

Electrochemical determination of iodide on a vanadium oxide–polypropylene carbonate coated glassy carbon electrode

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Abstract

A vanadium oxide-modified glassy carbon electrode was simply and conveniently fabricated by casting vanadium tri(isopropoxide) oxide ($\text{VO}(\text{OC}_3\text{H}_7)_3$) and poly(propylene carbonate) (PPC) onto the glassy carbon electrode surface. The electrochemical properties of iodide at the $\text{VO}(\text{OC}_3\text{H}_7)_3$ -PPC film-modified glassy carbon electrode were investigated by cyclic voltammetry, and an anodic peak was observed at approximately +0.71 V (vs. SCE). Based on this, a sensitive and convenient electrochemical method was proposed for the determination of iodide. Flow injection amperometry (FIA) exhibited a good linear relationship with the concentration of iodide in the range of $5 \times 10^{-7} \text{ mol L}^{-1}$ and $1 \times 10^{-3} \text{ mol L}^{-1}$, and the detection limit was $1 \times 10^{-7} \text{ mol L}^{-1}$. Quantitative recovery of iodide in synthetic samples has been obtained and the interferences from different cations and anions have been studied. The method has been successfully applied to the determination of iodide in dry edible seaweed. The concentrations of iodide measured by this method are in good agreement with those obtained by spectrophotometric method.

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Keywords: Vanadium tri(isopropoxide) oxide; Poly(propylene carbonate); Iodide; Flow injection amperometry; Electrocatalytic oxidation

1. Introduction

Iodine is an essential trace element playing an important role in mental development, growth and basic metabolisms [1]. The lack of this thyroid hormone causes poor mental and physical development in children and enlargement of the thyroid (goitre) in adults. At the same time, an excess of iodine or iodide can produce goitre and hypothyroidism as well as hyperthyroidism [2]. Therefore, the determination of iodide and iodine species is important in a variety of fields such as food [3], clinical and biological sciences [4,5], and also in environmental and industrial applications [6,7].

Several analytical methods have been applied for the determination of iodate and iodide, such as titrimetry [8], chemiluminescence [9], ion chromatography [10], ICP-MS [11],

diffuse reflectance spectroscopy [7], inductively coupled plasma-atomic emission spectrometry [6], flow-injection analysis [4], gas chromatography-mass spectrometry [12]. But most of them were expensive and relatively complicated. Some electrochemical procedures were proposed recently. Besides the well-known cathodic stripping voltammetry using mercury electrodes [13], were most often based on anodic or adsorptive stripping voltammetry, applying either potentiostatic accumulation [14,15] or preconcentration at open-circuit through the formation of ion pairs at chemically modified electrodes [16–22].

Polymer film-modified electrodes can be different from other modification methods such as adsorption and covalent bonding because they commonly involve multilayer adsorption, which can provide more ‘active sites’ resulting in obvious analytical signals. Together with its ease of preparation, good stability and reproducibility, the polymer film-modified electrode is particularly advantageous for electro-analytical research [23]. Many researches were put on the

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polymer matrix, such as poly(ethylene oxide) (PEO) [24], poly(vinyl chloride) (PVC) [25], poly(methyl methacrylate) (PMMA) [26] polyacrylonitrile (PAN) [27], poly(vinylidene fluoride-hexafluoropropylene) (PVDF-HFP) [28–30], and so on. Poly(propylene carbonate) (PPC) is a kind of hydroxyl-terminated aliphatic polycarbonate composed of carbon dioxide and propylene epoxide [31] that can be used as adhesives, solid electrolytes, photoresists, barrier materials, flexibilizers and plasticizers [32,33]. Because PPC and $\text{VO}(\text{OC}_3\text{H}_7)_3$ are analogous, they can possess miscibility; $\text{VO}(\text{OC}_3\text{H}_7)_3$ would be held well in PPC-modified film. In this paper, we developed a novel vanadium oxide-modified and polypropylene carbonate-coated glassy carbon electrode, and studied the electrochemical behavior of iodide on it and its application. Furthermore, this kind of electrode has been used to flow injection amperometric system for the determination of iodide. This new procedure has following advantages such as sensitivity, rapidity and excellent reproducibility.

