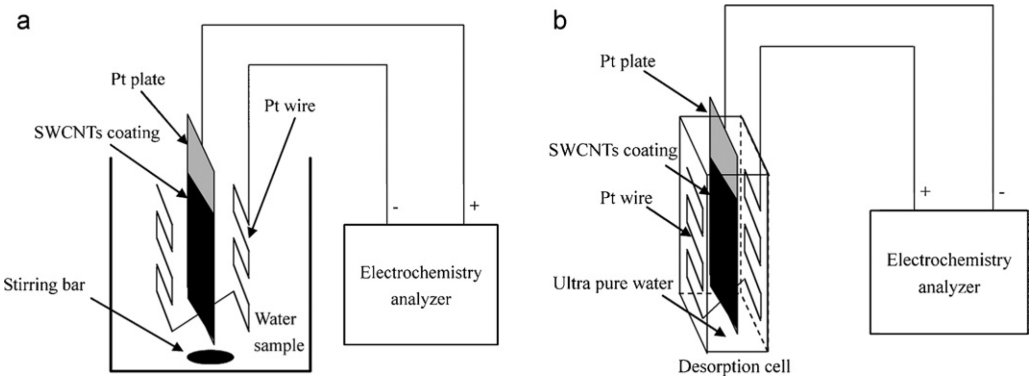

Fig. 2. Schematic of extraction (a) and desorption (b) of anions in EE-SPME with a SWCNTs@Pt plate.

After a period of time for extraction, the plate was removed from the solution for desorption.

Desorption was performed in a specially designed cell as shown in Fig. 2(b). This was made from polymethyl methacrylate with Pt wires attached to the longer inner walls. The inner volume of the cell was approximately 300 μL. The SWCNTs@Pt plate was placed into the cell and the cell filled with 200 μL of ultra pure water; then a constant reversed potential was applied where the plate was used as the cathode, finally the plate was removed at the designated time. In order to avoid the carryover of the SWCNTs@Pt plate, the desorption step described above was carried out one more time. 50 μL of the desorption solution obtained in the first desorption was used for IC analysis.

2.5. *Performance and analysis of deionized water samples*

The EE-SPME parameters were studied, including extraction potential and time, as well as desorption potential and time. Under the chosen parameters, the performance of our EE-SPME-IC system was investigated, and the proposed method was used to determine trace anions in three deionized water samples. One sample (labeled RO) was from the reverse osmosis water system in our laboratory, and the other two samples (labeled WT and WH) were both bottled water from a local supermarket. All samples were analyzed without any further treatment.

3. Results and discussion

3.1. *Extraction of anions*

The extraction of ions using EE-SPME is based on electrosorption, which is defined as a current or polarization potential-induced adsorption phenomenon on the surface of the charged electrodes [23]. When an external electrostatic field is imposed onto the surface of the electrodes immersed in an aqueous electrolyte solution, charged ions move towards the oppositely charged electrodes, resulting in the formation of strong electrical double layers on the surface of the electrodes [24].

The amount of SWCNTs coated on the Pt plate was about 10 mg, and thus the surface area of this electrode was approximately 3.8 m^2. Both the SWCNTs and the Pt plate had high conductivity and, therefore, the formation of a strong electrical double layer on the plate was expected during EE-SPME. Fig. 3 illustrates the typical current–time curve when a constant positive potential was applied on the plate during an extraction step. The current changed rapidly at the beginning, but then slowly. This phenomenon showed clearly the formation of a strong electrical double layer, in which the anions were enriched and kept. When negative potential was applied on the plate during the desorption step, these anions was released into the ultra-pure water.

3.2. *Effects of EE-SPME conditions*

The effects of EE-SPME conditions including extraction potential and time, as well as desorption potential and time, were investigated. The sample used in this section was the standard solution spiked with F^-, Cl^-, Br^-, $NO_3{}^-$ and $SO_4{}^{2-}$ each at 100 μg/L.

