



Novel two-step activation of biomass-derived carbon for highly sensitive electrochemical determination of acetaminophen

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ARTICLE INFO

Article history:

Received 13 September 2017

Received in revised form

27 November 2017

Accepted 11 December 2017

Available online 12 December 2017

Keywords:

Biomass-derived carbon

Activation

Modified electrode

Electrochemical sensor

Acetaminophen

ABSTRACT

The determination of the concentration of acetaminophen (AC) in the human body is of significant importance to carefully monitor and regular drug safety and public health. However, most conventional methods are insufficient due to their time-consuming, expensive and complicated nature. In this study, we report a novel two-step activation of biomass-derived carbon for the electrochemical determination of AC. The electrode material is prepared by a two-step activation process, which involves the initial activation of kelp powder with $ZnCl_2$, followed by an activation step with KOH. The activation procedure greatly increased the overall pore volume and specific surface area. The characterizations of $ZnCl_2$ -KOH activated kelp carbon (ZKAKC) were conducted with scanning electron microscopy (SEM), transmission electron microscopy (TEM), Raman spectroscopy, Brunauer-Emmett-Teller (BET) surface area analysis, and electrochemical impedance spectroscopy (EIS). The electrochemical characterization of ZKAKC was performed with cyclic voltammetry (CV) analysis of potassium ferricyanide. The sensing ability of ZKAKC/GCE toward acetaminophen was conducted using CV analysis and differential pulse voltammetry (DPV). The modified electrode showed high sensitivity, selectivity and a good detection limit for the determination acetaminophen with the detection limit of $0.004 \mu M$. Also, the modified electrode showed good result toward acetaminophen even in the presence of ascorbic acid and dopamine with the detection limit of $0.007 \mu M$. For the evaluation of sensing ability as an actual electrochemical sensor, a real sample test was conducted. The electrochemical performance was enhanced due to the increased physical and electrochemical surface area, which occurred during the two-step activation process. This approach for producing activated carbon is crucial for future development and can be applied to different carbon source materials.

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1. Introduction

Acetaminophen (AC), also known as paracetamol, is a well-known phenolic medicine for its pain-relieving, anti-inflammatory and antipyretic effects [1]. Typically, a small dosage (below 30 mg L^{-1} , about $200 \mu M$) of AC can be beneficial. However, an overdose ($30\text{--}300 \text{ mg L}^{-1}$, about $200\text{--}2000 \mu M$) of AC can cause health problems such as kidney failure and hepatic necrosis [2,3]. According to the 2015 report from the American Association of Poison Control Center, AC ranked in the top 25 pharmaceuticals that are responsible for the largest number of fatalities in the US [4]. Consequently, monitoring the concentration of AC in the

human body became important due to the drug safety and health care of people in the society. To achieve the efficient analysis of AC, numerous determination techniques have been investigated and developed. These various methods include high-performance liquid chromatography and spectrophotometry [5,6]. However, these methods have certain disadvantages such as needing a time-consuming pretreatment of the analyte, high cost and long series of complicated operating procedures. For the fast, facile and sensitive drug monitoring, electrochemical determination techniques using various modified electrodes were investigated for its simplicity, sensitivity, reproducibility and cost efficiency [7–14]. A more efficient, effective and enhanced determination of AC is expected by using electrochemical sensing techniques with modified electrodes.

In recent years, activated carbons has received considerable attention as a new electrode material and has been investigated

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for application in secondary batteries, supercapacitors and electrochemical sensors. [15–17]. This is because activated carbons usually possess high active surface area and high conductivity, which are the key aspects of advanced electronic device applications. To achieve this, the carbon material needs to undergo an activation process which alters the material's structure by either a physical or chemical process. Therefore, finding new and more suitable activation processes for activated carbon is crucial. In the case of chemical activation, heat treatment of carbon material with strong alkali salts like NaOH and KOH [18–21] in high activation temperature is frequently used, since it is a facile approach to developing porous structures and its ability to generate abundant pores that range from nanometer to micrometer in size. Particularly for biomass-derived activated carbon, the activation method with ZnCl_2 is commonly used in order to enhance the surface area and create micropores as well as mesopores [22–26].

Unlike conventional activated carbons, biomass-derived activated carbons prepared from numerous natural materials have been investigated in recent years for electrochemical applications such as lithium-ion battery, supercapacitor and electrochemical sensor with the help of activation processes [27–35]. This approach holds great potential, since the precursors for the electrode material are normally waste products. Among the candidates for biomass-derived carbon materials, kelp (seaweed) can be a promising source material of activated carbon. Kelp is well known as natural source of glutamate and is frequently used in Eastern Asian nations as a food additive [36]. The cost efficiency of kelp is quite high since the amount of seaweed is abundant in the ocean around the world. The relatively small human consumption of kelp leaves an abundant amount of seaweed unused, which could serve as source material for activated carbon.