2. Experimental

2.1. Reagents

Poly(propylene carbonate) (PPC) (Ave. $M_n \sim 50,000$) and vanadium tri (isopropoxide) oxide ($\text{VO}(\text{OC}_3\text{H}_7)_3$) were obtained from Aldrich Chemical Company, Inc. Potassium iodide, LiClO_4 and tetrahydrofuran (THF) were obtained from Tianjin Chemical Reagent Plant and used without further purification. Britton–Robinson (B–R) buffers were obtained by titrating a mixed acid solution (0.04 mol L^{-1} boric acid, 0.04 mol L^{-1} phosphoric acid and 0.04 mol L^{-1} acetic acid) with 0.2 mol L^{-1} NaOH to the required pH. Pure water was used throughout, obtained by means of Millipore Q water purification set. All other chemicals were of analytical grade and were used without further purification.

2.2. Measurements

Electrochemical experiments were performed with a CHI 630A Electrochemical Analyzer (CH instruments, USA) in a conventional three-electrode cell. The working electrodes were modified or unmodified glassy carbon electrodes (Model CHI 104). Platinum electrode was used as the counter electrode and a saturated calomel electrode (SCE) as the reference electrode. All potentials were measured and reported versus SCE.

Flow injection analysis experiments were performed with a FIA-3100 Flow Injection Analyzer (Beijing Titan Instruments Co., Ltd.) in the flow detection cell. Infrared spectrum of the sample was recorded with Perkin-Elmer SPECTRUM ONE FT-IR spectrophotometer in the $400\text{--}4000 \text{ cm}^{-1}$ regions using a sample on the KBr plate.

All experiments were carried out at room temperature. The buffer and sample solutions were purged with purified

nitrogen for at least 15 min to remove oxygen prior to the beginning of series of experiments.

2.3. Preparation of the film

The method of immobilizing $\text{VO}(\text{OC}_3\text{H}_7)_3$ on the surface of glassy carbon electrode is described briefly as follows. A glassy carbon electrode was polished before each modification with 1, 0.3 and $0.05 \mu\text{m}$ alumina powder, respectively, rinsed thoroughly with doubly distilled water between each polishing steps, then washed successively with 1:1 nitric acid, acetone and doubly distilled water in ultrasonic bath and dried in air. First, organically modified PPC gel was prepared by mixing PPC with LiClO_4 in THF for 24 h at room temperature. This solution was freshly prepared before use. Second, $\text{VO}(\text{OC}_3\text{H}_7)_3$ was dissolved in THF and saturated $\text{VO}(\text{OC}_3\text{H}_7)_3$ sol was obtained. Two microlitres of $\text{VO}(\text{OC}_3\text{H}_7)_3$ sol was dropped onto the surface of a GCE, finally coated with PPC sol, and allowed to dry at room temperature for 48 h. The $\text{VO}(\text{OC}_3\text{H}_7)_3$ –PPC film-modified GCE was obtained.

2.4. Procedure

The flow injection system used in this work was consisted of flow injection analyzer and electrochemical detection cell. The pump was started and pH 2.4 B–R buffer flowed through the system until a stable baseline was recorded. Then, different-concentration mixing solution of potassium iodide and pH 2.4 B–R buffer was injected into the cell. The anodic peak current was measured with electrochemical analyzer at $+0.8 \text{ V}$ versus SCE.

2.5. Sample preparation

A suitable amount of dry edible seaweed was weighed into a china crucible, and 2 ml of deionized water and 2 ml of 2 M KOH solution were added and stirred until a paste was formed. It was transferred into an oven for 1–2 h at $80\text{--}100^\circ\text{C}$, and then into a high-temperature oven for 4–6 h at $550\text{--}600^\circ\text{C}$. After carbonization, it was washed with hot deionized water, filtered into a 50 ml volumetric flask and adjusted to pH 7 with concentrated H_2SO_4 solution, and then diluted to the mark with deionized water. Thus, the sample is obtained.