3.2.1. *Desorption conditions*

The residual anions on the SWCNTs@Pt plate could cause carryover, and a significant amount of residual ions were found when the plate was desorbed in the cell for 8 min with a desorption potential of −1.5 V. Therefore, another desorption in ultra-pure water for 4 min with a potential of −1.5 V was carried out. The results showed that after being desorbed twice in ultra-pure water as described above, the residual ions on the plate could be neglected, and therefore the plate could be reused for the next measurement. These desorption conditions were adopted for all the following experiments.

3.2.2. *Extraction potential*

In EE-SPME, the extraction potential is a key parameter that affects the amount of ions extracted. Five potentials, 0.5, 1.0, 1.5, 1.8 and 2.0 V, were investigated, and the results are shown in Fig. 4. The amounts of ions extracted increased with the potential applied. A higher potential resulted in a stronger electrical double layer and thus more ions were constrained on the SWCNTs@Pt plate. When the applied potential was higher than 2.0 V, bubbles began to

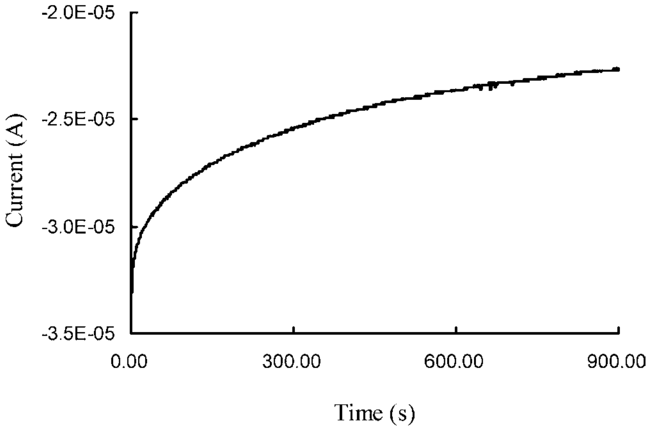

Fig. 3. Typical current–time curve for extraction.

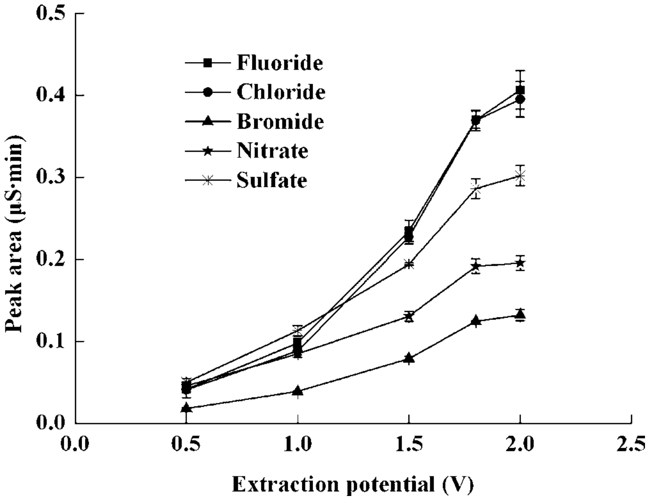

Fig. 4. Effect of extraction potential ($n=3$). Extraction time, 15 min; stirring rate, 200 rpm.

appear on the plate surface. This indicated the occurrence of water electrolysis. As a result, 1.8 V was chosen as the extraction potential.

3.2.3. Extraction time

The extraction time is another parameter that has a significant effect on the amount of ions extracted. Seven extraction times, 2.5, 5.0, 8.0, 10.0, 15.0, 20.0 and 25.0 min, were tested, and the results are shown in Fig. 5. The amount of anions extracted increased with the extraction time and the adsorption equilibrium was not reached after 25 min. Fig. 6 shows the SEM image of the SWCNTs@Pt plate. SWCNTs could be resolved clearly. Pores with diameters of nanometer, formed through overlaying of SWCNTs, present on the surface. The long adsorption equilibrium time is most likely due to these pores because the ions move more slowly in a pore than in the solution. Although the sensitivity rises with time, a long extraction time would reduce the sample throughput. Considering both efficiency and sensitivity, 15 min was chosen as the extraction time.

