

Determination of 1-Naphthol Concentration on Electrode Modified with Electrochemically Polymerized β -Cyclodextrin Film

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A glassy carbon electrode (GCE) modified with poly β -cyclodextrin (β -CD) film (β -CD/GCE) was prepared. The electrochemical properties of the β -CD/GCE were explored by cyclic voltammetry (CV) and AC impedance measurement, and the obtained results indicate significant improvements in impedance and electrocatalysis behavior. Further experimental results show that in HAc-NH₄Ac solution (pH 5.55), the electrocatalysis of 1-naphthol is very clear on the polymerized β -CD/GCE. The peak current increased linearly with the concentration of 1-naphthol in the range of 2.05×10^{-3} – 1.07×10^{-6} mol·L⁻¹, and the detection limit was 3.06×10^{-7} mol·L⁻¹ at an SNR of 3:1. The modified electrode was applied, with satisfactory results, to the determination of 1-naphthol concentration in tap water, and its recovery rate was between 97.3 and 103%.

1. Introduction

1-Naphthol, which is used as the raw material of the insecticide Sevin, is now in great demand. It is a large-scale industrial chemical that is widely used in the manufacture of dyes and insecticides, and is also a secondary product of chemical, paint, paper, and pesticide industries. In addition, it is a kind of phenolic compound with high toxicity and thus has a profound effect on the environment and health of people.^(1–3) Currently, the common methods of determining the concentration of 1-naphthol include high-performance liquid chromatography (HPLC),^(4–6) the use of quartz crystal microbalance (QCM),⁽⁷⁾ immune cytochemistry,⁽⁸⁾ fluorescence methods,^(9–14) and synchronous-derivative phosphorimetric determination.⁽¹⁵⁾ These methods have their own characteristics: some of them require expensive instruments and complicated sample processing, and they are not convenient to use in real-time field testing. Therefore, a rapid and accurate determination of 1-naphthol concentration is of practical significance. At present, the field environment real-time detection, mobile laboratory, portable detection instruments, and so on have a good prospect of development. The electrochemical method with simple and rapid features is gradually widely used. Therefore, in this study,

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we aim to establish a rapid and simple electrochemical method for the determination of 1-naphthol. β -cyclodextrin (β -CD) is a functional supramolecule with a hydrophobic interior and a hydrophilic exterior, and may selectively recognize some organic or inorganic small molecules through nonbonding intermolecular forces.⁽¹⁶⁾ 1-Naphthol molecules entering into the cavity of β -CD tend to accumulate on one side, which enhances the nonbonding forces between 1-naphthol and β -CD, resulting in an enhancement of the electrochemical response of 1-naphthol on a glassy carbon electrode modified with poly β -CD film (β -CD/GCE). In addition, β -CD, which has a special cylindrical structure with external hydrophilic and internal hydrophobic chambers, can be combined with 1-naphthol to form the host- and guest-containing compounds. Therefore, it can be applied to the detection of 1-naphthol. In a preliminary work, we carried out extensive research on polymer electrodes, such as an electrode modified with a Prussian blue analog doped with copper (II) ions^(17,18) and a poly(methyl red)-modified electrode.⁽¹⁹⁾ We have also conducted some research on β -CD-film-modified electrodes by electrochemical polymerization.⁽²⁰⁾ Using the results of previous work, we developed a new test system using the β -CD/GCE prepared by electrochemical polymerization, which has made the rapid and accurate determination of the concentration of 1-naphthol a reality. Moreover, linear calibration ranges are wider and the detection limit is higher than literature values.^(21,22)

