



## Original Research Paper

## Synthesis and characterization of heteroatom-enriched biochar from keratin-based and algous-based wastes

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## ARTICLE INFO

## Article history:

Received 10 July 2015

Received in revised form 26 March 2016

Accepted 24 April 2016

Available online 30 April 2016

## Keywords:

Hair

*Enteromorpha prolifera*

Biochar

Heteroatom-enriched

## ABSTRACT

In this work, human hair and *Enteromorpha prolifera* were firstly used to synthesize heteroatom-doped biochars. The effects of pyrolysis temperature and holding time on the yield and pore structure of the obtained biochars were investigated. The different pyrolysis characteristics of hair and *E. prolifera* were compared through thermogravimetric analyser under nitrogen atmosphere. The pore properties, surface morphology and surface chemical composition were studied by N<sub>2</sub> adsorption, scanning electron microscopy and X-ray photoelectron spectroscopy. The biochar from hair was typical dual O, N-doped material, which possessed 21.14 at.% of O-doped and 9.61 at.% of N-doped on the surface, while the biochar from *E. prolifera* was typical dual O, S-doped material, which possessed 30.68 at.% of O-doped and 5.18 at.% of S-doped on the surface. The present study provides a good prospect for development of heteroatom-enriched biochar materials from renewable biomass wastes.

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## 1. Introduction

Recently, a growing number of studies have demonstrated and highlighted the advantages of using biochar as energy carrier, soil amendment, precursor for the preparation of activated carbon and adsorbent [1–3]. Biochar served as a modifier or carbon sink in soil could improve soil properties, simultaneously reduce greenhouse gas emissions from decaying biomass, thereby effectively mitigating global warming. Additionally, biochar also has appreciable value for activated carbon production, which may help in relieving the production cost.

Biochar is a carbon-rich by-product generated through the pyrolysis of plant-derived biomass under low or absence of oxygen. Biochar can have fundamentally different chemical and physical properties depending on the pyrolysis conditions together with type of feedstock [4–6]. Presently, various types of ligneous and cellulosic biomasses, such as *Alternanthera philoxeroides* [7], safflower seed [8], straw [9], pinewood [10], vine shoot [11], olive [12] and sugarcane bagasse [12] have been developed as feedstock for biochar production. So far, the dominating studies on biochar were mainly focused on using it as soil or adsorbent [1–3,7]. Considering the potential application of activated carbon from biochar

in supercapacitors, the production of heteroatom-enriched biochar is highly attractive and worthwhile. A variety of methods have been developed for adding heteroatoms to the carbon surface, such as plasma, chemical treatment, oxyfluorination and chemical vapor deposition [13]. The main drawbacks of these methods were their complicated pre-treatment or post-treatment processes, which can be overcome by using heteroatom-containing raw material as precursor. Hair (HR) is a renewable, cheap, abundantly attainable biomaterial, which is primarily composed of keratin, lipids, melanin and trace elements [14,15]. *Enteromorpha prolifera* (EP) is a widely available marine macroalgae as a result of the eutrophication and sequentially forming green tides, which is made up of saccharide, fat, protein and some minerals [16]. Currently, most of them are often disposed in landfills for natural decomposition, leading to a secondary environmental problem by releasing greenhouse gases. Consequently, converting these biomass wastes into biochar may be as an effective solution for waste disposal and recycling option. The abundant oxygen, nitrogen and sulfur species nature in these two wastes endows themselves promising potential as interesting candidates for the preparation of heteroatom-enriched biochars. This proposal appears to achieve win-win through achieving low-cost precursor for synthesizing heteroatom-enriched activated carbon, while sequestering C in high-valued product.

To the best of our knowledge, there is a dearth of information on producing heteroatom-enriched biochar using keratin-based and algous-based wastes as raw material. Hence, the overarching

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objective of this study was to evaluate the feasibility of using HR and EP biomass source to produce biochar. As the pyrolysis parameters play an important role in the quality and quantity of the produced biochar [8,17]. Hence, the effects of pyrolysis temperature and holding time on the yield and pore characteristics of the obtained biochar were investigated. The detail information on BET surface area, pore volume, average pore size and surface chemical groups were also identified.

## 2. Experimental

### 2.1. Materials

The HR samples were obtained from a nearby barbershop, washed with deionized water to remove the impurities, air dried, cut into 3–5 cm pieces and stored in valve bags. The EP samples were collected from Qingdao city, Shandong Province of the middle part of China, air dried, ground to 0.125–0.75 mm fine particles and stored for further use.

