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Electrochemical determination of hydrogen sulfide at carbon nanotube modified electrodes

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Abstract

Carbon nanotube (CNT) modified glassy carbon electrodes exhibiting a strong and stable electrocatalytic response towards sulfide are described. A substantial (400 mV) decrease in the overvoltage of the sulfide oxidation reaction (compared to ordinary carbon electrodes) is observed using the CNT-modified electrode, with oxidation starting at ca. -0.3 V (versus Ag/AgCl; pH 7.4). The CNT-coated electrodes thus allow highly sensitive, low potential (+0.1 V), stable amperometric sensing. A wide linear dynamic range (1.25–112.5 μ M) was achieved with a detection limit of 0.3 μ M (9 ppb). The enhance sensitivity is coupled to an improved stability. Such ability of carbon nanotubes to promote the sulfide electron-transfer reaction suggests great promise for miniaturized sensors for hydrogen sulfide. © 2004 Elsevier B.V. All rights reserved.

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1. Introduction

There is widespread awareness of the toxicity of sulfide in its liberated hydrogen sulfide (H₂S) form. Even at a low concentration, H₂S can lead to personal distress whilst at a higher concentration it can result in loss of consciousness, permanent brain damage or even death due to the neurotoxic effect of the gas [1]. Hydrogen sulfide is a by-product of many industrial processes and it is produced in nature primarily by decomposition of organic materials by bacteria whilst it is also a constituent of natural gas, sulfur deposits, volcanic gases and sulfur springs [2]. The unmediated release of high concentrations of sulfide can effectively overwhelm the checks and balances that operate under natural conditions and can considerably threaten surrounding ecosystems. Hence, there has been an increasing need for highly sensitive, fast-responding, portable devices for monitoring trace levels of sulfide in a variety of environmental and industrial applications.

There are several avenues for the detection of both sulfide and hydrogen sulfide; these embrace most instrumental methodologies [3]. Electrochemical techniques have

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provided several procedures as realized in the plethora of available potentiometric [4–6], galvanostatic [7,8] and amperometric [9–18] devices.

This paper reports on the greatly enhanced electrochemical reactivity and detection of sulfide at carbon nanotube (CNT) modified glassy carbon and carbon fiber electrodes. Carbon nanotubes have recently come to the forefront of electrochemical research because of their unique mechanical, electrical and chemical properties [19]. The electronic properties of these materials have been exploited as means of promoting the electron-transfer reaction for a wide range of biologically significant species including, proteins [20], NADH [21], neurotransmitters [22–24], cytochrome c [25] and cysteine [26,27]. Similarly, in the following sections we report on the electrochemical oxidation of sulfide and its enhanced voltammetric detection at glassy carbon electrodes modified with CNT (dispersed in dimethylformamide [28,29]).

2. Experimental

2.1. Electrochemical apparatus and chemicals

All reagents were obtained from Sigma, of the highest grade available and used without further purification.

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All solutions and subsequent dilutions were prepared daily, using doubly distilled water. Cyclic voltammetric measurements were conducted using a PGSTAT12 computer controlled potentiostat (Eco-Chemie BV, Utrecht, Netherlands) whilst a bioanalytical system (BAS) CV-27 voltammograph, in connection with a BAS X-Y recorder were used for the amperometric experiments. A standard three-electrode configuration was employed along with a typical cell volume of 15 mL. A modified (see below) or unmodified glassy carbon (BAS, 3 mm diameter) electrodes or a carbon fiber cylinder microelectrode (Alfa Aesar, Ward Hill, MA, USA, 5 mm length, 7 µm diameter) served as the working electrode with a platinum wire counter electrode and a Ag/AgCl (3 M KCl, model CHI111, CH Instruments, Austin, TX) reference electrode. The glassy carbon electrodes were polished using 1 and 0.05 µm alumina slurries on a polishing cloth (Buehler). A magnetic stirrer provided the convective transport during the amperometric measurements.

