

# Determination of 1-Naphthol Concentration on Electrode Modified with Electrochemically Polymerized $\beta$ -Cyclodextrin Film

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A glassy carbon electrode (GCE) modified with poly  $\beta$ -cyclodextrin ( $\beta$ -CD) film ( $\beta$ -CD/GCE) was prepared. The electrochemical properties of the  $\beta$ -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-NH<sub>4</sub>Ac solution (pH 5.55), the electrocatalysis of 1-naphthol is very clear on the polymerized  $\beta$ -CD/GCE. The peak current increased linearly with the concentration of 1-naphthol in the range of  $2.05 \times 10^{-3}$ – $1.07 \times 10^{-6}$  mol·L<sup>-1</sup>, and the detection limit was  $3.06 \times 10^{-7}$  mol·L<sup>-1</sup> 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.<sup>(1–3)</sup> Currently, the common methods of determining the concentration of 1-naphthol include high-performance liquid chromatography (HPLC),<sup>(4–6)</sup> the use of quartz crystal microbalance (QCM),<sup>(7)</sup> immune cytochemistry,<sup>(8)</sup> fluorescence methods,<sup>(9–14)</sup> and synchronous-derivative phosphorimetric determination.<sup>(15)</sup> 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.  $\beta$ -cyclodextrin ( $\beta$ -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.<sup>(16)</sup> 1-Naphthol molecules entering into the cavity of  $\beta$ -CD tend to accumulate on one side, which enhances the nonbonding forces between 1-naphthol and  $\beta$ -CD, resulting in an enhancement of the electrochemical response of 1-naphthol on a glassy carbon electrode modified with poly  $\beta$ -CD film ( $\beta$ -CD/GCE). In addition,  $\beta$ -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<sup>(17,18)</sup> and a poly(methyl red)-modified electrode.<sup>(19)</sup> We have also conducted some research on  $\beta$ -CD-film-modified electrodes by electrochemical polymerization.<sup>(20)</sup> Using the results of previous work, we developed a new test system using the  $\beta$ -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.<sup>(21,22)</sup>

## 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.  $\beta$ -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 $\beta$ -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  $0.10 \text{ V/s}$  until the peak current became stable, thus attaining an activation electrode. Next, the activation electrode was put in a saturated solution of  $\beta$ -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  $0.05 \text{ V/s}$  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  $\beta$ -CD/GCE.

### 2.3 Analytical procedure

A conventional three-electrode system, including a  $\beta$ -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 $\beta$ -CD/GCE electrode

In  $1.01 \times 10^{-3} \text{ mol}\cdot\text{L}^{-1}$  potassium ferricyanide solution containing  $0.1 \text{ mol}\cdot\text{L}^{-1}$  potassium chloride, the  $\beta$ -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  $0.10 \text{ V/s}$ . 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  $\beta$ -CD/GCE. The peak potentials are  $0.015$  and  $0.268$  V. These results indicate that the  $\beta$ -CD/GCE shows good electrocatalytic action for potassium ferricyanide, but a bare GCE does not. The polymerized  $\beta$ -CD/GCE was successfully prepared by electrochemical polymerization, and the electrocatalytic property of this modified GCE showed a significant change.

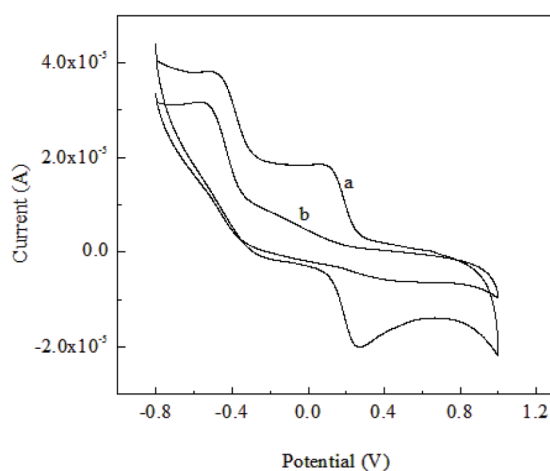


Fig. 1. Cyclic voltammograms for (a)  $\beta$ -CD/GCE and (b) GCE in  $\text{K}_3[\text{Fe}(\text{CN})_6]$  solution.

Typical complex impedance plots for the  $\beta$ -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  $\beta$ -CD/GCE. These results show that poly-( $\beta$ -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  $\beta$ -CD/GCE, the peak current of potassium ferricyanide markedly increases and its impedance decreases. These results show that the poly-( $\beta$ -CD) participates in the electron transfer process on the electrode surface and enhances the electron conductivity.

### 3.2 Electrochemical behavior of 1-naphthol on $\beta$ -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  $\beta$ -CD/GCE (curves a and b) and the bare GCE (curves c and d) in the pH 5.55 HAc-NH<sub>4</sub>Ac 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  $\beta$ -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  $\beta$ -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  $\beta$ -CD on the GCE greatly improved the electrochemical reactivity of 1-naphthol and markedly enhanced the electrochemical response of 1-naphthol on the  $\beta$ -CD/GCE.

