



Article

# Preparation of ZnO-Loaded Lignin-Based Carbon Fiber for the Electrocatalytic Oxidation of Hydroquinone

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**Abstract:** To improve the hydroquinone (HQ) determination limit in wastewater and contribute to the comprehensive utilization of lignin, a zinc oxide-loaded lignin-based carbon fiber (ZCF) was prepared by a combination of electrospinning and thermal treatment processes, and was applied in electrocatalytic oxidation of HQ using cyclic voltammetry (CV). The characterization of composites was conducted by X-ray diffraction (XRD), scanning electron microscopy (SEM), and energy dispersive spectrometer (EDS). The CV curves demonstrate that a ZCF-modified electrode can efficiently enhance the electrochemical signal and provide a fast response to HQ with a linear range from  $1 \times 10^{-6}$  to  $5 \times 10^{-3}$  mol/L and a determination limit of  $2.5 \times 10^{-7}$  M. Compared with modification materials reported in other literature, it performs excellent. In addition, the effect of the scan rate and the stability of modified materials were also investigated to illustrate the electrochemical behavior on HQ determination. Under optimum conditions, the ZCF-modified electrode was also used for the simultaneous determination of HQ, catechol (CC), and resorcinol (RS), which could well separate the oxidation peaks of the three isomers.

**Keywords:** lignin; ZnO; carbon fiber; hydroquinone; electrocatalytic oxidation

#### 1. Introduction

Due to the high toxicity and low degradability of hydroquinone (HQ) in cosmetic materials, dyes, pesticides, and the environment, the determination sensitivity of HQ is of great significance in the field of environmental contaminant analysis. Developing a suitable and efficient analysis method has attracted the interest of many researchers in recent years [1]. So far, methods such as high-performance liquid chromatography (HPLC) [2], pH-based flow injection analysis [3], and synchronous fluorescence [4] have been employed for the determination of hydroquinone. However, the methods above are limited in many applications due to their high cost and complex analysis procedures. Over the past few years, the electrochemical method, for its high sensitivity and relative simplicity in the determination of HQ, has been a hot topic. Similarly, the modified materials for electrodes, a key part of the electrochemical method, have also received much attention in the determination of HQ. Wei et al. and Hathoot [5,6] reported the achievements of electrocatalytic oxidation of HQ at poly 1,8-diaminonaphtalene derivatives and multiwall nanotube composite-modified electrodes. Guo [7] and his coworkers researched the simultaneous determination of HQ and catechol (CC) with an electrode modified by pyridinic nitrogen-doped grapheme, and the results demonstrated that this modified material has a good stability. Another researcher fabricated a composite that combines a three-dimensional glassy carbon electrode (GCE) with one-dimensional

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CNTs [8] and was applied to detect CC and HQ. From their study, a determination limit (S/N = 3) of  $1 \times 10^{-7}$  M was obtained. Besides, Qi et al. [9] modified a glassy carbon electrode by multiwall carbon, and this new modified electrode shows a better determination limit with an HQ concentration of about 1  $\mu$ M.

Compared with those modified materials mentioned above, the unique structure, good electrical conductivity, mechanical strength, and low cost make carbon materials excellent materials in many fields including adsorption, catalysis, and electroanalysis. Liu et al. [10], Qi et al. [9], Wei et al. [5], and Alshahrani et al. [11] have researched the application of carbon nanotubes (CNTs) in electrocatalytic oxidation of HQ as modified material and demonstrated its superiority. Carbon fiber, which has the same advantages as carbon nanotubes, has already been used to modify electrodes. Compared with carbon nanotubes, carbon fibers are beneficial to the electron transfer because of its textural property with more edges and irregular shapes [12–14]. Recent reports [15–18] have also pointed out that the carbon fiber has a good prospect in improving the electron transfer process, and can be used for electrode modification. However, the hydrophobicity of commercial carbon fiber leads to a significant obstacle for the diffusion of electrons from the electrolyte to the electrode, resulting in difficulty in the further application of carbon fiber. It is well known that the metal oxide particle has been used extensively in electrochemical research for its electrical conductivity and excellent physicochemical properties. Huo et al. [19] reported the application of a gold particle-modified electrode for the determination of ascorbic acid (AA) and achieved remarkable results. Electrodes modified with iron [10], zinc [20], aluminum [21], cobalt [22], palladium [23], and their oxides have also been prepared and applied for electrocatalytic oxidation of HQ by many researchers. Therefore, the functional modification of carbon fiber on the preparation process, performed by adding metal salt to an electrospinning solution, is considered in this study.