The pH dependence of the cyclic voltammetric response was examined in a Britton–Robinson buffer (40 mM CH₃CO₂H, 40 mM H₃PO₄ and 40 mM H₃BO₃). The pH of the solution was adjusted using concentrated sodium hydroxide. The water sample was collected from the Rio Grande river in Las Cruces (NM), and was used without any treatment.

2.2. Modification of the glassy carbon electrode

The nanotubes were immobilized onto the glassy carbon electrode by using dimethylformamide (DMF) as the dispersing agent [28,29]. In all cases the CNT were purified prior to use by stirring in a 2 M nitric acid solution for 20 h. Unless otherwise stated the casting solution was prepared by introducing 2 mg of nanotubes into 1 mL of DMF. These solutions were then sonicated for 3h to aid in the dissolution of the nanotubes. A 20 µL aliquot of these sonicated solutions was placed directly onto the glassy carbon surface and left to dry. In these studies two different nanotubes were used: the first, purchased from NanoLab Inc. (diameter 20-40 nm, length 1-5 µm, Newton, MA, USA) was prepared using a chemical vapor deposition (CVD), whilst the second was received from Bucky USA (Bu-201, Houston, TX, USA) and was prepared using the ARC discharge method.

2.3. Modification of the carbon fiber electrode

The carbon fiber electrodes were modified with CVD-CNT by successive dipping of carbon fiber electrode into a DMF solution containing 4 mg/mL CNT. The first coating was carried out by placing the electrode in the solution for a period of 20 s after which it was allowed to dry for 30 min. This was followed by three, 2 s dippings (with an intermediate 10 min drying).



Fig. 1. Cyclic voltammetric response (100 mV s^{-1}) of (A) the CVD-CNT and (B) the ARC-CNT-modified glassy carbon electrodes and (C) the bare glassy carbon electrode when placed in phosphate buffer (pH 7.4, 0.05 M) solution both in the presence and absence (dashed line) of 500 μ M sulfide.

3. Results and discussion

Fig. 1 displays cyclic voltammograms (CV) at CVD-CNT (A) and ARC-CNT (B) modified glassy carbon electrodes, as well as at the bare carbon surface (C) in the absence (dashed line) and presence of 500 µM sulfide. With both the unmodified and ARC-CNT electrodes a broad oxidation wave is observed in the presence of sulfide with a peak potential of +0.20 V. In contrast, the CVD-CNT-modified electrode exhibits remarkably different voltammetric behavior with a substantial negative shift in the oxidation-peak potential. At this electrode, the sulfide oxidation starts at -0.3 V, with the current rising until 0.0 V, and a leveling off thereafter. Notice also the different current intensities (from the different scales A, B versus C). No new reduction waves are observed in the reversed scan, indicating that the electrochemically generated product cannot be reduced in the potential range studied (see discussion below). The reduction wave observed both in the absence of sulfide at the CVD-CNT layer can be attributed to the reduction of redox active moieties on the carbon nanotube surface. This was consistent with previous results for the reduction of redox active quinone moieties on the electrode surface [30]. This was further confirmed by scan rate studies. The peak current was found to vary linearly with scan rate indicating a surface confined process occurring. This is entirely consistent with the reduction of the attached redox moieties on the electrode surface.

The apparent conflicting electrochemical behavior of the CVD-CNT layer compared to both the ARC-CNT-modified and bare carbon electrodes is in agreement with data for the oxidation of NADH and hydrogen peroxide [31]. The oxidation of both NADH and hydrogen peroxide was shown to occur at lower potentials at the CVD-CNT layer compared to the ARC layer [31]. The exact reason for the different electrochemical behavior of the ARC and CVD based CNT-modified electrodes is unclear at this point. Analysis of the background response of the individual electrodes (dashed lines, Fig. 1) reveals that the CVD-CNT-modified electrode exhibits a greatly enhanced residual current compared to the bare and ARC-CNT-modified electrodes. Furthermore, it can be seen that the CVD-CNT response shows a slight oxidative wave at +0.09 V along with a corresponding reduction wave at -0.19 V. This redox response is consistent with those observed previously and can be attributed to the presence of electroactive functionalities on the nanotube surface as discussed above [30-32].