3.2.4. Stirring rate

Three stirring rates, 200, 500 and 1000 rpm, were tested, and the results are shown in Fig. 7. The amount of anions extracted decreased slightly with the stirring rate. Therefore 200 rpm was used in this study.

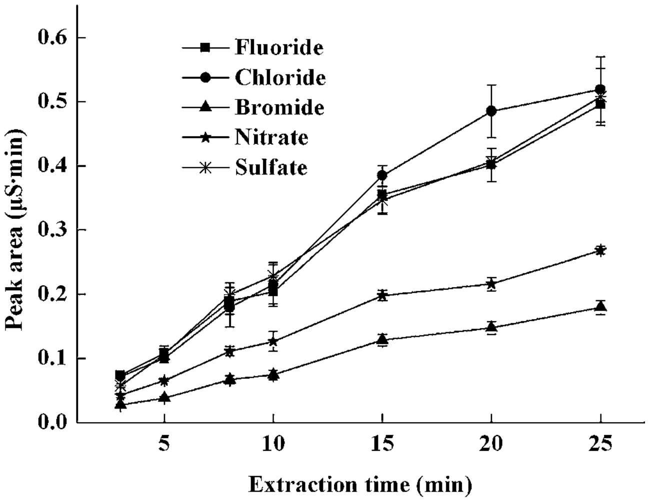

Fig. 5. Effect of extraction time ($n=3$). Extraction potential, 1.8 V; stirring rate, 200 rpm.

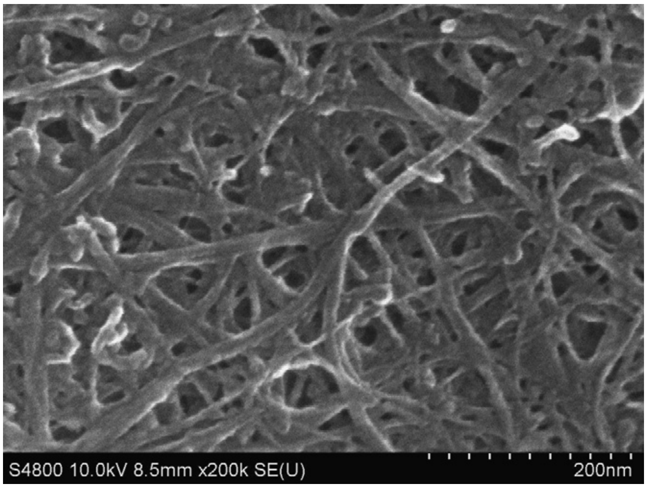

Fig. 6. SEM image of the surface of SWCNTs@Pt plate.

3.3. Life time of the SWCNTs@Pt plate

The life time of the SWCNTs@Pt plate was evaluated by extracting anions from a spiked sample of 100 μg/L after it had been used 0, 30, 50, 70, 90 and 110 times. The results in Fig. 8 indicate that after being used 50 times, the extraction efficiency of the SWCNTs declined, indicating a life time of 50 operations. This decline might have been due to worsening contact between the SWCNTs coatings and the Pt plate as a result of the repeated operation of adsorption and desorption under a relatively high potential. Compared with the SPME device used in EC-SPME [7,13], the SWCNTs@Pt plate had a much longer life time. This was because EE-SPME was based on electrosorption, whereas the SWCNT coatings were not involved in chemical reaction.