### 2.2. Preparation of the biochar

The pyrolysis process was carried out by using an electrically heated chamber type electric resistance furnace equipped with a temperature controller (KSY-4D-16) [18]. The feedstock was placed into a quartz crucible and housed with a lid. Then, the crucible was placed into the center of the furnace. In each pyrolysis test, 5 g of biomass sample was heated from room temperature to the setting temperature under oxygen-limited conditions. To find out the influence of temperature, the pyrolysis was conducted at different temperature of 300 °C, 400 °C, 500 °C, 600 °C and 700 °C at a heating rate of 10 °C min<sup>-1</sup> for 60 min. To find out the influence of holding time, the pyrolysis was maintained for 30 min, 60 min, 90 min, 120 min and 150 min at a heating rate of 10 °C min<sup>-1</sup> and a designated temperature of 500 °C. By the time the furnace has cooled down to room temperature, the sample was collected, ground and sieved for the following analysis. For convenience, the biochar produced from hair and *E. proliferans* were named as HRBC-*x-y* and EPBC-*x-y*, where *x* stands for the pyrolysis temperature, and *y* refer to the holding time. For example, sample HRBC-500-60 signifies the biochar originated from the pyrolysis of hair at 500 °C for 60 min.

### 2.3. Characterizations of the biochar

The yield of biochar is expressed as the ratio of the final weight of the char to the original weight of dry feedstock. To compare the changes in weight loss of feedstock in relation to the pyrolysis conditions during the biochar generated process, the TGA-DTG analysis were recorded using a thermal analyzer (SHI-MADZU, TGA-50). To avoid the heat and mass transfer limitations, low sample masses of 5–10 mg was used [10,12]. Each run was performed using N<sub>2</sub> as carrier gas (100 mL min<sup>-1</sup>) under different conditions. The surface area, pore volume and pore size distribution of the biochar were measured by N<sub>2</sub> adsorption/desorption at -196 °C using a BK122T surface area pore analyzer (JWGB, China). The analysis of micropore was based on *t*-plot method. The analysis of mesopore and macropore was according to the BJH method. The specific surface area was determined from multi-point BET adsorption isotherm. The morphology analysis of the biochar was performed using a JEOL, JSM 7600F scanning microscope. The surface chemical property of the biochar was achieved by using an Avatar 370 Fourier transform infrared spectroscopy and an ESCALAB 250 spectrometer equipped with Mg Ka X-ray source.

## 3. Results and discussion

### 3.1. Effect of parameters on biochar yield

The yields of biochar prepared as a function of different pyrolysis temperature and holding time are presented in Fig. 1. An increase of pyrolysis temperature and holding time both resulted in a decrease in the yields of HRBC and EPBC. Furthermore, the yields of HRBC were lower than the yields of EPBC. Specifically, the yields of HRBC changed from 33.20% to 25.05% and the yields of EPBC decreased from 44.82% to 27.62% with the temperature increasing from 300 to 700 °C. By contrast, the yields of HRBC dropped from 29.8% to 24.90% and the yields of EPBC decreased from 40.75% to 36.11% while the holding time increasing from 30 min to 150 min. The decline in biochar yields was predominately attributed to the secondary decomposition of some organic materials at higher pyrolysis temperatures and longer holding time. Veksha et al. suggested that the release of volatile species with increasing temperature led to the decrease of biochar yield [19]. Al-Wabel et al. also found that the destruction of some compounds could reduce the biochar yield [4]. Similarly, the excessive holding time could lead to excessive thermal decomposition as well.

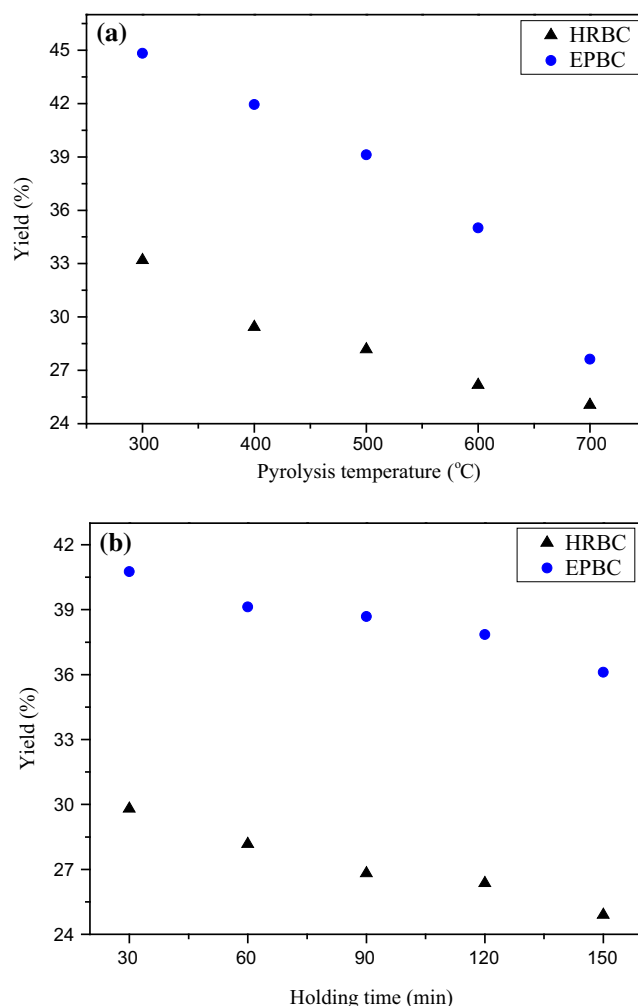


Fig. 1. Effect of (a) pyrolysis temperature and (b) holding time (pyrolysis temperature=500 °C) on the yield of the produced biochars.

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