Further evaluation of the oxidation process was conducted by studying the effect of scan rate on the anodic current for all three electrode substrates. It was found that the sulfide oxidation current increased linearly with the square root of scan rate over the 25–400 mV s⁻¹ range, suggesting that the process is transport controlled with little or no fouling of the electrode; a similar dependence was observed previously at boron-doped diamond electrodes [16].

To understand the reaction pathway for the electrochemical oxidation of sulfide at the CNT-modified electrode an investigation into the effect of the pH on the oxidation-peak potential was undertaken over the pH range of 3-11.5. The voltammetric profiles (not shown) were analogous to those detailed in Fig. 1A, with a new oxidative peak in the presence of sulfide and no new reduction signal. This indicates that the oxidation products cannot be reduced (in the potential range studied) over the entire pH range. The corresponding plot of oxidation-peak potential against pH is detailed in Fig. 2. This shows that as the pH is decreased the oxidation potential increases. Furthermore, it can be seen that the plot can be split into two linear regions which intersect each other. This intersection reflects the point at which the hydrogen sulfide becomes deprotonated to form the mono-protonated sulfide anion. Analysis of the figure shows that this occurs at pH 6.9, which compares favorable with literature values for the pK_a 's of



Fig. 2. A plot of the oxidation-peak potential against pH for the oxidation of 0.5 mM sulfide at the CVD-CNT-modified electrode. The peak potentials were measured using cyclic voltammetry recorded in a Britton–Robinson buffer over a wide pH range (scan rate = 100 mV s^{-1}).

sulfide [33]:

$$H_2S \rightarrow HS^- + H^+, \quad pK_a = 6.88,$$

 $HS^- \rightarrow S^{2-} + H^+, \quad pK_a = 14.15$

The analysis of the two linear regions was subsequently conducted; we first turn our attention to pH of the region (7–14) in which the presence of the mono-protonated sulfide anion dominates. These data produced a linear response with a slope of 32 mV/pH unit consistent with a two electron, one proton transfer occurring at the electrode surface. This can be tentatively attributed to the direct oxidation of the sulfide anion to sulfur as outlined below:

$$HS^- \rightarrow S + 2e^- + H^+$$

Analysis of the data detailed over the pH range of 3-6 produced a linear response with a slope 75 mV/pH unit. This process can be tentatively attributed to the oxidation of hydrogen sulfide to sulfate:

$$4H_2O + H_2S \rightarrow SO_4^{2-} + 8e^- + 10H^+$$

After understanding the mechanism for the oxidation of the sulfide under both acidic and alkaline conditions we turned our attention to optimizing the analytical response of the CVD-CNT sensor. This was initiated by studying the hydrodynamic voltammetric response as a means of determining the optimum potential for the amperometric detection of sulfide. In these experiments the potential was varied from -0.4 to 1.0 V at intervals of 0.1 V in the presence of 100 µM sulfide. Fig. 3 compares the hydrodynamic voltammograms obtained at different electrode surfaces (x: CVD, \bullet : ARC, +: bare). Analysis of the voltammetric response of the CVD-CNT-modified electrode (\times) reveals an oxidative current across the entire potential range; the current rises sharply around -0.3 V and levels off above -0.2 V. This result is qualitatively consistent with the cyclic voltammetric data of Fig. 1A, in which the sulfide oxidation



Fig. 3. Hydrodynamic voltammograms for $100 \,\mu\text{M}$ sulfide in pH 7.4, 0.05 M phosphate buffer at the CVD-CNT (×) and ARC-CNT (•) modified electrodes, as well as at the bare electrode (+).

wave emerges at -0.3 V. At both ARC-CNT-modified (\bullet) and bare glassy carbon (+) electrodes the oxidation process starts at 0.0 V, and the current rises rapidly until +0.3 V, then more slowly and faster again above +0.6 V. Note also the larger response of the ARC-CNT electrode, reflecting its larger surface area. These profiles are qualitatively consistent with the cyclic voltammetric signals of Fig. 1B and C. All subsequent CVD-CNT amperometric work employed a potential of +0.1 V. The electrocatalytic detection of hydrogen sulfide at the CVD-CNT sensor is coupled to a greatly improved stability. Fig. 4 compares current–time tracings for 100 μ M sulfide recorded at the CVD-CNT-modified (A) and bare (B) glassy carbon electrodes over a 60 min period. The response of the bare electrode diminishes rapidly with 85% decay observed over the entire period indicating a fast surface passivation. A substantially slower decay of 41% is observed at the CVD-CNT-modified electrode during the same operation. This enhancement in stability can be tentatively attributed to the macroporous structure of the nanotube layer.