3.4. Performance of the method

In order to evaluate the extraction efficiency of EE-SPME, 50 μL of the standard solution of 100 μg/L was analyzed using IC but without EE-SPME. The chromatogram obtained is shown in Fig. 9 and compared to that with EE-SPME. The peak height of the anions increased greatly with EE-SPME, and the enrichment factors for F^-, Cl^-, Br^-, $NO_3{}^-$ and $SO_4{}^{2-}$ were 15, 25, 21, 28 and 38.

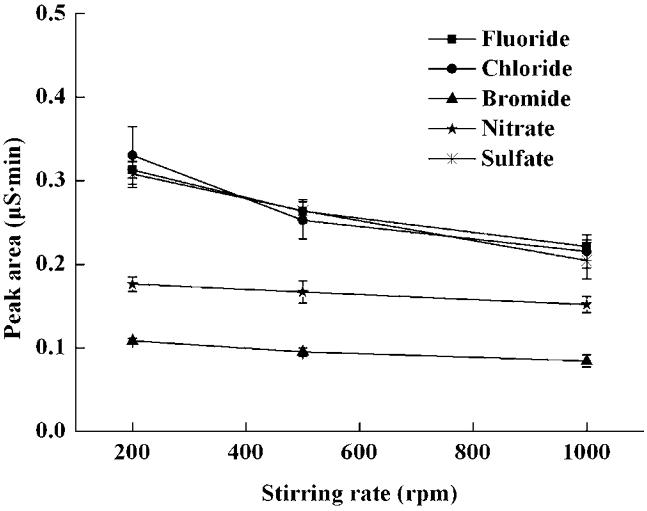

Fig. 7. Effect of stirring rate ($n=3$). Extraction time, 15 min; extraction potential, 1.8 V.

Table 1
Characteristic data of the EE-SPME-IC method for the determination of anions.[a]

Anion	Linear range (μg/L)	R	LOD (μg/L)	LOQ (μg/L)	RSD (%) (n = 7)
F^-	1.0–150.0	0.998	0.06	0.19	2.1
Cl^-	1.0–150.0	0.997	0.06	0.21	4.8
Br^-	1.0–150.0	0.998	0.26	0.85	8.0
NO_3^-	1.0–150.0	0.998	0.22	0.72	5.6
SO_4^{2-}	1.0–150.0	0.994	0.23	0.78	7.0

LOD and LOQ were calculated based on 3 and 10 times the average background noise and divided by the detection sensitivity.

[a] The analytical curves were constructed with 8 concentration levels of each anion. The concentration of the standard solution for the test of repeatability was 100 μg/L for each anion.

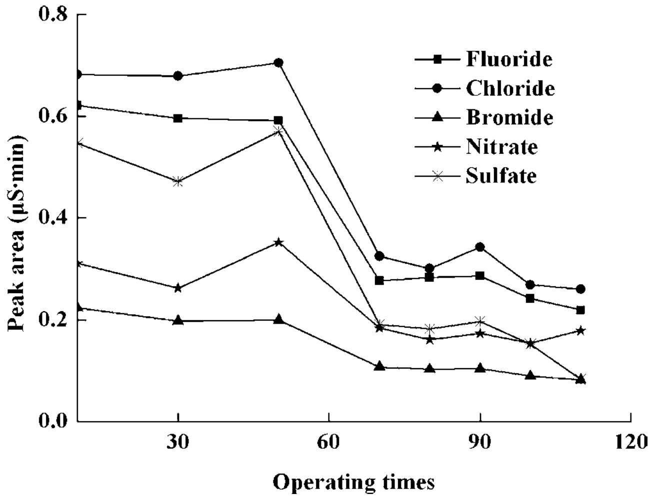

Fig. 8. Durability of the SWCNTs@Pt plate.