In order to obtain the apparent peak currents and well-separated peak potentials, researchers have combined carbon fiber and metal oxide particles and thus have fabricated a new composite for electrocatalytic oxidation of HQ [23]. In this study, a composite fabricated by combination of carbon fiber and zinc oxide was used for the electrocatalytic oxidation of HQ. The difference between this and others is that the lignin as the carbon source was used to prepare the carbon fiber. Moreover, the metal oxide will be fixed on the carbon fiber in the heat treatment process, resisting the intrinsic tendency of the metal oxide to bundle and aggregate in aqueous media. Considering the comprehensive utilization of lignin in biomass waste, this paper describes the ZnO-loaded carbon fiber prepared by a combination of electrospinning lignin and thermal treatment processes. The electrocatalytic performance of the electrode modified with a zinc oxide-loaded lignin-based carbon fiber (ZCF) on HQ was studied using the cyclic voltammogram (CV) method, and the influences of the hydroquinone concentration, scan rates, and the coexistence of its dihydroxybenzene isomers catechol (CC) and resorcinol (RS) were investigated at the zinc oxide-loaded carbon fiber-modified electrode.

## 2. Results and Discussion

# 2.1. The XRD Pattern of ZCF

In the process of preparation of metal-based carbon fiber, the metal salt was directly added into the spinning solution; then, the fiber obtained from the electrospinning process was treated with pre-oxidation and carbonization processes. The presence of oxygen on the surface of the carbon fiber makes a uniformly dispersed metal oxide formed at the heat treatment process. X-ray diffraction was performed on the characterization of the zinc phase, and the results are shown in Figure 1. It can be seen that there are obvious diffraction peaks at 20 of  $31.6^{\circ}$  and  $56.439^{\circ}$ , suggestive of the peak of ZnO. In addition, the diffraction peaks at  $27.316^{\circ}$  and  $66.160^{\circ}$  are attributed to the pattern of carbon. The synergistic effect of carbon material and metal oxide on electrocatalytic oxidation of HQ has been evidenced by other researchers [24–26].

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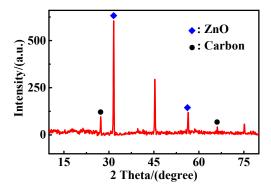


Figure 1. The XRD pattern of the zinc oxide-loaded lignin-based carbon fiber (ZCF).

## 2.2. The Surface Morphology of ZCF and CF

To illustrate the effect of textural characteristic of samples on the electrocatalytic oxidation of HQ, the ZCF and CF were characterized by BET, and the results shown in Table 1 illustrate that the sample CF affords a remarkable surface area and pore volume compared with the sample ZCF. In general, a typical adsorption-controlled process of an electron transport reaction in aqueous media is affected by the surface area of the modified electrode, where the higher the surface area is, the easier the diffusion of the electrolyte to the surface of the electrode is [27]. However, the electrocatalytic data indicate that the catalytic property of ZCF is better than CF, with good reversibility and a higher peak current on the electrocatalytic oxidation of HQ. This may be due to the formation of zinc oxide in the thermal process. Therefore, Figures 2 and 3 give the surface information of the ZCF and CF, as well as the actual content of each element on the surface of ZCF, obtained from SEM and EDS analyses.

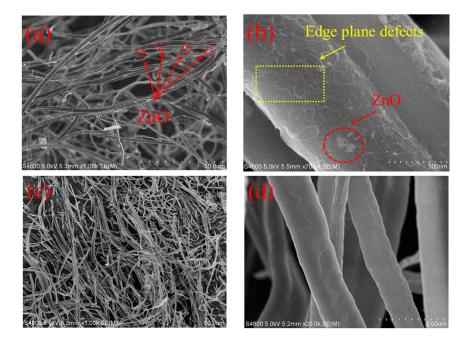
Table 1. Textural characteristic of ZCF and CF.

Sample	ZCF	CF
$S_{BET}$ (m <sup>2</sup> /g)	486.02	554.90
$V_t (cm^3/g)$	0.222	0.363

 $S_{BET}$ : Brunauer–Emmett–Teller surface area;  $V_t$ : total pore volume.