3. Results and discussion

3.1. Spectral characteristics

In Fig. 1, typical FT-IR spectral patterns are shown for $\text{VO}(\text{OC}_3\text{H}_7)_3$ –PPC, $\text{VO}(\text{OC}_3\text{H}_7)_3$, PPC and V_2O_5 . The characteristic bands of $\text{VO}(\text{OC}_3\text{H}_7)_3$ and PPC can be observed in Fig. 1b and c, respectively. The spectrum of $\text{VO}(\text{OC}_3\text{H}_7)_3$ –PPC composite (Fig. 1a) is similar to that

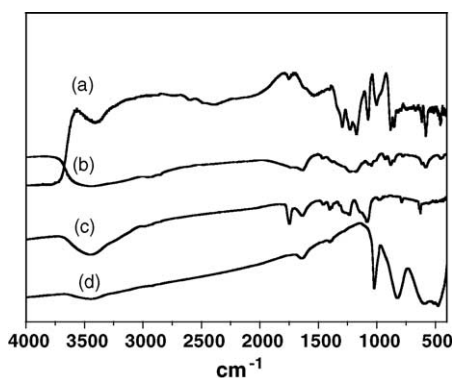


Fig. 1. FT-IR spectra of (a) $\text{VO}(\text{OC}_3\text{H}_7)_3$ -PPC, (b) $\text{VO}(\text{OC}_3\text{H}_7)_3$, (c) PPC and (d) V_2O_5 .

of $\text{VO}(\text{OC}_3\text{H}_7)_3$ (Fig. 1b). It was also observed from the spectra that there was a slight change in the band positions of Fig. 1b, suggesting a weak interaction between the $\text{VO}(\text{OC}_3\text{H}_7)_3$ and PPC. Besides, it is possible to note that the bands for $\text{VO}(\text{OC}_3\text{H}_7)_3$ -PPC are narrower than those for the $\text{VO}(\text{OC}_3\text{H}_7)_3$. This effect can be attributed to coulombic interactions between the two organic polymeric chains, although we are not able to discern the contribution of each one and more works are underway to further investigation of this point. Fig. 1d shows the spectra of V_2O_5 . From Fig. 1b and d, the difference of main bands between $\text{VO}(\text{OC}_3\text{H}_7)_3$ and V_2O_5 can be seen. It illuminated that $\text{VO}(\text{OC}_3\text{H}_7)_3$ in the film is not hydrolyzed.

3.2. Electrochemical behavior of the $\text{VO}(\text{OC}_3\text{H}_7)_3$ -PPC film-modified glassy carbon electrode

Fig. 2 shows the cyclic voltammograms at a bare glassy carbon electrode, a PPC film-modified glassy carbon electrode and a $\text{VO}(\text{OC}_3\text{H}_7)_3$ -PPC film-modified glassy carbon electrode, respectively. It can be seen that in the potential range from +1.1 to -1.0 V, no redox peak was obtained at the bare glassy carbon electrode (Fig. 2a). There is a ca-

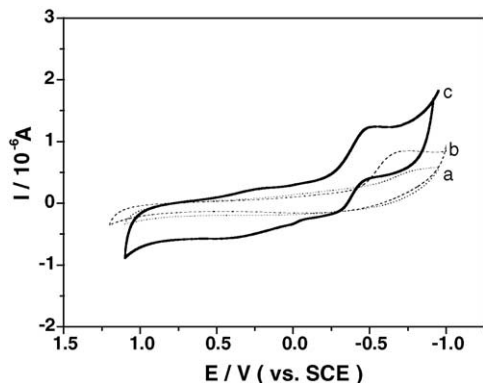
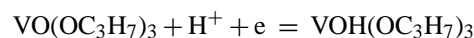


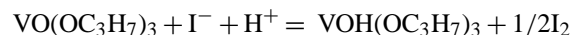
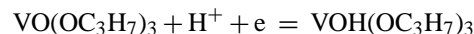
Fig. 2. Cyclic voltammograms of a bare glassy carbon electrode (a), PPC film-modified glassy carbon electrode (b) and $\text{VO}(\text{OC}_3\text{H}_7)_3$ -PPC film-modified glassy carbon electrode (c) in pH 2.4 B-R buffer. Scan rate: 50 mV s^{-1} .

