



Palygorskite–expanded graphite electrodes for catalytic electro-oxidation of phenol

Yong Kong^a, Xiaohui Chen^a, Junhua Ni^a, Shiping Yao^b, Wenchang Wang^a, Zhenyu Luo^a, Zhidong Chen^{a,*}

^a Faculty of Chemistry and Chemical Engineering, Jiangsu Polytechnic University, Changzhou, 213164, China

^b ChemChina Petroleum Development Corporation, Beijing, 100080, China

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ABSTRACT

A palygorskite–expanded graphite (EG) electrode was developed for degradation of phenol. Phenol was determined by differential pulse voltammetry (DPV) with this electrode. Compared to carbon paste electrodes and unmodified EG electrodes, the novel electrode appeared to induce catalytic electro-oxidation of phenol. The dependence of signals on operating conditions was studied. The modified electrode exhibited a linear response over a wide range of phenol concentrations from 50 to 500 μM , with a detection limit of 4.5 μM ($s/n = 3$). Low cost and very simple manufacturing procedure allow for the novel electrodes to be applied as disposable devices for catalytic electro-oxidation of phenol.

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1. Introduction

Recent decades have seen a considerable effort invested in the determination of phenolic compounds in environmental, industrial, food and clinical matrices. Phenols are usually detected using various spectrophotometric and chromatographic methods (Chiricwell et al., 1975; Abdullah et al., 2006; Shi et al., 2008; Gavrilenko et al., 2006). In recent years, electrochemical sensors for organic pollutants determination have attracted a great deal of interest due to their analytical performances, such as low detection limits, wide linear response range, good stability and reproducibility (Ma et al., 2009; Yang et al., 2009; Kong et al., 2009; Liu et al., 2005).

Expanded graphite (EG) is a promising material, which has been widely used as gasket, thermal insulator, fire-resistant composite, etc. (Celzard et al., 2005; Afanasov et al., 2009; Lei et al., 2008). Recently, the application of EG as electrode material to electrochemically determine 4-chlorophenol and oxalic acid was reported (Manea et al., 2008). In our laboratory, EG was firstly used as electrode for electrochemical detection of tryptophan due to its low background current and large window for electric potential polarization (Zhao et al., 2009).

Modified electrodes offer a simple and convenient technology for investigating the interactions among various substrates and phenol and its derivatives (Nistor et al., 1999; Liu et al., 2000, 2003). Electrodes modified by porous material, such as clay minerals, have been a very active research area (Itaya et al., 1987; Scavetta et al.,

2002, 2005; Mousty, 2004). Palygorskite is a crystalline hydrated magnesium aluminum silicate with reactive surface –OH groups, and it is a natural nano-structural material. Palygorskite has been applied to modify glassy carbon electrodes for the research of electrochemical behavior of hemoglobin and cytochrome (Xu et al., 2007a,b). In particular, palygorskite has been shown to be a good adsorbent for the adsorption of phenol due to its large specific surface area (SSA) (Huang et al., 2007). Studies about the determination of phenol with palygorskite modified electrodes have not been reported up to now.

In the present work, a palygorskite modified EG electrode was prepared for the electro-oxidation of phenol, and the construction of the palygorskite–EG electrode is very simple. The electrode provides catalytic electro-oxidation of phenol after its adsorption on the modified electrode. The results reported here may be useful in the application of the palygorskite–EG electrodes for efficient detection of phenolic contaminants, which are very toxic to humans.

2. Experimental

2.1. Reagents and apparatus

Palygorskite was obtained from Jiangsu NDZ Technology Company (Changzhou, China). Natural flake graphite (99%, grain size 50 mesh) was obtained from Haida Graphite Company (Qingdao, China). Other compounds were of analytical reagent grade. All the solutions were prepared with twice distilled water.

The electrochemical experiments were performed with the CHI 410A electrochemical analyzer (CHI, USA) with a conventional three-electrode cell. Scanning electron microscope (SEM) observation of the EG and palygorskite–EG electrode was carried out with a JSM-6360LA

* Corresponding author. Tel./fax: +86 519 86330239.

E-mail address: czd_chen@yahoo.com.cn (Z. Chen).

SEM instrument equipped with a EX-54175 JMU energy dispersive X-ray spectrometer (EDS). A ASAP2010C surface area and porosimetry system was used to determine the SSA of the modified electrode by the Brunauer–Emmet–Teller (B.E.T) method.

2.2. Preparation of expanded graphite (EG)

EG was prepared from flake graphite, as reported previously (Zhao et al., 2009). Briefly, graphite, concentrated nitric acid, phosphoric acid and potassium permanganate were mixed at a mass ratio of 1:5.6:20:0.25, with stirring at 300 K for 60 min. The filtrated product was dried at 380 K for 2 h after washing thoroughly with distilled water. Expansion of the product was performed in a temperature-programmed oven at 1200 K for 60 s.

2.3. Preparation of the electrode

EG, solid paraffin and palygorskite were mixed at a mass ratio of 15:4:3 and heated to 65 °C to melt the paraffin. During the heating process, the mixture was stirred constantly to get a uniform mixing. After the mixture was cooled to room temperature, it was filled into a glass tube (5 mm inner diameter). The electrode was carefully polished and rinsed with distilled water. The electrode was then successively sonicated in alcohol and distilled water and allowed to dry at room temperature. Finally, a copper wire (2 mm diameter) was inserted into the mixture from the one end of the tube for connecting to the measuring instrument.