Figure 2a,b are the surface morphologies of ZCF, and Figure 2c,d represent the structure of CF. From Figure 2a,c, the network-like structure of ZCF and CF could be observed, which makes important contributions to the surface area that are responsible for the increase in the current of the reaction [10]. In addition, uniform zinc oxide particles with a diameter of about 50 nm can be observed in Figure 2b. Moreover, compared with Figure 2d, the apparent edge plane defects such as the surface out-of-flatness can be clearly observed in Figure 2b. The distinctive morphology of ZCF can improve the electrocatalytic properties of the electrode. Craig et al. [12] have reported that the edge plane defects of materials exhibited high electrocatalytic activities, and in their research apparent improvements with lower oxidation potential and higher peak currents were achieved. The excellent electrocatalytic properities of the lignin-based ZCF in subsequent experiments also prove the availability of this material. Moreover, Figure 3 displays the actual content of each element on the surface of ZCF, in which the content of Zn is 7.56%. In this paper, the 5 mg as-prepared sample was mixed with 5 mL of 0.5% Nafion solution and 5 mL of DMF solution to prepare the suspension, after which the working electrode with a diameter of 5 mm was modified by adding 10 μL of the suspension onto the surface of the electrode. Therefore, combined with the results of EDS, the zinc content on the electrode can be obtained by calculation—9.8 mg/m<sup>2</sup>. The results demonstrate the promoting effect of the sample ZCF on the electrocatalytic oxidation of HQ, mainly because of the uniform dispersion of zinc oxide particles on the surface of carbon fibers makes the electrocatalytically active sites on the surface of the electrodes are better exposed, and the electron transfer ability is enhanced [10,20].

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**Figure 2.** SEM image of ZCF and CF (**a**) surface morphologies of ZCF with 1 K magnification; (**b**) surface morphologies of ZCF with 70 K magnification; (**c**) surface morphologies of CF with 1 K magnification; (**d**) surface morphologies of CF with 20 K magnification.

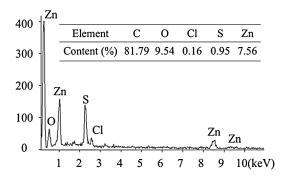
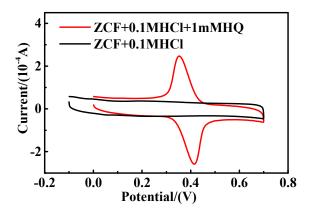


Figure 3. EDS spectrum of ZCF.

# 2.3. Electrochemical Measurements

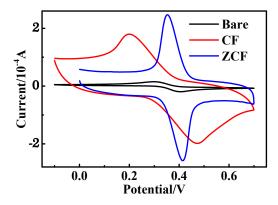
The cyclic voltammetry curves of ZCF/GCE at 0.1 mol/L hydrochloric acid electrolyte and at the 0.1 mol/L hydrochloric acid electrolyte containing 1 mM HQ are presented in Figure 4. No electrochemical reaction in 0.1 mol/L hydrochloric acid electrolyte in a voltage range from -0.1 to 0.7 V after 10 cycles of scanning was observed, demonstrating the stability of ZCF on the surface of GCE. Moreover, the cyclic voltammetry curves of the ZCF/GCE showed a pair of almost completely symmetric redox peaks in the 0.1 mol/L hydrochloric acid containing 1 mM HQ electrolyte, which is responsible for the electron transfer in the redox reaction of hydroquinone.

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**Figure 4.** The cyclic voltammetry curves of ZCF/glassy carbon electrode (GCE) in 0.1 M hydrochloric acid electrolyte with and without 1 mM hydroquinone (HQ) (scan rate was 20 mV/s).