thodic peak at about -0.7 V in the PPC film-modified glassy carbon electrode (Fig. 2b). Two pair of redox peaks appeared in the $\text{VO}(\text{OC}_3\text{H}_7)_3$ -PPC film-modified glassy carbon electrode (Fig. 2c). The mean peak potential $E_{1/2} = (E_{\text{pa}} + E_{\text{pc}})/2$ are +0.38 V (1, 1') and -0.40 V (2, 2') with peak potential separations of 0.28 V (1, 1') and 0.20 V (2, 2'), respectively. These peaks can be due to two non-equivalent sites in the $\text{VO}(\text{OC}_3\text{H}_7)_3$ -PPC film. At the higher potential ($E_{1/2} = +0.38 \text{ V}$), H^+ is incorporated into the $\text{VO}(\text{OC}_3\text{H}_7)_3$ and, at the same time, V^{5+} is reduced to V^{4+} by accepting an electron. In addition, the peak currents were linearly proportional to the square root of scan rates in the range of 10–500 mV s^{-1} , which indicated the electron transfer process in this film is controlled by diffusion. At the lower potential ($E_{1/2} = -0.4 \text{ V}$), the wide and asymmetric pattern of the redox peaks can be related to the overlapping of the cathodic peak of $\text{VO}(\text{OC}_3\text{H}_7)_3$ and PPC. Its peak currents were not proportional to scan rates or the square root of scan rates in the range of 10–500 mV s^{-1} , which also indicated this peak suggested a weak interaction between the $\text{VO}(\text{OC}_3\text{H}_7)_3$ and PPC. The electrochemical reaction process can be represented as follows:



3.3. Electrochemical responses of iodide at the $\text{VO}(\text{OC}_3\text{H}_7)_3$ -PPC film-modified glassy carbon electrode

The voltammetric behavior of iodide on bare glassy carbon electrode and $\text{VO}(\text{OC}_3\text{H}_7)_3$ -PPC-modified glassy carbon electrode was investigated using cyclic voltammetry between 1.1 and -1.0 V. The cyclic voltammograms were obtained in the presence of $2 \times 10^{-4} \text{ mol L}^{-1} \text{ I}^-$ at bare glassy carbon electrode (Fig. 3A). The catalytic oxidation of iodide on the $\text{VO}(\text{OC}_3\text{H}_7)_3$ -PPC film-modified electrode can be seen clearly in Fig. 3B. On adding iodide to the cell, it was found that the anodic peak current at the $\text{VO}(\text{OC}_3\text{H}_7)_3$ -PPC film-modified electrode was larger than at the bare glassy carbon electrode. The anodic peak potential (E_{pa}) is about 714 mV (vs. SCE), which is 64 mV lower than that at bare glassy carbon electrode. As we know, the increment of catalytic current and decrease in overpotential are two important factors to evaluate the catalytic effect. These results show that the $\text{VO}(\text{OC}_3\text{H}_7)_3$ -PPC film-modified glassy carbon electrode can catalyze oxidation of iodide and this film can be used as a sensor for determination of iodide. The oxidation of iodide can be ascribed to the $\text{VO}(\text{OC}_3\text{H}_7)_3/\text{VOH}(\text{OC}_3\text{H}_7)_3$. The electrochemical reaction process can be represented as follows:



where $\text{VO}(\text{OC}_3\text{H}_7)_3$ and $\text{VOH}(\text{OC}_3\text{H}_7)_3$ represent the oxidized and reduced forms of the vanadium redox couple. I_2