The voltammetric data of Figs. 1 and 3 suggest that the CVD-CNT-modified electrode facilitates the low potential amperometric detection of sulfide. Fig. 5A examines this possibility by comparing the response at +0.1 V of the CVD-CNT (I) and ARC-CNT (II) modified electrodes, along with the response of the naked glassy carbon surface (III) to successive 12.5 μ M sulfide additions. As expected from the voltammetric profiles, the bare and ARC-CNT-modified electrodes display only negligible signals over the 12.5–125 μ M sulfide range. In contrast, the CVD-CNT sensor responds favorably to these sulfide additions, yielding steady-state signals within 10 s. Such low-potential sensitive measurements are indicated also in Fig. 5B from the response of the CVD-CNT electrode



Fig. 4. Stability of the response to $100 \,\mu$ M sulfide using CVD-CNT-modified (A) and unmodified (B) glassy carbon electrodes. Applied potential at +0.1 V (A) and +0.4 V (B). Electrolyte: pH 7.4, 0.05 M phosphate buffer.



Fig. 5. (A) Current-time recordings to successive 12.5 μ M sulfide additions at the (I) CVD-CNT-modified, (II) ARC-CNT-modified and (III) unmodified glassy carbon electrodes. Conditions as in Fig. 4A. (B) Current-time recordings to successive 1.25 μ M sulfide additions CVD-CNT-modified glassy carbon electrode; potential and buffer as in Fig. 4A.

Table 1

Analytical parameters obtained at the various nanotube modified and unmodified glassy carbon electrodes

Modification procedure	Sensitivity, $\mu A/\mu M$ (sulfide)	Linear range (µM)	n ^a	Limit of detection (µM) ^b
CVD	0.115	1.25-112.5	9	0.3
ARC	0.005	12.5-87.5	6	12.5
Bare	0.002	12.5-50	4	12.5

^a Number of data points in linear regression data.

^b Based on S/N = 3.

to successive additions of greatly lower $(1.25 \,\mu\text{M})$ sulfide concentrations. The favorable signal-to-noise characteristics of these data suggest a detection limit of around $0.3 \,\mu\text{M}$ (9 ppb, based on S/N = 3). Table 1 summarizes the analytical parameters resulting from the calibration experiments of Fig. 5. These data indicate that the CVD-CNT sensor leads to a highly linear response up to 112.5 μ M (3500 ppb) sulfide with a sensitivity of 0.113 μ A/ μ M (correlation coefficient, 0.996). A comparison of the analytical parameters obtained at the CVD-CNT-modified electrode with those of amperometric methodologies currently available for sulfide detection [3,9–18] reveals that the detection limit of 9 ppb compares favorably with those reported for these procedures (commonly in the range of 10–30 ppb [3]). The extension of the linear range up to 3500 ppb also competes favorably with the ranges reported for earlier devices [3]. The new protocol compares favorably with

Table 2									
Analytical	parameters	as	a	function	of	the	CNT	surface	loading

Concentration of nanotube (μg)	Sensitivity, $\mu A/\mu M$ [sulfide]	Linear range (µM)	n	Limit of detection $(\mu M)^b$
20	0.063	12.5–50	4	5.4
40	0.119	12.5-112.5	9	2.1
80	0.259	12.5–125	10	1.6

^a Based on the response to 10 successive $12.5\,\mu M$ sulfide additions.

^b Based on S/N = 3.

existing procedures [3] in terms of sensitivity, stability, and simplicity.

