



Highly sensitive determination of paeonol using porous alumina microfibers modified electrode



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ABSTRACT

A kind of porous alumina microfibers was prepared via hydrothermal reaction, and then used to modify the carbon paste electrode (CPE). Compared with the unmodified CPE, the alumina microfibers modified CPE is more active for the oxidation of paeonol, and greatly increases the oxidation signals of paeonol. The oxidation mechanism of paeonol on the alumina microfibers modified CPE was studied. The oxidation process involves one electron and one proton. Based on the remarkable signal enhancement effects of alumina microfibers, a highly sensitive electrochemical method was developed for the determination of paeonol. The linear range is from 10 to 1000 nM, and the detection limit is 6.7 nM. It was used in the analysis of traditional Chinese medicine samples. The results obtained by this method and high-performance liquid chromatography are in good agreement.

1. Introduction

Paeonol with chemical name of 2'-hydroxy-4'-methoxyacetophenone is a major active component of Moutan Cortex, a Chinese medicine prepared from the root bark of *Paeonia suffruticosa* Andrews. Paeonol has a variety of biological and pharmacological activities, and then extensively used as traditional Chinese medicine for treating various diseases. Such activities include anti-oxidation [1], anti-inflammation [2], anti-depressant [3], renoprotective effect [4], protecting against endoplasmic reticulum stress [5], and so on. Therefore, it is quite important to develop sensitive, simple and rapid analytical method for paeonol.

The commonly-used analytical methods for paeonol are chromatography and chromatography–mass spectrometry [6,7]. Compared with these methods, electrochemical determination is superior in terms of handling convenience, rapidness, analysis cost and on-site monitoring. From the molecular structure that shown in Fig. 1, we can find that paeonol should be electrochemical active because it contains phenolic hydroxy group. Until now, a few electrochemical methods have been developed for the determination of paeonol. For example, a *N*-methyl-2-pyrrolidone (NMP) exfoliated graphene nanosheets modified electrode [8], a multi-walled carbon nanotubes and ZnSe quantum dots hybrids (MWCNTs-ZnSe QDs) modified electrode [9], an acetylene black (AB) nanoparticles modified electrode [10], and a Nafion-MWCNTs composite film modified electrode [11] have been reported for the detection of

paeonol.

The main objective of this work is to develop a novel electrochemical method for the determination of paeonol utilizing the excellent properties of porous alumina microfibers. Due to many unique features such as regular fiber-like structures, numerous micropores, large surface area and strong adsorption ability, alumina microfibers have been proved to be a superior electrode material with strong signal amplification ability [12,13]. Herein, a kind of alumina microfibers in diameter of 250 nm was prepared through hydrothermal reaction using aluminum nitrate as the precursor. After that, the prepared alumina microfibers were used to modify the carbon paste electrode (CPE), constructing a novel electrochemical sensing platform for paeonol. Compared with the unmodified CPE, the alumina microfibers modified CPE greatly enhances the oxidation signals of paeonol, showing remarkable enhancement effects. Based on the signal amplification strategy of porous alumina microfibers, a highly sensitive electrochemical method with detection limit of 6.7 nM has been successfully developed for the determination of paeonol.

2. Experimental section

2.1. Reagents

All chemicals were of analytical grade and used as received. 0.01 M standard solution of paeonol (National Institute for the Control of

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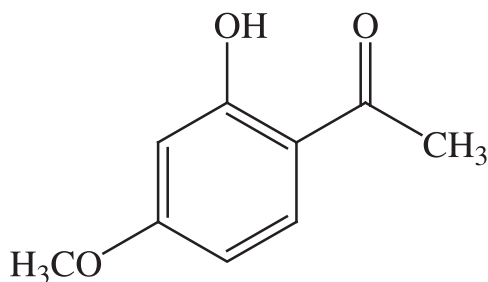


Fig. 1. Molecular structure of paeonol.

Pharmaceutical and Biological Products, Beijing, China) was prepared using ethanol, and stored at 4 °C. Urea, aluminum nitrate, graphite powder (spectral reagent) and paraffin oil were purchased from Sinopharm Chemical Reagent Company (Shanghai, China). Ultrapure water (18.2 MΩ·cm) was obtained from a Milli-Q water purification system and used throughout.

2.2. Instruments

Electrochemical measurements were performed on a CHI 830C electrochemical workstation (Chenhua Instrument, Shanghai, China). The working electrode was alumina microfibers modified CPE, the reference electrode was saturated calomel electrode (SCE), and the auxiliary electrode was Pt wire. Transmission electron microscopy (TEM) measurements were taken on a Tecnai G220 microscope (FEI Company, Netherlands). Scanning electron microscopy (SEM) images were conducted with a Quanta 200 microscope (FEI Company, Netherlands). X-ray diffraction (XRD) patterns were measured using an X'Pert PRO diffractometer (PANalytical B.V. Company, Netherlands). Nitrogen adsorption-desorption isotherms were obtained on an ASAP 3000 nitrogen adsorption apparatus.