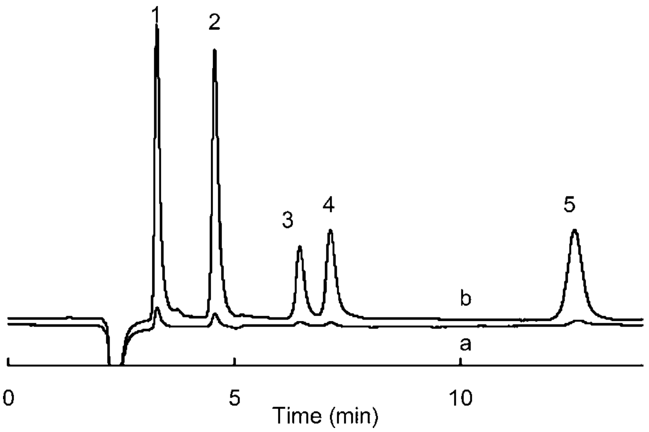

Fig. 9. Chromatograms of standard solution without (a) and with (b) EE-SPME. (1) F^-, (2) Cl^-, (3) Br^-, (4) NO_3^-, (5) SO_4^{2-}.

The calibration curves were constructed with 8 concentration levels of each anion, i.e., 1.0, 5.0, 10.0, 25.0, 50.0, 100.0, 125.0, and 150.0 μg/L. The linear range, correlation coefficient, limit of detection (LOD), limit of quantification (LOQ) and repeatability of the EE-SPME-IC method for the analysis of anions in a spiked water sample under the chosen conditions are listed in Table 1. The analytical curves were constructed with eight concentration levels of each anion and exhibited good linearity ($r > 0.994$). The concentration of the standard solution for the repeatability test was 100 μg/L for each anion. The linear range, the LOD ($S/N = 3$), the LOQ ($S/N = 10$) and the repeatability (RSD%, $n = 7$) of the EE-SPME-IC method were 1.0–150.0 μg/L, 0.06–0.26 μg/L, 0.19–0.85 μg/L and 2.1–8.0%.

Nowadays the pre-concentration of ions on a concentrator column followed by on-line IC analysis (CC-IC) is well established and routinely used for analysis of trace ions in high-purity waters [25,26]. More recently an alternative IC method based on high-volume direct-injection technique (HVDI-IC) has been successfully established [25,27]. Table 2 shows the characteristics of EE-SPME-IC and those of CC-IC [26] and HVDI-IC [27] reported in the literature. The LODs of EE-SPME-IC are similar to those of HVDI-CC, but HVDI is simpler in analytical system and with shorter analysis time than EE-SPME-IC because it does not need additional hardware and the concentration step. With similar enrichment time, CC-IC has much lower LODs than EE-SPME-IC. At present, EE-SPME-IC cannot reach the analytical performance of CC-IC and LVDI-IC, further work is needed. For example, much lower LODs could be achieved if more water sample (25 mL in this study), longer extraction time (15 min in this study), electrode of bigger size (1.6 cm × 0.8 cm in this study), more desorption solution injected into IC (only 50 μL was injected in to IC in this study) and SWCNTs of higher surface area (380 m^2/g in this study) were applied. When a high volume of desorption solution such as 1 mL (200 μL in this study) was obtained in EE-SPME, the combination of EE-SPME and HVDI-IC would result in very low LODs.

SWCNTs coatings are hydrophobic and porous, thus have strong affinity for organic compounds. The adsorption of these compounds would definitely foul the electrode because they could not be removed from the electrode in the desorption step. As a result, the method described in this paper is suitable for the sample with clean matrix and not suitable for that with complex matrix.

3.5. *Analysis of deionized water samples*

Three deionized water samples, RO, WT and HT, were analyzed with the EE-SPME-IC method which we established, and the chromatogram obtained with the RO sample is shown in Fig. 10. The chromatogram of RO, obtained without EE-SPME, is also shown for comparison. When direct IC was applied, very small peaks were found for F^- and NO_3^-, and no peaks for Cl^- and SO_4^{2-}. On the other hand, large peaks were recorded for the four ions with EE-SPME.