The surface loading of the CNT modifier has a profound effect upon the sulfide response. We examined the sensitivity to sulfide of electrodes modified with 20, 40 and 80 μ g of CVD-CNT. As indicated from Table 2, the sensitivity increases linearly with the CNT loading (from 0.063 to 0.259 μ A/ μ M, between 20 and 80 μ g) with a decrease in the detection limit observed as the loading was increased.

The detection process was next examined against a range of potential environmental interferences. The three main inorganic interferences investigated were nitrite, sulfite and nitrate. The corresponding hydrodynamic voltammograms obtained in the presence of 100 μ M of sulfide (×), 100 μ M sulfite (•) and 100 μ M nitrite (•) are detailed in Fig. 6. These profiles reveal that each analyte produces an oxidative signal with oxidation waves emerging at +0.20 V for sulfite and +0.70 V for nitrite. However, it can be seen that no signal is observed at +0.10 V (the detection potential for sulfide) and as such the presence of both nitrite and sulfite would not interfere with the analytical signal. Furthermore, no signal was observed for nitrate over the entire potential range studied.

The applicability of the approach was demonstrated through the recovery of sulfide in river water obtained from the Rio Grande. A standard addition protocol was adopted to enable the recovery of sulfide from the river water. In this case the water was spiked with 9 μ M sodium sulfide and diluted with an equi-volume of phosphate buffer (pH 7.4). Analysis of the corresponding standard addition plots



Fig. 6. Hydrodynamic voltammograms obtained at the CVD-CNT layer in 100 μ M sulfide (×), 100 μ M sulfite (\blacklozenge) and 100 μ M nitrite (\blacklozenge). Conditions as in Fig. 3.



Fig. 7. Current–time recordings to successive $25 \,\mu$ M sulfide additions at (A) the CVD-CNT-modified, and (B) unmodified carbon fiber electrodes. Potential and buffer as in Fig. 4A.

(not shown) produced a $95.5 \pm 4.4\%$ (n = 3) recovery of sulfide from the river water.

Finally, the possibility of detecting sulfide at nanotubemodified carbon fiber microelectrodes was examined. The development of an easily fabricated miniaturized, portable sensor is of high importance to both on-site industrial and environmental applications [2,8–11]. Fig. 7 details the current–time response of CNT-coated (A) and bare (B) carbon fiber electrodes (poised at +0.1 V) to increasing 25 μ M additions of sulfide. These results reveal how the CNT-modified carbon fiber provides a significant enhancement in the analytical signal compared to that of the bare carbon fiber electrode with a substantial increase in the sensitivity from 0.01 to 0.07 nA/ μ M and a corresponding decrease in the limit of detection (based on S/N = 3) from 40 to 12 μ M.

4. Conclusions

The voltammetric response of hydrogen sulfide at a glassy carbon electrode modified with both CVD- and ARC-prepared carbon nanotubes was examined and compared with those obtained at the bare glassy carbon electrode. The CVD-CNT-modified electrode exhibited an attractive low potential response towards sulfide in a manner analogous to the improvements observed previously for the oxidation of NADH [21] and hydrogen peroxide [34]. The sulfide oxidative response has been examined over a wide pH range and suitable oxidative pathways have been suggested for the oxidation process under both acidic and alkaline conditions.

The analytical utility of the device has been assessed for the direct determination of sulfide. A low detection limit of $0.3 \,\mu\text{M}$ was observed along with a wide linear dynamic range of 1.25-112.5 µM. Such performance characteristics, along with the high stability, compare favorably to those of existing methods for detecting sulfide [3]. Furthermore, the sulfide response was found to be selective and could be tailored by varying the surface loading of the carbon nanotube. The analytical utility of such a protocol was demonstrated with a river water sample. Finally the electrochemical response of a CVD-CNT-modified carbon fiber has been assessed. The dramatic enhancement in the sensitivity (compared to that obtained at the bare carbon fiber electrode) holds great promise for the development of a miniaturized sensor for hydrogen sulfide in connection to coverage with a gas permeable membrane, such as Gore Tex [8,15,17] or silicone [10].

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