2. Materials and Methods

2.1 Apparatus and reagents

All the electrochemical measurements were carried out on a CHI660C electrochemical workstation (Chenhua Instrument Co., Ltd., Shanghai, P. R. China). The KQ-400DB numerical control ultrasonic wave washer was purchased from Ultrasonic Instrument Co., Ltd. (Kunshan, P. R. China). A saturated calomel electrode (SCE), a platinum electrode, a glassy carbon electrode (GCE) of 4 mm diameter, a pH meter (PHS-3C, Shanghai Dapu Instrument Co., Ltd.), and the BS110S electronic balance (Shanghai Jingsheng Scientific Instrument Co., Ltd.) were also used. β -CD, 1-naphthol, $K_3[Fe(CN)_6]$, concentrated H_2SO_4 , concentrated HNO_3 , KCl, NaOH, KH_2PO_4 , K_2HPO_4 , HAc, and NH_4Ac reagents were of analytical grade and used without further purification.

2.2 Preparation of β -CD/GCE electrode

The GCE ($\Phi = 4$ mm) was polished with emery paper, then washed ultrasonically for 3 min each in 1:1 NaOH solution, 1:1 HNO_3 solution, pure alcohol, and distilled water, successively. The cleaned GCE was put in a $0.5 \text{ mol}\cdot\text{L}^{-1}$ solution of sulfuric acid, then cyclic voltammetry (CV) experiments were carried out in a potential range from -1.0 to 1.0 V with a scan rate of 0.10 V/s until the peak current became stable, thus attaining an activation electrode. Next, the activation electrode was put in a saturated solution of β -CD containing $0.1 \text{ mol}\cdot\text{L}^{-1}$ potassium chloride and $0.01 \text{ mol}\cdot\text{L}^{-1}$ sodium hydroxide for cyclic voltammetric experiments in a potential range from -1.0 to 1.0 V with a scan rate of 0.05 V/s until the peak current became stable. The working

electrode was taken out, rinsed, and then dried in air. A baby-blue film was observed on the surface of the working electrode, indicating the successful preparation of a steady β -CD/GCE.

2.3 Analytical procedure

A conventional three-electrode system, including a β -CD/GCE or glassy carbon working electrode, a platinum-wire counter electrode, and a reference SCE, was employed using the CHI660C electrochemical workstation. Impedance from 0.1 MHz down to 1 Hz was measured. The amplitude of the sinusoidal voltage was chosen to be 5 mV. Cyclic voltammograms were recorded from -0.8 to 0.9 V under various conditions, for example, scan rate, electrolyte, and 1-naphthol concentration. All the experiments were carried out at room temperature, and nitrogen was bubbled into the sample to remove the oxygen.

3. Results and Discussion

3.1 Characterization of β -CD/GCE electrode

In 1.01×10^{-3} mol·L $^{-1}$ potassium ferricyanide solution containing 0.1 mol·L $^{-1}$ potassium chloride, the β -CD/GCE and activation electrode were used as the working electrodes and the cyclic voltammograms were recorded from -0.8 to 0.9 V with a scan rate of 0.10 V/s. The obtained results are presented in Fig. 1. Compared with the CV response on a bare GCE, a large well-defined peak appears on the β -CD/GCE. The peak potentials are 0.015 and 0.268 V. These results indicate that the β -CD/GCE shows good electrocatalytic action for potassium ferricyanide, but a bare GCE does not. The polymerized β -CD/GCE was successfully prepared by electrochemical polymerization, and the electrocatalytic property of this modified GCE showed a significant change.

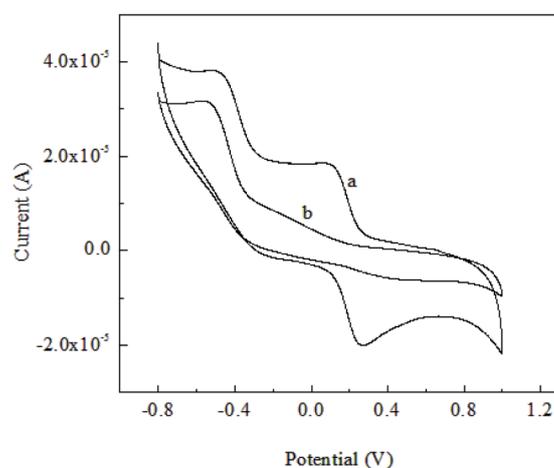


Fig. 1. Cyclic voltammograms for (a) β -CD/GCE and (b) GCE in $K_3[Fe(CN)_6]$ solution.