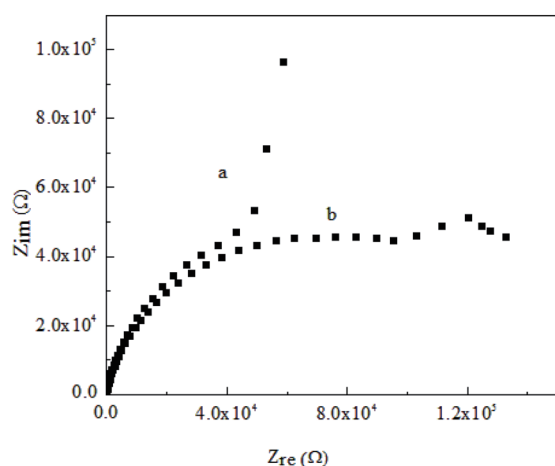


Fig. 2. Impedance plane plots for potassium ferricyanide on (a)  $\beta$ -CD/GCE and (b) GCE.

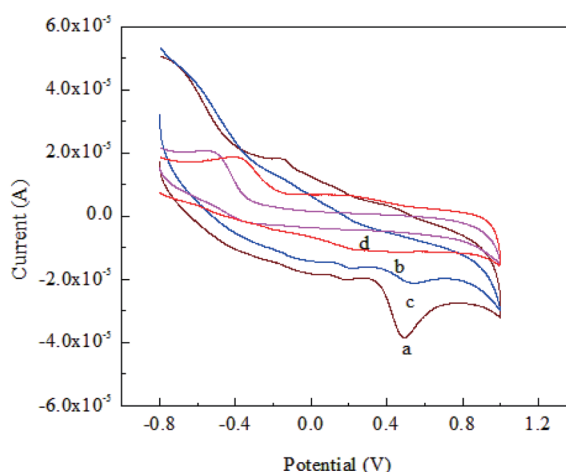


Fig. 3. (Color online) Cyclic voltammograms of  $\beta$ -CD/GCE (a, b) and GCE (c, d) in the HAc-NH<sub>4</sub>Ac solution with  $1.03 \times 10^{-3} \text{ mol}\cdot\text{L}^{-1}$  1-naphthol (a, c) and the blank solution (b, d).

### 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  $\beta$ -CD/GCE in the HAc-NH<sub>4</sub>Ac 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  $\beta$ -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-NH<sub>4</sub>Ac 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.<sup>(23)</sup>

#### 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  $\beta$ -CD/GCE in HAc-NH<sub>4</sub>Ac 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  $I_p \text{ (A)} = -7.714 \times 10^{-5} v \text{ (V s}^{-1}\text{)} + 4.257 \times 10^{-7}$ ,  $R = -0.9931$ .

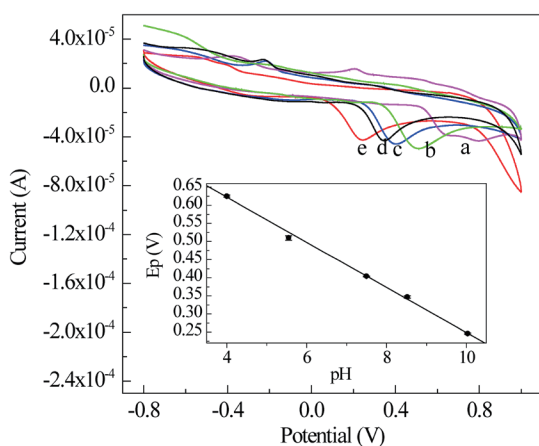


Fig. 4. (Color online) Cyclic voltammograms of  $1.03 \times 10^{-3} \text{ mol}\cdot\text{L}^{-1}$  1-naphthol on the  $\beta$ -CD/GCE in HAc-NH<sub>4</sub>Ac buffer solution with various pH values: (a) 4.00, (b) 5.55, (c) 7.50, (d) 8.52, and (e) 10.03. The inset shows the plot of  $E_p$  vs pH.

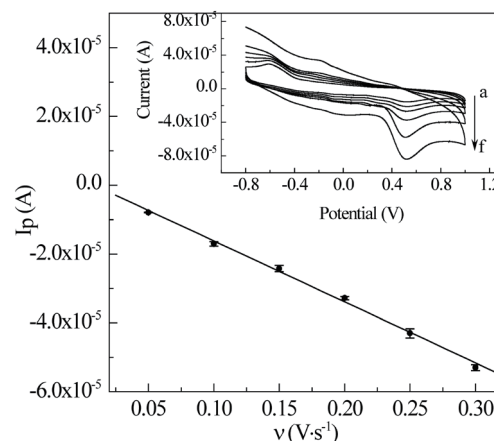


Fig. 5. Relationship between peak current and scan rate. The inset shows cyclic voltammograms of  $1.03 \times 10^{-3} \text{ mol}\cdot\text{L}^{-1}$  1-naphthol on  $\beta$ -CD/GCE in HAc-NH<sub>4</sub>Ac buffer solution of pH 5.55 with different scan rates: (a) 0.05, (b) 0.10, (c) 0.15, (d) 0.20, (e) 0.25, and (f) 0.30 V s<sup>-1</sup>.