In this study, we report a novel two-step activation process of kelp-derived carbon for sensitive electrochemical determination of AC. To produce ZnCl_2 -KOH activated kelp carbon (ZKAKC), the initial activation of dried kelp powder was performed with ZnCl_2 , followed by activation with KOH. By adopting a two-step activation, the surface area significantly increased. The electrochemical specific surface area was examined with cyclic voltammetry (CV) of ferricyanide. The cyclic voltammetry analysis of AC using ZKAKC modified electrode showed enhanced electrochemical sensing performance. To investigate the sensitivity of the modified electrode when the AC is exposed to other organic molecules, differential pulse voltammetry (DPV) analysis was performed. The result showed a low detection limit in the presence of ascorbic acid (AA) and dopamine (DA). Finally, the activated carbon modified electrode was tested using a real sample as an actual electrochemical sensor. By applying a two-step activation, the sensitivity and selectivity of the AC highly increased. This novel approach for synthesizing activated carbon is important for future development and can possibly be applied to different carbon source materials.

2. Materials and methods

2.1. Reagents

All chemicals except dried kelp powder were analytical grade and used without further purification. Dried kelp powder was purchased from the local supermarket. Potassium hydroxide (KOH) and potassium phosphate dibasic (K_2HPO_4) were purchased from Sigma-Aldrich (USA). Citric acid and zinc chloride were purchased from Samchun Chemicals (South Korea). The electrolyte for the determination of AC was 0.1 M phosphate buffer solution (PBS, pH 7.4), which was prepared by mixing the predetermined amount of citric acid, potassium phosphate dibasic and distilled water.

2.2. Apparatus

Cyclic voltammetry (CV) and differential pulse voltammetry (DPV) were conducted by using the conventional three-electrode system by Autolab potentiostat (Metrohm, USA). Glassy carbon electrode (0.07 cm^2 in surface area, Bioanalytical Systems, Inc.) was used as working electrode. Ag/AgCl electrode (Bioanalytical Systems, Inc.) and platinum wire electrode were used as reference and counter electrodes, respectively. The produced materials were characterized by field-emission scanning electron microscope (FE-SEM, Hitachi S-4800), energy-filtering transmission telescope (EF-TEM, LIBRA 120), Raman spectroscopy (HORIBA Scientific-T64000) and electrochemical impedance spectroscopy (EIS, ZIVE SP1). The specific surface area and pore volume were analyzed via nitrogen adsorption-desorption isotherm (Micrometrics ASAP 2010).

2.3. Preparation of kelp carbon (KC)

The kelp powder (1 g) was washed with distilled water and freeze dried for 2 days. Then, the powder was carbonized in a horizontal quartz tube furnace under Ar flow at $700\text{ }^\circ\text{C}$ ($10\text{ }^\circ\text{C}/\text{min}$) for 3 h. After the treatment, the kelp carbon was washed with diluted HCl solution and distilled water, followed by drying in $60\text{ }^\circ\text{C}$ oven for 1 day. Finally, the produced kelp carbon was denoted as KC.

2.4. Preparation of ZnCl_2 activated kelp carbon (ZAKC)

The kelp powder (1 g) was washed with distilled water and freeze dried for 2 days. Then, the powder was mixed with ZnCl_2 (2 g) and added to distilled water (10 mL), followed by drying in $60\text{ }^\circ\text{C}$ oven. The mixture was heated in a horizontal quartz tube furnace under Ar flow at $700\text{ }^\circ\text{C}$ ($10\text{ }^\circ\text{C}/\text{min}$) for 3 h. After the treatment, the activated kelp carbon was washed with diluted HCl solution and distilled water, followed by drying in $60\text{ }^\circ\text{C}$ oven for 1 day. Finally, the produced kelp carbon was denoted as ZAKC.

2.5. Preparation of KOH activated kelp carbon (KAKC)

The kelp powder (1 g) was washed with distilled water and freeze dried for 2 days. Then, the powder was mixed with KOH (1 g) and added to distilled water (10 mL), followed by drying in $60\text{ }^\circ\text{C}$ oven. The mixture was heated in a horizontal quartz tube furnace under Ar flow at $800\text{ }^\circ\text{C}$ ($10\text{ }^\circ\text{C}/\text{min}$) for 1 h. After the treatment, the activated kelp carbon was washed with diluted HCl solution and distilled water, followed by drying in $60\text{ }^\circ\text{C}$ oven for 1 day. Finally, the produced kelp carbon was denoted as KAKC.

2.6. Preparation of ZnCl_2 -KOH activated kelp carbon (ZKAKC)

Dried kelp powder (1 g) was washed with distilled water several times. Then, ZnCl_2 (2 g) was added into distilled water (10 mL) and stirred using a magnetic bar for 2 h. For the first activation, the mixture was freeze dried for 2 days and heated in a horizontal quartz tube furnace under Ar flow at $700\text{ }^\circ\text{C}$ ($10\text{ }^\circ\text{C}/\text{min}$) for 3 h. This pre-activated carbon was washed with diluted HCl solution, followed by drying in $60\text{ }^\circ\text{C}$ oven for 1 day. For the second activation, the KOH was added into the pre-activated carbon with the determined weight ratio (1:1). Then the mixture was heated in a quartz tube furnace under Ar flow at $800\text{ }^\circ\text{C}$ ($10\text{ }^\circ\text{C}/\text{min}$) for 1 h. Finally, the mixture was washed with a diluted HCl solution and water, and dried in $60\text{ }^\circ\text{C}$ oven for 1 day and denoted as ZKAKC.

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