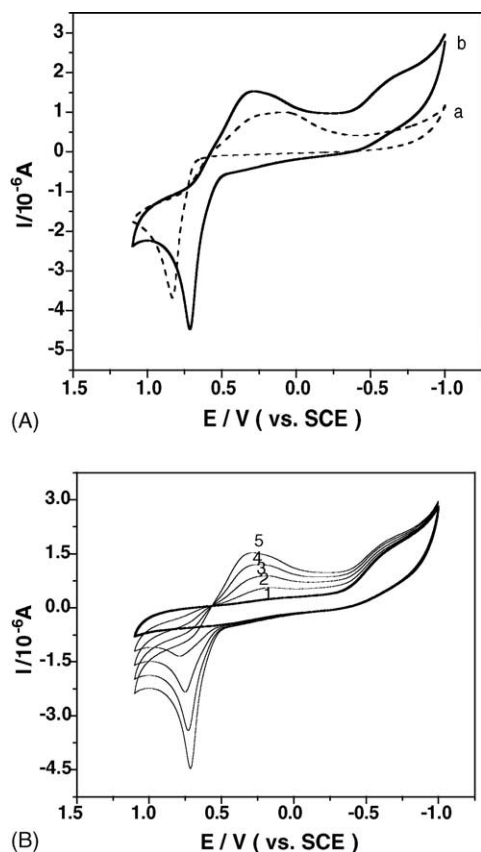


Fig. 3. (A) Cyclic voltammograms of $2 \times 10^{-4} \text{ mol L}^{-1}$ iodide in the pH 2.4 B–R buffer at bare glassy carbon (a) and a $\text{VO}(\text{OC}_3\text{H}_7)_3$ -PPC film-modified glassy carbon electrode (b). (B) Cyclic voltammograms of the different concentrations of iodide in the pH 2.4 B–R buffer at a $\text{VO}(\text{OC}_3\text{H}_7)_3$ -PPC film-modified glassy carbon electrode: (1) 0 mol L^{-1} , (2) $5 \times 10^{-5} \text{ mol L}^{-1}$, (3) $1 \times 10^{-4} \text{ mol L}^{-1}$, (4) $1.5 \times 10^{-4} \text{ mol L}^{-1}$, (5) $2.0 \times 10^{-4} \text{ mol L}^{-1}$. Scan rate: 50 mV s^{-1} .

obtained in the anodic process is reduced again in the cathodic process.

Fig. 4 shows that the peak currents were linearly proportional to the square root of scan rates in the range

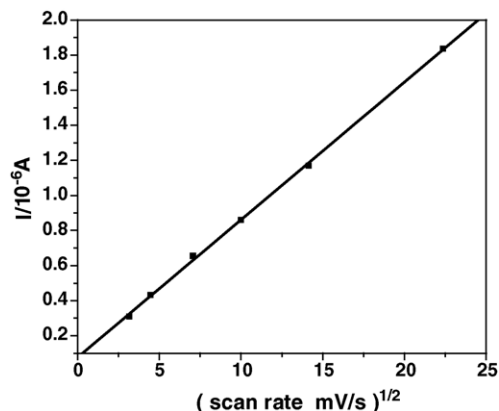


Fig. 4. Influence of the scan rate on the anodic peak current at $\text{VO}(\text{OC}_3\text{H}_7)_3$ -PPC film modified glassy carbon electrode in the presence of $5 \times 10^{-5} \text{ mol L}^{-1}$ iodide. Scan rates: 10 mV s^{-1} , 20 mV s^{-1} , 50 mV s^{-1} , 100 mV s^{-1} , 200 mV s^{-1} , 500 mV s^{-1} .

of $10\text{--}500 \text{ mV s}^{-1}$. Thus, the oxidation of iodide at the $\text{VO}(\text{OC}_3\text{H}_7)_3$ -PPC film-modified glassy carbon electrode in pH 2.4 B–R buffer is controlled by diffusion.

3.4. Effect of supporting electrolyte

To optimize the determination conditions of iodide, the effects of pH value and buffer systems such as, pH 2.0–7.0 B–R buffer, pH 3.5–6.0 acetic acid–acetate buffer and pH 2.0–4.0 H_2SO_4 – Na_2SO_4 , were examined. The result showed that highest peak current of iodide was obtained in pH 2.4 B–R buffer, otherwise the background current is very low and the oxidation peak is well defined. Thus, pH 2.4 B–R buffer was used as the supporting electrolyte for determining iodide.

3.5. Stability studies of $\text{VO}(\text{OC}_3\text{H}_7)_3$ -PPC film modified electrode

In general, the modified electrode has a disadvantage of leakage of the modifier. So, the stability study of the polymer-modified electrode is necessary. The stability of the $\text{VO}(\text{OC}_3\text{H}_7)_3$ -PPC film electrode was investigated. In the potential range $+1.1$ to -1.0 V versus SCE, in pH 2.4 B–R buffer and at 50 mV s^{-1} scan rate, after 100 cycles the current response was almost unchanged. When the modified electrode was stored in the air for more than 1 month, the current response decreased by 5%. The current response of the modified electrode towards iodide has also good repeatability. The relative standard deviation (R.S.D) is 3.5% for eight determinations of $2.0 \times 10^{-4} \text{ mol L}^{-1}$ iodide in pH 2.4 B–R buffer. These experiments indicate that the $\text{VO}(\text{OC}_3\text{H}_7)_3$ -PPC film modified electrodes have good stability and reproducibility.