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  $\beta$ -CD/GCE in HAc-NH<sub>4</sub>Ac buffer solution in the concentration range of  $2.05 \times 10^{-3}$ – $1.03 \times 10^{-6}$  mol·L<sup>-1</sup>. The linearity equation was defined as  $I_p$  (A) =  $-0.0143c$  (mol·L<sup>-1</sup>) –  $4.347 \times 10^{-6}$ ,  $R = 0.9979$ , and the detection limit was  $3.06 \times 10^{-7}$  mol·L<sup>-1</sup>.

Under optimized test conditions, the cyclic voltammograms of  $1.03 \times 10^{-3}$  mol·L<sup>-1</sup> 1-naphthol on the  $\beta$ -CD/GCE were recorded every day for a week. After testing, the  $\beta$ -CD/GCE was rinsed and preserved in HAc-NH<sub>4</sub>Ac buffer solution of pH 5.55. Three weeks later, the peak current remained roughly stable, indicating that the  $\beta$ -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  $1.03 \times 10^{-3}$  mol·L<sup>-1</sup> 1-naphthol on the  $\beta$ -CD/GCE. Therefore, the  $\beta$ -CD/GCE also has good reproducibility.

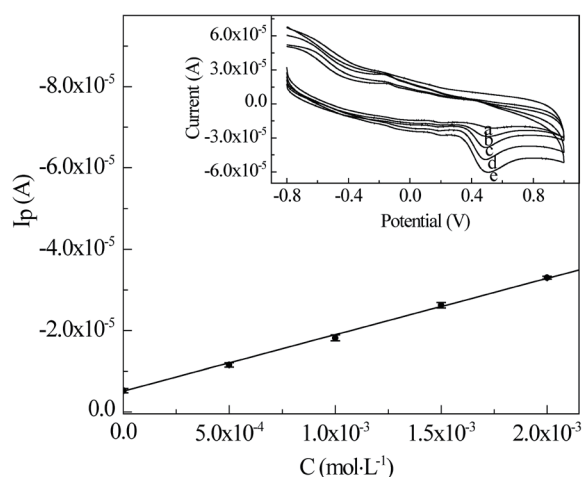


Fig. 6. Relationship between peak current and 1-naphthol concentration. The inset shows cyclic voltammograms for different 1-naphthol concentrations: (a)  $1.03 \times 10^{-6}$ , (b)  $5.1 \times 10^{-4}$ , (c)  $1.03 \times 10^{-3}$ , (d)  $1.52 \times 10^{-3}$ , and (e)  $2.05 \times 10^{-3}$  mol·L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

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  $\text{Al}^{3+}$ ,  $\text{Zn}^{2+}$ ,  $\text{Ca}^{2+}$ ,  $\text{Mg}^{2+}$ ,  $\text{SO}_4^{2-}$ ,  $\text{CO}_3^{2-}$ ,  $\text{NO}_3^-$ ,  $\text{NH}_4^+$ , and  $\text{Na}^+$ , 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-NH<sub>4</sub>Ac 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-NH<sub>4</sub>Ac 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  $0.9 \text{ V}$  at a scan rate of  $0.12 \text{ V s}^{-1}$ . 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
$\text{Al}^{3+}$	500 $C_0$	-1.817
$\text{Zn}^{2+}$	500 $C_0$	-1.816
$\text{Ca}^{2+}$	500 $C_0$	-1.815
$\text{Mg}^{2+}$	500 $C_0$	-1.812
$\text{SO}_4^{2-}$	500 $C_0$	-1.813
$\text{CO}_3^{2-}$	500 $C_0$	-1.817
$\text{NO}_3^-$	500 $C_0$	-1.818
$\text{NH}_4^+$	500 $C_0$	-1.810
$\text{Na}^+$	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	$7.50 \times 10^{-5}$	$1.52 \times 10^{-4}$	$2.25 \times 10^{-4}$	97.3
2	$1.52 \times 10^{-4}$	$1.52 \times 10^{-4}$	$3.09 \times 10^{-4}$	103
3	$3.02 \times 10^{-4}$	$1.52 \times 10^{-4}$	$4.49 \times 10^{-4}$	98.3

## 5. Conclusions

A GCE modified with a poly  $\beta$ -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  $2.05 \times 10^{-3}$ – $1.07 \times 10^{-6}$  mol·L<sup>-1</sup>, and its detection limit was  $3.06 \times 10^{-7}$  mol·L<sup>-1</sup> (3SNR). This method may also hold promise for potential applications in environmental analysis and measurement.

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