Typical complex impedance plots for the β -CD/GCE and GCE in the $1.01 \times 10^{-3} \text{ mol}\cdot\text{L}^{-1}$ potassium ferricyanide solution containing $0.1 \text{ mol}\cdot\text{L}^{-1}$ potassium chloride are shown in Fig. 2. There is a high-frequency semicircle on the GCE and a quarter-circle on the β -CD/GCE. These results show that poly-(β -CD) was successfully modified on the GCE by electrochemical polymerization, resulting in a clear change in impedance. By combining Figs. 1 and 2, we can see that the peak current of potassium ferricyanide is low for a bare GCE, and the corresponding impedance is larger. However, for the β -CD/GCE, the peak current of potassium ferricyanide markedly increases and its impedance decreases. These results show that the poly-(β -CD) participates in the electron transfer process on the electrode surface and enhances the electron conductivity.

3.2 Electrochemical behavior of 1-naphthol on β -CD/GCE

Figure 3 shows the cyclic voltammograms of $1.03 \times 10^{-3} \text{ mol}\cdot\text{L}^{-1}$ 1-naphthol solution (curves a and c) and the blank solution (curves b and d) on the β -CD/GCE (curves a and b) and the bare GCE (curves c and d) in the pH 5.55 HAc-NH₄Ac buffer solution measured with a scan rate of 0.12 V/s. In the blank solution, there is no clear peak current on either the β -CD/GCE or the bare GCE. In the $1.03 \times 10^{-3} \text{ mol}\cdot\text{L}^{-1}$ 1-naphthol solution, the CV response of 1-naphthol on the β -CD/GCE exhibits a large well-defined oxidation peak whose peak current is $-1.871 \times 10^{-5} \text{ A}$. Compared with the 1-naphthol peak current of $-4.779 \times 10^{-6} \text{ A}$ for the bare GCE, the peak current has almost quadrupled. This indicates that the electrochemical polymerization of β -CD on the GCE greatly improved the electrochemical reactivity of 1-naphthol and markedly enhanced the electrochemical response of 1-naphthol on the β -CD/GCE.

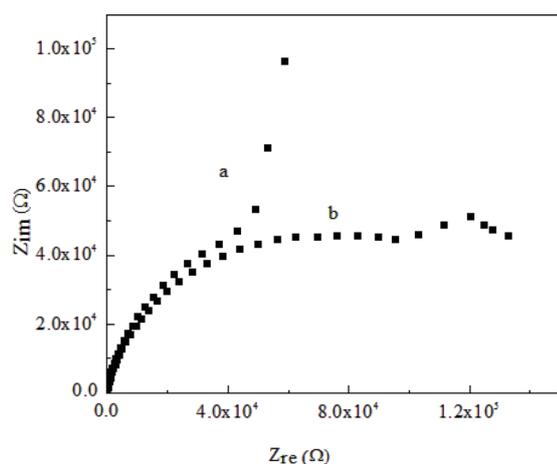


Fig. 2. Impedance plane plots for potassium ferricyanide on (a) β -CD/GCE and (b) GCE.