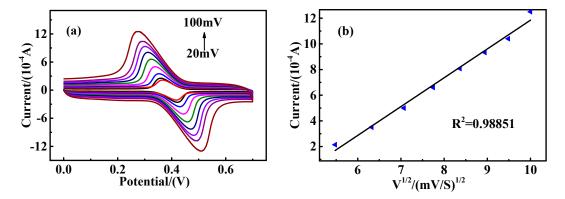
The CV responses of HQ at three different GCEs (bare GCE, ZCF/GCE, CF/GCE) were examined by cyclic voltammerey in the 0.1 M hydrochloric acid solution containing 1 mM HQ with a scan rate of 20 mV/s and the results are shown in Figure 5. A pair of redox peaks can be observed in Figure 5. It can be found that the oxidation peak current Ipa and the reduction peak current Ipc of hydroquinone on the modified electrode are significantly increased compared to the bare glassy carbon electrode. The three electrodes showed electrocatalytic activity with different degrees in this experiment, and the ZCF/GCE performed best. The oxidation peak potential and the reduction peak potential of CF are 0.519 V and 0.233 V, where the peak distance, ΔEp, is 286 mV. However, the ZCF-modified glassy carbon electrode had a relative cathodic peak potential (Epc) and anodic peak potential (Epa) at 0.353 V and 0.414 V. Obviously, the oxidation peak potential of HQ negative shifts by 67 mV, the reduction peak potential positive shifts by 105 mV, and the  $\Delta$ Ep reduces by 225 mV. In addition, as can be seen, the CF/GCE can improve the redox current but make the reversibility poor, indicating the catalysis of the formation of zinc oxide particle. Those results can be explained by the differences in SEM between ZCF and CF above. These improved properties can be ascribed to the synergy effect of carbon fiber and zinc oxide particles. It has been proved that the interaction between metal oxide and the carriers can accelerate the electron transfer rate with the addition of metal oxides [28–30]. Furthermore, the addition of metal oxides will lead to the introduction of an ionic bond, a covalent bond, and electron-hole pairs, which could increase the fixation of carbon fibers and could be the electron acceptor, thus increasing the electron transfer rate and further increasing the sensitivity and reversibility of ZCF [10]. The oxidation peak current of hydroquinone at the ZCF-modified electrode greatly increases, and the electrochemical reversibility is much improved, verifying the feasibility of this lignin-based carbon fiber as a catalytic material in detecting HQ.



**Figure 5.** The cyclic voltammetry curves of bare GCE, ZCF/GCE, and CF/GCE in 0.1 M hydrochloric acid solution containing 1 mM HQ at a scan rate of 20 mV/s.

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For the sake of obtaining the relationship between the scan rate and the peak current, the effect of scan rate on electrocatalytic HQ was investigated, and the results are shown in Figure 6. It is clear that the peak current increases with the increases in scan rate, and the oxidation peak current was linearly proportional to the square root of the scan rate ( $v^{1/2}$ ) with a correlation coefficient  $R^2$  of 0.9885, suggestive of the typical adsorption-controlled process of this modified electrode reaction [25]. Generally, the diffusion of analytes on the electrode is influenced by the surface area of modified electrode [24], and the results of BET on Table 1 prove its excellent electrocatalytic properties of ZCF on the oxidation of HQ. With the increase in the scan rate, the peak current increases, but the polarity becomes worse. Therefore, we chose 20 mV/s as the scan rate in the following experiments.

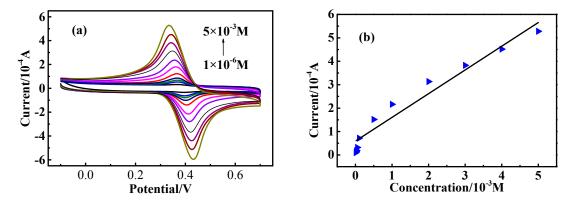


**Figure 6.** The cyclic voltammetry curve of ZCF-modified GCE in 1 mM hydroquinone solution with different scan rates (20 mV/s, 30 mV/s, 40 mV/s, 50 mV/s, 60 mV/s, 70 mV/s, 80 mV/s, 90 mV/s, and 100 mV/s) (a) the current varies with the scan rate; (b) the relation curve of oxidation peak current and  $v^{1/2}$ .