Table 2
Characteristics of EE-SPME-IC, concentrator column-IC and high-volume direct-injection-IC for the analysis of trace anions in high-purity water.

Analytical methods	Injection volume (μL)	Enrichment sample volume (mL)	Enrichment time (min)	LODs (μg/L)	References
EE-SPME-IC	50	25	15	0.06–0.26	This study
Concentrator column-IC	–	20	20	0.01–0.03	[26]
High-volume direct-injection-IC	1000	–	–	0.04–0.27	[27]

LODs are those of F^-, Cl^-, Br^-, NO_3^-, and SO_4^{2-}.

Table 3
Concentrations of anions in the deionized water samples and the recoveries of the spiked samples.

Anion	RO		WT		WH	
	Concentration (μg/L, $n = 3$)	Recovery (%, $n = 3$)	Concentration (μg/L, $n = 3$)	Recovery (%, $n = 3$)	Concentration (μg/L, $n = 3$)	Recovery (%, $n = 3$)
F^-	9.3 ± 1.3	90.1 ± 1.2	ND	70.1 ± 7.3	4.8 ± 0.6	65.3 ± 3.4
Cl^-	9.5 ± 0.9	103.4 ± 4.4	6.1 ± 1.1	80.2 ± 5.7	29.5 ± 0.5	83.0 ± 11.8
Br^-	ND	84.2 ± 9.4	ND	79.7 ± 0.8	ND	70.7 ± 2.7
NO_3^-	45.7 ± 4.7	105.8 ± 3.9	123.6 ± 11.2	104.4 ± 2.4	130.9 ± 2.5	121.1 ± 3.4
SO_4^{2-}	4.5 ± 0.2	77.0 ± 7.0	4.4 ± 0.2	78.0 ± 4.7	53.2 ± 2.2	67.3 ± 7.2

RO, WT and WH were spiked at 20, 20 and 50 μg/L for each anion, respectively; ND stands for not detected.

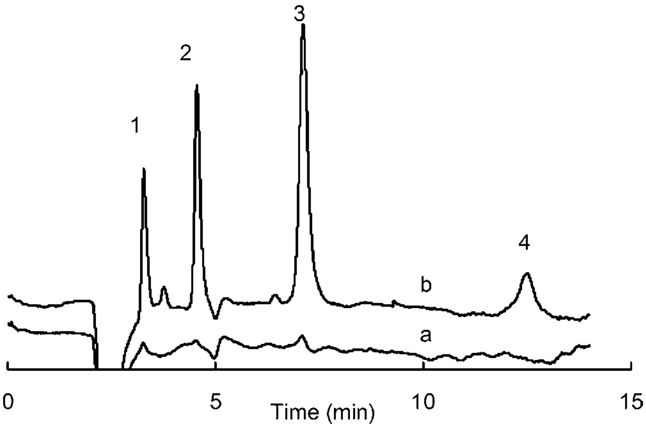

Fig. 10. Chromatograms of deionized water (RO) without (a) and with (b) EE-SPME. (1) F^-, (2) Cl^-, (3) NO_3^-, (4) SO_4^{2-}.

The concentrations of the anions in the deionized water samples, determined using EE-SPME-IC, are listed in Table 3. RO, WT and WH were spiked, respectively, at 20, 20 and 50 μg/L. The recovery of each anion is also shown in Table 3. The repeatability ($n = 3$) was in the range 0.6–11.2%, and acceptable recoveries were between 65.3 and 121.1% in the spiked samples. The results revealed that the EE-SPME-IC which we established could be used to analyze trace anions in water samples with a clean matrix, such as deionized water.

4. Conclusions

In this study, a Pt plate coated with SWCNTs was prepared with EPD and used as an EE-SPME device for the analysis of trace anions in water samples. The proposed device showed the features of high extraction efficiency and long life time. Our EE-SPME-IC method based on this device could be applied to determine trace anions in water samples with a clean matrix, such as deionized water.

Acknowledgements

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