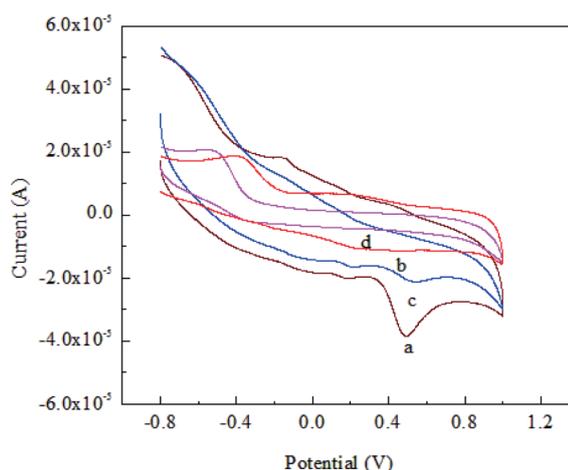


Fig. 3. (Color online) Cyclic voltammograms of β -CD/GCE (a, b) and GCE (c, d) in the HAc-NH₄Ac solution with $1.03 \times 10^{-3} \text{ mol}\cdot\text{L}^{-1}$ 1-naphthol (a, c) and the blank solution (b, d).

3.3 Optimization of determination conditions

3.3.1 Effect of solution pH

With a scan rate of 0.12 V/s, we carried out CV analysis on the β -CD/GCE in the HAc-NH₄Ac buffer solution with $1.03 \times 10^{-3} \text{ mol}\cdot\text{L}^{-1}$ 1-naphthol at different pH values of 4.00, 5.55, 7.50, 8.52, and 10.03. The cyclic voltammograms are presented in Fig. 4. The plot of E_p vs pH is shown in the inset of Fig. 4. A marked electrocatalytic action of 1-naphthol on the β -CD/GCE is observed in the electrolyte at any pH. E_p and I_p are affected by the pH of the electrolyte. When the pH is 4.00, the peak shape is imperfect and the peak current is low, preventing the quantitative analysis of 1-naphthol. When the pH is above 8.52, 1-naphthol decomposes, resulting in a large measurement error. I_p is maximal and the electrocatalytic action is the most marked when the HAc-NH₄Ac buffer solution of pH 5.55 is used in the CV analysis. The pH dependence of E_p obeys the equation $E_p = -0.06128 \text{ pH} + 0.86273$ ($R = 0.9985$). The slope of 61.28 mV/pH shows that equal numbers of protons and electrons were involved in the oxidation of 1-naphthol.⁽²³⁾

3.3.2 Effect of scan rate

Figure 5 shows the plot of I_p and scan rate, and the cyclic voltammograms of $1.03 \times 10^{-3} \text{ mol}\cdot\text{L}^{-1}$ 1-naphthol on the β -CD/GCE in HAc-NH₄Ac buffer solution of pH 5.55 with different scan rates (0.05, 0.10, 0.15, 0.20, 0.25, and 0.30 V/s) are also shown in the inset. It is found from Fig. 5 that the oxidation peak current of 1-naphthol increased with the scan rate, which obeys the linear equation $I_p \text{ (A)} = -7.714 \times 10^{-5} v \text{ (V s}^{-1}\text{)} + 4.257 \times 10^{-7}$, $R = -0.9931$.

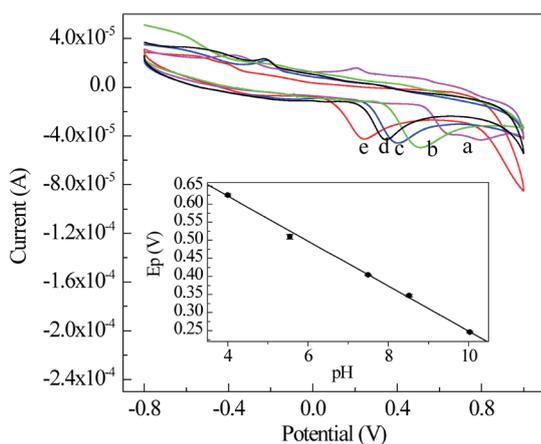


Fig. 4. (Color online) Cyclic voltammograms of $1.03 \times 10^{-3} \text{ mol}\cdot\text{L}^{-1}$ 1-naphthol on the β -CD/GCE in HAc-NH₄Ac buffer solution with various pH values: (a) 4.00, (b) 5.55, (c) 7.50, (d) 8.52, and (e) 10.03. The inset shows the plot of E_p vs pH.