In addition to scan rates, the concentration of the hydroquinone solution is another factor that cannot be ignored in detecting HQ when use a new modified electrode. Therefore, the cyclic voltammograms of the ZCF-modified electrode in hydrochloric acid buffers containing kinds of concentrations of hydroquinone is presented in Figure 7. It can be seen that the peak current increases with an increase in hydroquinone concentration. From Figure 7, it can be noted that the oxidation peak current has a linear relationship with the hydroquinone concentration in the range of  $1 \times 10^{-6} \sim 5 \times 10^{-3}$  mol/L ( $R^2 = 0.9917$ ). There are some slight fluctuations in the linear trend of peak current with concentration, which is mainly due to the effect of the by-products produced on the electrode surface during the electrocatalytic process. The minimum determination concentration (S/N = 3) was  $2.5 \times 10^{-7}$  M, and the comparison of this result with other results can be obtained from Table 2. Apparently, the ZCF-modified electrode exhibits excellent determination limits and a wider linear range compared to others. This advantage illustrated that the ZCF-modified electrode fabricated in the present study can be used to detect HQ with high sensitivity.

For the better utilization of the lignin-based carbon fiber, it is important to study its stability and repeatability as a modified material. Therefore, the stability of the ZCF/GCE was tested in a 0.1 M hydrochloric acid solution containing 1 mM HQ at a scan rate of 20 mV/s. The results shown in Figure 8 find that the peak current of HQ only decreases a little and the reversibility keeps well after 200 instances of continuous testing. Further, considering its repeatability, six identical electrodes were chosen to be modified with ZCF and to be used in electrocatalytic HQ under the same conditions, and the results show that the peak current error can be controlled within 5.8%, further illustrating its availability.

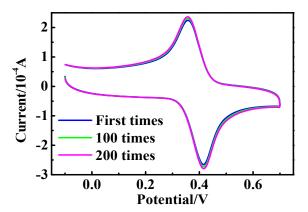
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**Figure 7.** Cyclic voltammograms of ZCF-modified GCE with the HQ concentration between  $1 \times 10^{-6} \sim 5 \times 10^{-3}$  mol/L at a scan rate of 20 mV/s (a) the current varies with the HQ concentration; (b) the relation curve of oxidation peak current and HQ concentration.

**Table 2.** Comparison of electrocatalytic oxidation performance of the HQ on different modified electrodes.

Modified Electrode	Determination Limit	Linear Range	Reference
Carbon atom wires	$6 \times 10^{-7} \text{ M}$	10~10,000 μM	[31]
Nafion/multi-walled carbon			
nanotubes/carbon dots/multi-walled	$0.7  imes 10^{-7} \mathrm{\ M}$	1~200 μM	[5]
carbon nanotubes			
Pyridinic nitrogen doped graphene	$3.8 \times 10^{-7} \mathrm{M}$	5~200 μM	[ <del>7</del> ]
Cobalt hydroxide film	$5  imes 10^{-7}  \mathrm{M}$	5~125 μM	[22]
Thermally reduced carbon nano-fragment	$4 imes10^{-7}~{ m M}$	10~120 μM	[32]
Zinc nanoparticles-loaded carbon fiber	$2.5 \times 10^{-7} \text{ M}$	1~300 μM	This work



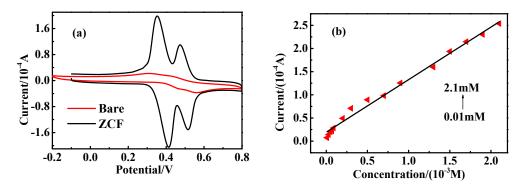
**Figure 8.** Circulation curves of ZCF/GCE in 0.1 M hydrochloric acid solution containing 1 mM HQ at a scan rate of 20 mV/s.

The research above mainly aimed at the application of the as-prepared material in the electrocatalytic oxidation of HQ. In the actual waste water, however, HQ usually does not exist alone, but coexists with its isomers catechol and resorcinol. HQ and CC are electrochemically active, their electrically active groups are very similar, and the overlap of redox peaks occurs with common electrodes, which can lead to difficulty in the quantitative determination of HQ. Consequently, the next investigation emphatically considers the influence of the coexistence of CC and RC under optimal conditions.