2.4. Electroanalytical procedures

A three-electrode set up was used: the working electrode was the palygorskite–EG electrode with an area of 19.6 mm², the auxiliary electrode was a platinum electrode, and a saturated calomel electrode (SCE) was the reference electrode. All experimental solutions were deaerated by bubbling nitrogen for at least 10 min, and a nitrogen atmosphere was kept over the solution during the measurements.

The differential pulse voltammetry (DPV) of phenol was recorded in 0.1 M NaCl solution. 1 M sodium hydroxide solution was used to adjust pH to 12.0. Pulse amplitude and potential incremental were set as 0.1 V and 0.01 V. Sampling width was set as 0.05 s. In most studies, the potential range applied in DPV was 0.2–1.2 V.

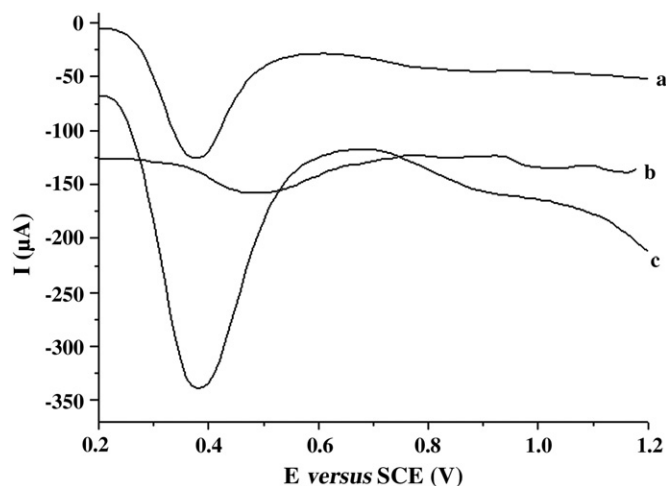


Fig. 1. Differential pulse voltammograms of EG (a), carbon paste (b) and palygorskite modified EG (c) electrodes in 0.1 M NaCl solution (pH = 12.0) with 10 mM phenol.

3. Results and discussion

3.1. Electrochemical detection of phenol

The differential pulse voltammograms (Fig. 1) obtained during oxidation of phenol showed well-defined anodic peaks, which suggested a high sensitivity for phenol.

There was a great difference between the voltammograms of the three electrodes in phenol solution. At the carbon paste electrode (Fig. 1 b), a phenol oxidation peak showed up at 490 mV and the peak current was 30 μA. At the unmodified and the palygorskite–EG electrode (Fig. 1 a, c), the oxidation peak of phenol shifted to more

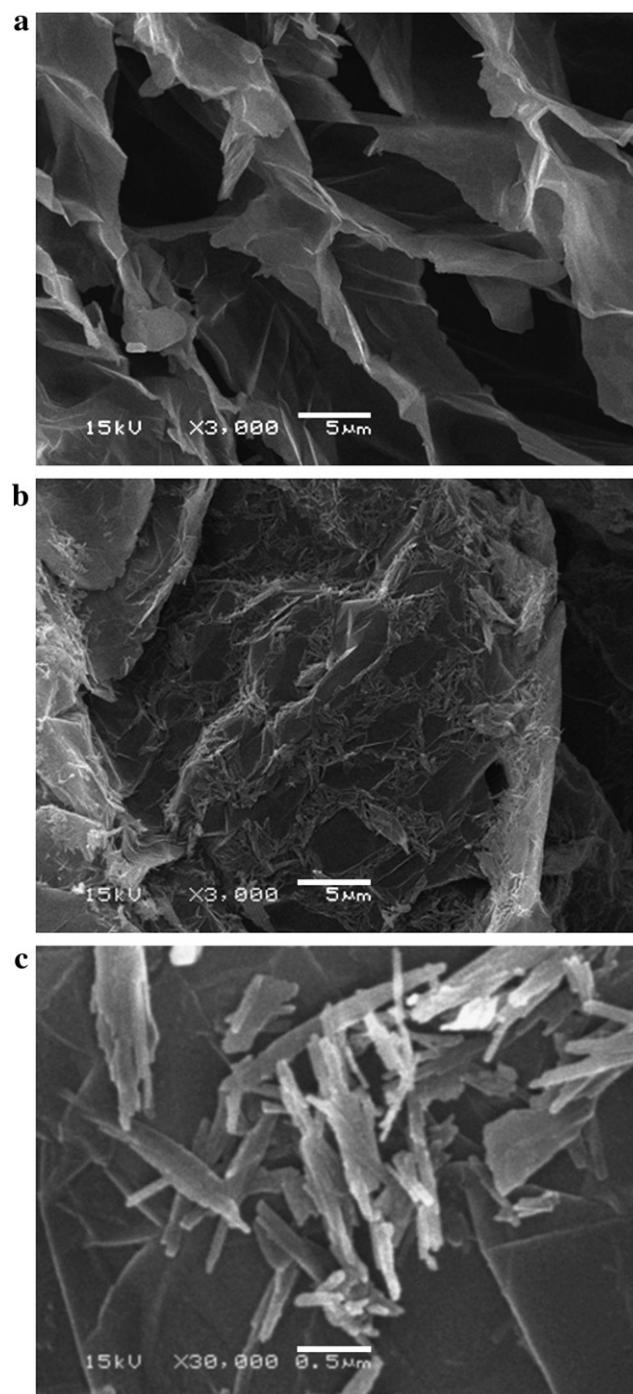


Fig. 2. SEM micrographs of pure EG (a), palygorskite modified EG electrode magnified 3000 times (b) and 30,000 times (c).

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