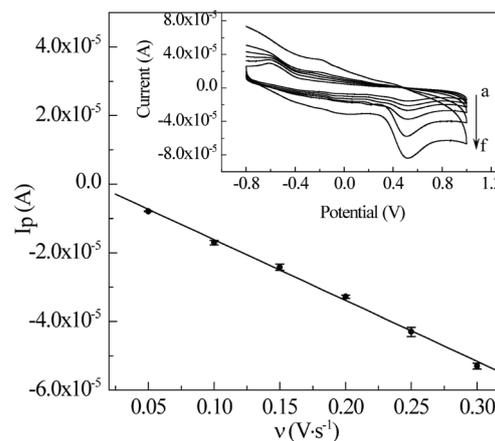


Fig. 5. Relationship between peak current and scan rate. The inset shows cyclic voltammograms of $1.03 \times 10^{-3} \text{ mol}\cdot\text{L}^{-1}$ 1-naphthol on β -CD/GCE in HAc-NH₄Ac buffer solution of pH 5.55 with different scan rates: (a) 0.05, (b) 0.10, (c) 0.15, (d) 0.20, (e) 0.25, and (f) 0.30 V s⁻¹.

This result shows that the electrocatalytic process was an irreversible process controlled by adsorption. When the scan rate reached 0.30 V/s, the peak shape changed and the background current became higher, which was not suitable for the determination of peak current. Therefore, the scan rate of 0.25 V/s was used in the CV analysis.

3.4 Linear relationship and detection limit, reproducibility, and stability

Figure 6 shows the plot of I_p and 1-naphthol concentration, and the inset shows the cyclic voltammograms of 1-naphthol on the β -CD/GCE in HAc-NH₄Ac buffer solution in the concentration range of 2.05×10^{-3} – 1.03×10^{-6} mol·L⁻¹. The linearity equation was defined as I_p (A) = $-0.0143c$ (mol·L⁻¹) – 4.347×10^{-6} , $R = 0.9979$, and the detection limit was 3.06×10^{-7} mol·L⁻¹.

Under optimized test conditions, the cyclic voltammograms of 1.03×10^{-3} mol·L⁻¹ 1-naphthol on the β -CD/GCE were recorded every day for a week. After testing, the β -CD/GCE was rinsed and preserved in HAc-NH₄Ac buffer solution of pH 5.55. Three weeks later, the peak current remained roughly stable, indicating that the β -CD/GCE has good stability. The results are shown in Table 1. Under the same experimental conditions, a relative standard deviation of 1.35% was obtained for ten repetitions of measuring 1.03×10^{-3} mol·L⁻¹ 1-naphthol on the β -CD/GCE. Therefore, the β -CD/GCE also has good reproducibility.

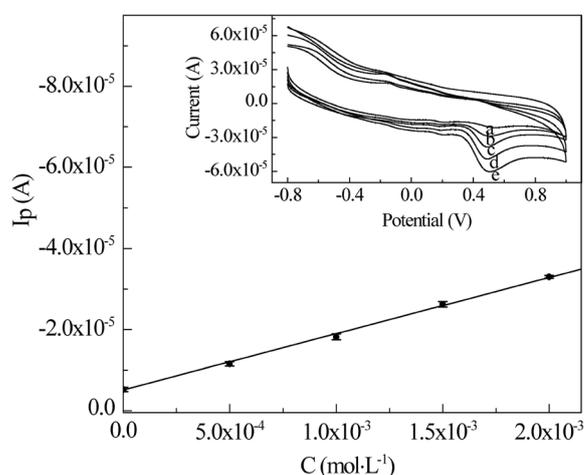


Fig. 6. Relationship between peak current and 1-naphthol concentration. The inset shows cyclic voltammograms for different 1-naphthol concentrations: (a) 1.03×10^{-6} , (b) 5.1×10^{-4} , (c) 1.03×10^{-3} , (d) 1.52×10^{-3} , and (e) 2.05×10^{-3} mol·L⁻¹.