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#### 2.4. The Influence of the Coexistence of CC and RC

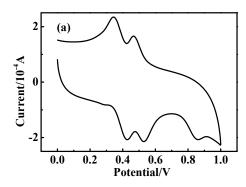
In order to observe the effect of isomers on the electrocatalytic oxidation of HQ by ZCF/GCE, the cyclic voltammograms of ZCF/GCE on electrocatalytic HQ and CC (the concentration of HQ was 1 mM, and the concentration of catechol was from 0.01 to 2.1 mM) were tested in Figure 9. It is clear that two complete reduction peaks (peak potentials are 421 mV and 317 mV) representing HQ and CC were obtained on the ZCF-modified electrode. Compared with the bare GCE, it can be seen that the coexistence of CC has no effect on the determination of HQ, and the two pairs of redox peaks can be separated well on the ZCF-modified electrode. This may be ascribed to the high specific surface area and the surface defects of carbon fiber, which is beneficial for electron transfer. At the same time, the catalysis of zinc oxide on carbon fiber surface can promote its electrocatalysis [20]. With the increase in the concentration of CC, the oxidation peak current has a linear relationship in the range of  $2 \times 10^{-6} \sim 2.1 \times 10^{-3}$  mol/L ( $R^2 = 0.9976$ ) as shown in Figure 9b, which illustrates that the sample ZCF can not only detect HQ but also detect CC within a certain concentration in waste water.

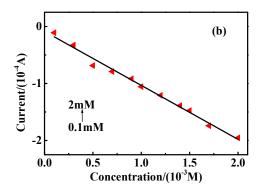


**Figure 9.** The electrocatalytic behavior of ZCF/GCE in the mixture solution of HQ and CC (a) the current in the mixture solution of HQ and CC; (b) the relation curve of oxidation peak current and CC concentration.

In addition, the cyclic voltammetry curves of three isomers—HQ, RS, and CC—were studied at the concentration of 1 mM, and the results are shown in Figure 10a. Apparently, three different oxidation peaks represent three different structures of dihydroxybenzene. Because of the unique properties of the composite, such as high surface area, high electron transfer capacity, and porosity, it makes the sample more responsive to electron transfer in the redox reaction of hydroquinone [33,34]. From the literature review, various modified electrodes were used for the simultaneous determination of those three isomers. Palladium nanoparticle-loaded carbon nanofibers [23] and multiwall carbon nanotubes [9] were fabricated to simultaneously determine these three isomers, and the oxidation peak potential of HQ and CC were separated by up to 101 mV, 105 mV, and 110 mV, respectively. Compared with them, the ZCF-modified electrode can effectively separate the oxidation peak potential of hydroquinone from catechol by up to 104 mV, and the improved current also demonstrated that the prepared materials have a strong conductivity and electron transfer capacity. The curve of reduction peak current with the increase in concentration of RS in Figure 10b shows that the oxidation peak current has a linear relationship in the range of  $1 \times 10^{-5} \sim 2 \times 10^{-3}$  mol/L ( $R^2 = 0.9985$ ).

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**Figure 10.** The electrocatalytic behavior of ZCF/GCE in the mixture solution of HQ, CC and RS (a) the current in the mixture solution of HQ, CC and RS; (b) the relation curve of reduction peak current and RS concentration.

#### 3. Materials and Methods

#### 3.1. Materials

Acetic acid, lignin,  $ZnCl_2$ , PVA solution, concentrated hydrochloric acid, N,N-dimethylformamide (DMF), and the isomers of dihydroxybenzene (HQ, CC, and RC) were from Sinopharm Chemical Reagent Co. Ltd. (Shanghai, China) and used for the preparation of lignin-based carbon fibers and the electrocatalytic oxidation of HQ. In the experiment, the 0.5% Nafion solution was obtained by diluted 5% Nafion solution (Sigma-Aldrich, Shanghai, China) in ethanol. Glassy carbon electrodes used in this paper were obtained from Huachen Electronic Instrument Co. Ltd. (Shanghai, China). The hydrochloric acid buffer solution was made up of concentrated hydrochloric acid. The chemical regents are all of analytical grade. All of the solutions needed for the experiment were prepared by ultrapure water (resistance above 18 M $\Omega$ /cm), and the ultrapure water was obtained by a Milli-Q purification system (Easypure, Branstead, NH, USA).