2.3. Preparation of alumina microfibers and modified electrode

Alumina microfibers were prepared using hydrothermal reaction according to the reported method [13]. In a typical synthesis, 15.0 g of $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ was firstly dissolved in 65.0 mL of ultrapure water, and 21.6 g of urea was then added. After being totally dissolved, the mixture was transferred to a Teflon-lined stainless-steel autoclave, and reacted at 100 °C for 24 h. Finally, the solid was filtered, washed with ultrapure water, dried at 80 °C, and finally calcined at 500 °C for 2 h.

To prepare alumina microfibers modified carbon paste electrode, 0.125 g alumina microfibers and 0.875 g graphite powder were mixed in a carnelian mortar. After that, 0.25 mL paraffin oil was added, and

then mixed homogeneously. Finally, the resulting carbon paste was tightly pressed into the end cavity of electrode body (3 mm in diameter), and the electrode surface was polished on a smooth paper. The total mass of alumina microfibers and graphite powder was controlled at 1.00 g, and the modification amount of alumina microfibers was 12.5%. The unmodified CPE was prepared just using 1.00 g graphite powder and 0.25 mL paraffin oil for the controlled experiments.

2.4. Sample preparation

The cortex moutan samples were purchased from a local Pharmacy, and pulverized and then treated in the following way. The powder (about 0.2 g) was weighed, and then ultrasonicated in 20 mL ethanol for 1 h. The extraction was repeated, and the extract was diluted to 100.0 mL with ethanol for measurement.

2.5. Analytical procedure

0.1 M phosphate buffer solution with pH of 7.0 was used for the determination of paeonol. After 2-min accumulation, the differential pulse voltammograms were recorded from 0.30 to 1.10 V, and the oxidation peak currents at 0.81 V were measured for paeonol. The pulse amplitude was 50 mV, the pulse width was 40 ms, and the scan rate was 40 mV s⁻¹.

3. Results and discussion

3.1. Characterization of the prepared alumina samples

The morphology and the structure of the prepared alumina samples were characterized using SEM and TEM. From the images that was shown in Fig. 2, we clearly found that the obtained alumina samples consisted of fibers, and the diameter was about 250 nm. In addition, porous structures were clearly observed on the surface of alumina microfibers. Otherwise, the crystal structure and phase purity of the prepared alumina samples were characterized using XRD. As shown in Fig. 3, it was found that the diffraction peaks were clear and sharp, revealing good crystallinity of the prepared alumina samples. Moreover, these peaks consisted with the reported results [14], and no addition peaks were observed in the XRD pattern, indicating high purity.

In addition, the surface area and pore structures were measured using nitrogen adsorption-desorption isotherm, as shown in Fig. 4. The sample can be described to have type IV gas adsorption isotherm with type H1 hysteresis loop at high relative pressure. The specific surface area was calculated from the Brunauer-Emmett-Teller (BET) equation, and the value was 195.9 m² g⁻¹. The pore volume was 0.46 cm³ g⁻¹, and the average pore size that derived from the desorption branches

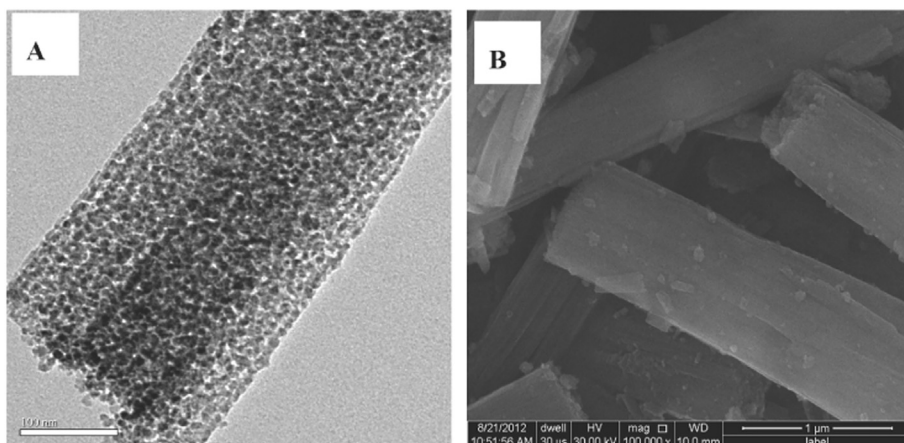


Fig. 2. TEM (A) and SEM (B) images of the prepared alumina samples.

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