Table 1
Results of long time stability.

Days	1	2	3	4	5	6	7	21
I_p ($\times 10^{-5}$ A)	-1.815	-1.822	-1.815	-1.811	-1.816	-1.813	-1.801	-1.779

3.5 Interference study

Under the same experimental conditions as above and within the error allowed, the factors affecting the determination of $1.03 \times 10^{-3} \text{ mol}\cdot\text{L}^{-1}$ 1-naphthol were studied. The results indicate that the 500-fold higher concentrations of Al^{3+} , Zn^{2+} , Ca^{2+} , Mg^{2+} , SO_4^{2-} , CO_3^{2-} , NO_3^- , NH_4^+ , and Na^+ , the 100-fold higher concentrations of p-nitrophenol and p-aminophenol, and the 10-fold higher concentrations of hydroquinone, pyrocatechol, phenol, and bisphenol A did not affect the determination of 1-naphthol. However, a 50-fold higher concentration of 2-naphthol interfered with the determination. The results are shown in Table 2.

4. Standard Addition Analysis of Samples

A standard solution of $1.52 \times 10^{-3} \text{ mol}\cdot\text{L}^{-1}$ 1-naphthol was prepared in pH 5.55 HAc-NH₄Ac buffer solution. Then, three samples were obtained by transferring 2.5, 5.0, and 10.0 mL of $1.52 \times 10^{-3} \text{ mol}\cdot\text{L}^{-1}$ 1-naphthol standard solution to a 50 mL volumetric flask, diluting with pH 5.55 HAc-NH₄Ac buffer solution to make 50 mL volume, and mixing. The samples were labeled from 1 to 3, respectively. The standard addition analysis of the samples was carried out by CV with scanning from -0.8 to 0.9 V at a scan rate of 0.12 V s^{-1} . The recovery rate was from 97.3 to 103%. The results are shown in Table 3.

Table 2
Interference study.

Interfering ions	Concentration	Peak current ($\times 10^{-5} \text{ A}$)
Nothing	—	-1.815
Al^{3+}	500 C_0	-1.817
Zn^{2+}	500 C_0	-1.816
Ca^{2+}	500 C_0	-1.815
Mg^{2+}	500 C_0	-1.812
SO_4^{2-}	500 C_0	-1.813
CO_3^{2-}	500 C_0	-1.817
NO_3^-	500 C_0	-1.818
NH_4^+	500 C_0	-1.810
Na^+	500 C_0	-1.814
p-nitrophenol	100 C_0	-1.815
p-aminophenol	100 C_0	-1.817
hydroquinone	10 C_0	-1.813
pyrocatechol	10 C_0	-1.813
phenol	10 C_0	-1.811
Bisphenol A	10 C_0	-1.815
2-naphthol	50 C_0	—

Table 3
Results of standard addition analysis and recovery test.

Sample	Original ($\text{mol}\cdot\text{L}^{-1}$)	Added ($\text{mol}\cdot\text{L}^{-1}$)	Measured ($\text{mol}\cdot\text{L}^{-1}$)	Recovery (%)
1	7.50×10^{-5}	1.52×10^{-4}	2.25×10^{-4}	97.3
2	1.52×10^{-4}	1.52×10^{-4}	3.09×10^{-4}	103
3	3.02×10^{-4}	1.52×10^{-4}	4.49×10^{-4}	98.3

5. Conclusions

A GCE modified with a poly β -CD film was prepared by electrochemical polymerization. The sensor was developed for use in a new test system for the measurement of the 1-naphthol concentration. The peak current of CV increased linearly with the 1-naphthol concentration in the range of 2.05×10^{-3} – 1.07×10^{-6} mol·L⁻¹, and its detection limit was 3.06×10^{-7} mol·L⁻¹ (3SNR). This method may also hold promise for potential applications in environmental analysis and measurement.

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