3.6. Detection of iodide by flow injection analyzer

In this paper, amperometric i - t curve was used in the different potentials covering the range $0.3\text{--}1.0 \text{ V}$ in the pH 2.4 B–R buffer containing $5 \times 10^{-5} \text{ mol L}^{-1} \text{ I}^-$. The response time required to reach 95% of the steady-state current was less than 10 s after the addition of iodide. These results show that the current response of the modified electrode is rapid. So, FIA can be used to detect the concentration of iodide.

In the FIA detection process, the injection time or sampling time, the washing time, the flowing rate of buffer solution and applied potential are important factors. The peak current increased with the injection time increasing in the range of 2 and 6 s, but the ratio of the signal and background decreased. So, 2 s is selected as an optimum injection time. The current went back the baseline in about 20 s, so 30 s is optimum washing time. The less the flowing rate is, the wider the peak width is. The optimum flowing rate was selected as 4 mL/min . In FIA detection of the different potentials covering the range $0.3\text{--}1.0 \text{ V}$ in the pH 2.4 B–R buffer in the presence of $5 \times 10^{-4} \text{ mol L}^{-1}$, the peak current is largest at 0.8 V (Fig. 5).

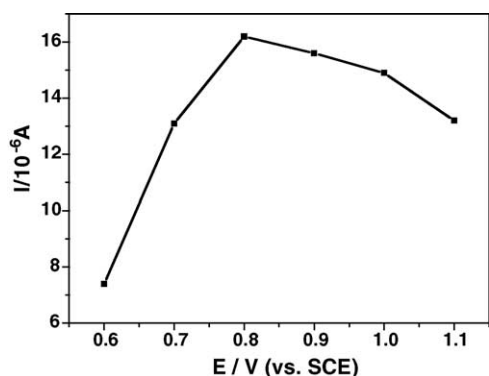


Fig. 5. Relationship between the current and applied potential at VO(OC₃H₇)₃-PPC film-modified glassy carbon electrode in the presence of 5×10^{-4} mol L⁻¹ iodide.

As stated above, the optimum conditions were as follows: the injection time, 2 s; the washing time, 30 s; the flowing rate, 60 r/min and applied potential, 0.8 V.

Under the selected conditions, FIA *i-t* graphs obtained for a series of iodide solutions with various concentrations are shown in Fig. 6A. Fig. 6B shows that in the range of 5×10^{-7} mol L⁻¹ and 1×10^{-3} mol L⁻¹, the response cur-