### 3.2. Preparation of Zinc Oxide-Loaded Carbon Fibers

Using lignin as a carbon source, the carbon fibers in this study were prepared by the following steps. First, the ZnCl<sub>2</sub> powder, lignin, 10% PVA solution, and acetic acid were mixed according to the ratio of 0.01:1:1:3 (w/w/v/v), and the mixture was heated at 80 °C in a water bath for 30 min to prepare the electrostatic spinning solution. Second, electrospinning was accomplished on the high voltage electrostatic spinning equipment with the optimized parameters (voltage: 22 kV, flow: 1 mL/h, receiving distance: 15 cm). Third, the fiber obtained from electrospinning after preoxidation (at 250 °C with a heating rate of 0.5 °C/min) and carbonization (800 °C) to construct zinc oxide-loaded carbon fibers. For comparison, the carbon fiber without metal salt was prepared by the same procedures as above but without ZnCl<sub>2</sub> in an electrostatic spinning solution. After these steps, the zinc oxide carbon fibers and carbon fibers were cut into small fragments for better dissolution in 0.5% Nafion and DMF solution. The prepared zinc oxide loaded on the carbon fiber and the carbon fibers without zinc oxide were marked as ZCF and CF, respectively.

#### 3.3. Preparation of ZCF- and CF-Modified Electrodes

Initially, the GCE (=3 mm) was polished on the gauze. After that, the surface of this GCE was polished with a 0.3  $\mu$ m and 0.05  $\mu$ m alumina slurries to mirror smoothness. Finally, the GCE was comprehensively washed with doubly distilled water and subsequently treated with ultrasonication for 20 min before the modification. Meanwhile, zinc oxide carbon fibers and the untreated carbon fibers were all cut into small pieces and suspended in the mixture solution of 0.5% Nafion and the  $N_iN$ -dimethylformamide (DMF) solution under ultrasonic treatment for 30 min. After that, 10  $\mu$ L of

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the above suspension solution were spotted onto the surface of the GCE to form both the ZCF/GCE and the CF/GCE. Finally, the modified GCEs were completely dried under room temperature.

#### 3.4. Characterization

An X-ray diffractometer (Smart Lab, Tokyo, Japan) was used to obtain the crystal structure of ZCF and CF with Cu K $\alpha$  radiation under a 0.02° scanning step at 40 KV and 40 mA. The scanning 2 $\theta$  angle was from 10° to 80°. A scanning electron microscope (S-4800, Hitachi, Japan) was used to observe the surface morphology of the materials. In addition, energy dispersive spectrometer (VarioELcube, Elementar, Shanghai, China) was used to characterize the elemental composition of ZCF. The pore texture parameters were acquired by a physical adsorption instrument (ASAP 2020, Micromeritics, Norcross, GA, USA) with nitrogen adsorption at 77 K.

#### 3.5. Electrochemical Measurements

All electrochemical experiments in this paper were performed on a CHI 660E electrochemical cell at ambient temperature. This consists of a three-electrode system containing the glassy carbon electrode, platinum wire, and a calomel electrode, which represent the working electrode, the auxiliary electrode, and the reference electrode, respectively. Electrochemical cyclic voltammograms of hydroquinone were carried out from -0.1 to 0.7 V with a scan rate of 20 mV/s.

#### 4. Conclusions

In summary, by using the electrospinning method, the lignin-based carbon fiber and ZnO-based carbon fiber were prepared and used for the determination of HQ. The results show that the ZCF/GCE can detect HQ with high sensitivity in the concentration range of  $1 \times 10^{-6} \sim 5 \times 10^{-3}$  mol/L, and its detection limit is  $2.5 \times 10^{-7}$  M. With the increase in scan rate, the oxidation peak current is linearly proportional to the square root of the scan rate ( $v^{1/2}$ ), indicating that the electrocatalytic oxidation of HQ on ZCF/GCE is controlled by diffusion. The characterization of ZCF and CF indicates that the prepared material with a mount edge planes can enhance the diffusion of HQ molecule to the interface of GCE. Besides, the stability and repeatability investigations further reveal the feasibility of lignin-based carbon fiber in HQ determination. Finally, the results gained from the influence of isomer coexistence indicate that ZCF-modified GCE exhibits activity in the simultaneous determination of the three isomers HQ, RC, and CC. It can be concluded that the zinc oxide-loaded lignin-based carbon fiber composite is promising as a sensor material for detecting HQ.

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**Author Contributions:** Min Song and Yuexing Wei conceived of and designed the experiments. Yuexing Wei, XinHong Tang, and Lei Yu performed the experiments. Yuexing Wei analyzed the data. Yuexing Wei wrote the paper.

Conflicts of Interest: The authors declare no conflict of interest.

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