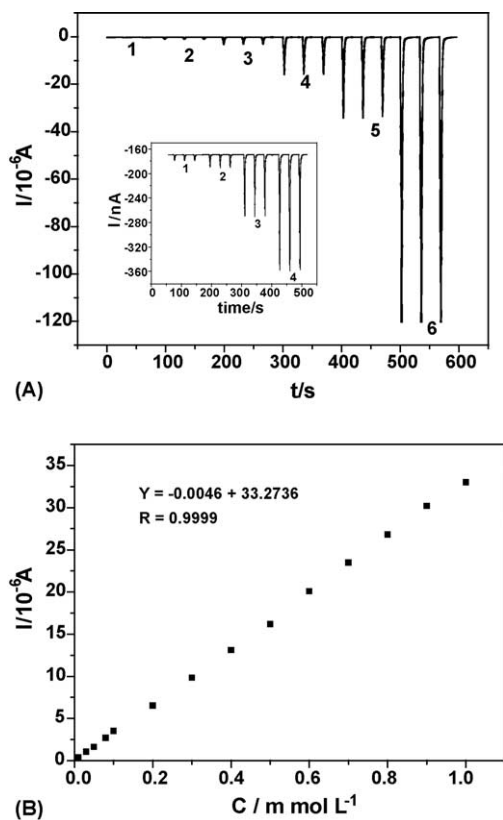


Fig. 6. (A) Variation of the response currents with iodide concentrations in the pH 2.4 B-R buffer at a VO(OC₃H₇)₃-PPC film-modified GCE. Applied potential: 0.8 V. (B) FIA *i-t* graphs of the different concentrations of iodide in the pH 2.4 buffer at a VO(OC₃H₇)₃-PPC film-modified GCE: (1) 1×10^{-5} mol L⁻¹, (2) 5×10^{-5} mol L⁻¹, (3) 1×10^{-4} mol L⁻¹, (4) 5×10^{-4} mol L⁻¹, (5) 1×10^{-3} mol L⁻¹, (6) 5×10^{-3} mol L⁻¹.

rents exhibited a good linear relationship with the concentrations of iodide and the detection limit was 1×10^{-7} mol L⁻¹. The relative standard deviation of the peak current *I*_{pc} to 10 repeated injections of 5×10^{-4} mol L⁻¹ iodide was 3.2%. The linear response range and mainly the detection limit were much better than those for amperometric sensors for iodide described recently in the literature [13–22].

3.7. Interferences

Interference effects were investigated by testing the responses of the modified electrode to Cl⁻, Br⁻, ClO₃⁻, BrO₃⁻, IO₃⁻, SO₄²⁻, Na⁺, Mg²⁺, Ca²⁺, Ba²⁺ and As³⁺ because these species often exist in the sample with I⁻. In order to study the influence of various species on the determination of iodide, a fixed amount of iodide was taken with different amounts of foreign species and the recommended procedure was followed. A relative error of 5% was considered tolerable. Keeping the concentration of iodide at a level of 5.0×10^{-6} mol L⁻¹, no significant interference was observed from the following species: Cl⁻ (50), Br⁻ (50), ClO₃⁻ (250), and BrO₃⁻ (150), where the values in parentheses are the concentration ratios. The other species did not interfere in the experiments. So the VO(OC₃H₇)₃-PPC film-modified electrode has good selectivity for iodide.

3.8. Analytical application

To examine the possible use of the modified electrode in a practical application, artificial water samples were determined. The composition of the artificial water samples was as follows: 1.0×10^{-4} mol L⁻¹ Cl⁻, Br⁻, BrO₃⁻, ClO₃⁻, SO₄²⁻, and 0.1 – 1.0×10^{-3} mol L⁻¹ I⁻. Typical results obtained for the water samples are summarized in Table 1. The results for the determination of iodide are in agreement with the known contents.

In addition, the modified electrode was applied to the analysis of iodide in dry edible seaweed. In order to evaluate the validity of the modified electrode for the determination of iodide, the recovery of detecting samples were carried out by iodide standard increment. An appropriate volume of the sample was added to the cell and tested according to the above procedure. The results obtained are shown in Table 2. These results were compared with that from a spectrophotometric method and were shown to be in agreement. Thus, the proposed method can be successfully applied to real samples.

Table 1
Results of the determination of iodide ion in artificial water samples

Sample number	Iodide added (mmol L ⁻¹)	Iodide found ^a (mmol L ⁻¹)
1	0.10	0.13
2	0.30	0.27
3	0.50	0.48
4	0.80	0.84
5	1.00	0.98

^a Average of six determinations.

Table 2
Results of iodide in different samples

Sample number	Found ^a (I, mg/kg)	Added (I, mg/kg)	Total ^a (I, mg/kg)	Recovery (%)	Spectrophotometry (I, mg/kg)
1	24.2	10.0	35.1	103.7	23.6
2	31.8	20.0	50.6	96.2	33.1
3	40.1	20.0	61.8	104.2	38.4

^a Average of six determinations.

4. Conclusions

In this paper, VO(OC₃H₇)₃–PPC film can be simply and conveniently modified on the surface of a glassy carbon electrode. With this work it was proved that VO(OC₃H₇)₃ is a good modifier, which can catalyze oxidation of iodide. In addition, the modified electrode was successfully applied to the analysis of iodide in dry edible seaweed.

When compared to the previous reports for this determination, the advantages are: (1) without pre-treatment, (2) without preconcentration and (3) lower detection limit. So, this methodology could be used for rapid detection of iodide.

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