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Facile one-step synthesis of functionalized biochar from sustainable prolifera-green-tide source for enhanced adsorption of copper ions

Yuan Gao[1,2], *Xiuzhen Zhu*[1], *Qinyan Yue*[1,*], *Baoyu Gao*[1,*]

1. *Shandong Provincial Key Laboratory of Water Pollution Control and Resource Reuse, School of Environmental Science and Engineering, Shandong University, Jinan 250100, China*

2. *Key Laboratory of Industrial Ecology and Environmental Engineering (Ministry of Education), School of Environmental Science and Technology, Dalian University of Technology, Linggong Road 2, Dalian 116024, China*

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ABSTRACT

The use of biochars formed by hydrothermal carbonization for the treatment of contaminated water has been greatly limited, due to their poorly developed porosity and low content of surface functional groups. Also, the most common modification routes inevitably require post-treatment processes, which are time-consuming and energy-wasting. Hence, the objective of this research was to produce a cost-effective biochar with improved performance for the treatment of heavy metal pollution through a facile one-step hydrothermal carbonization process coupled with ammonium phosphate, thiocarbamide, ammonium chloride or urea, without any post-treatment. The effects of various operational parameters, including type of modification reagent, time and temperature of hydrothermal treatment, and ratio of modification reagent to precursor during impregnation, on the copper ion adsorption were examined. The adsorption data fit the Langmuir adsorption isotherm model quite well. The maximum adsorption capacities (mg/g) of the biochars towards copper ions followed the order of 40-8h-1.0-APBC (95.24) > 140-8h-0-BC (12.52) > 140-8h-1.0-TUBC (12.08) > 140-8h-1.0-ACBC (7.440) > 140-8h-1.0-URBC (5.277). The results indicated that biochars modified with ammonium phosphate displayed excellent adsorption performance toward copper ions, which was 7.6-fold higher than that of the pristine biochar. EDX and FT-IR analyses before and after adsorption demonstrated that the main removal mechanism involved complexation between the phosphate groups on the surface of the modified biochars and copper ions.

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Introduction

Heavy metals in surface or groundwater effluents may lead to serious toxic effects on living species, which has become an urgent worldwide problem (Sun et al., 2017; Zhao et al., 2016; Zhang et al., 2018). A variety of techniques, including membrane separation, precipitation, adsorption, bioremediation and ion exchange, have been developed for the treatment of heavy metal-contaminated water or soil (Feizi and Jalali, 2015; Sun et al., 2016). Among them, adsorption has been one of the most successfully applied methods for removing heavy metals, due to its easy operational process and high efficiency (Uddin, 2017; Qin et al., 2017). Various sorbents, including activated carbon, zeolite, graphene, clay, ghee residue and resin, have been used

* *Corresponding authors. E-mail:* qyyue58@aliyun.com (Qinyan Yue), baoyugao_sdu@aliyun.com (Baoyu Gao).

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to remove heavy metals (Hao et al., 2017; Guo et al., 2017). However, the large-scale application of these adsorbents has been highly restricted by high manufacturing cost, resulting in an urgent need for the development of new, alternative and economical adsorbents (Liang et al., 2017; Gwenzi et al., 2017).

Biochar, a value-added product, refers to pyrogenic carbonaceous residues derived from the thermochemical decomposition of carbohydrate-rich biomass in a limited oxygen or anoxic environment (Zama et al., 2017; Yang et al., 2017). The original purpose for producing biochar was to alleviate global warming through sequestering carbon from the atmosphere into soil (Agegnehu et al., 2016; Woolf et al., 2010). Recent studies suggest that biochar can be considered as one of the most promising, emerging and cheap sorbents for environmental remediation, especially heavy metal-contaminated water (Petrovic et al., 2016; Kim et al., 2014; Khorram et al., 2016; Wang et al., 2017a, 2017b, 2017c). Theoretically, all carbon-rich materials, including plant residues, agricultural wastes, animal litters and solid wastes, can be used to produce biochars *via* slow pyrolysis, fast pyrolysis, flash pyrolysis, vacuum pyrolysis, intermediate pyrolysis or hydropyrolysis (Manyá, 2012). Currently, more and more research attention has been focused on producing biochar by hydrothermal carbonization (HTC) (Wang et al., 2017a, 2017b, 2017c; Liu and Balasubramanian, 2014; Wiedner et al., 2013). HTC, also called wet pyrolysis, has been regarded as a highly feasible, simple, low energy consumption and facile technique to transform various types of natural raw biomass into biochar (Reza et al., 2013). HTC is commonly performed by simulating the natural coalification process, with subcritical water as the reaction medium at moderate temperatures of 180–300°C and saturated pressure (Eyser et al., 2015; Chen et al., 2017). Thus, HTC is a mild route and "green" process for the preparation of biochars in contrast to other pyrolysis methods, from the viewpoint of mitigating the emission of greenhouse gases and the formation of tars. Additionally, biochars with more oxygen functional groups, including carboxyl, phenolic and lactonic groups, can be fabricated by the HTC method compared to other pyrolysis processes.

Nevertheless, it has been reported that the effective and straightforward application of HTC biochars for the treatment of contaminated water is greatly limited due to their poorly developed porosity and low content of surface functional groups. In order to improve the practical adsorption performance of biochars towards pollutants, several techniques, including steam activation, heat treatment, oxidation and grafting, have been explored to enhance the chemical reactivity and physical properties of the biochars (Liu et al., 2017; Wang et al., 2016; Yang et al., 2017; Wang et al., 2017a, 2017b, 2017c). For example, ammonium citrate was used to modify marine biomass-based biochar with amino and carboxyl groups. Wang et al. (2016) reported that the modified biochar exhibited outstanding sorption capability (362.32 mg/g) compared to the pristine biochar (170.36 mg/g) for La(III) removal. Yang et al. (2017) prepared nitrogen-functionalized biochars from wheat straw *via* a molten salt strategy, and the resultant biochar exhibited good adsorption performance toward atrazine. Wang et al. (2017a, 2017b, 2017c) found that the adsorption capacity of pristine sawdust biochar towards p-nitrophenol and Pb(II) was improved by iron and zinc doping. Furthermore, they found that the affinity between oxygen-containing hydrophilic sites and pollutants was the predominant removal mechanism. Generally, it can be found that these modification routes inevitably require a post-treatment process, which is time-consuming and energy-wasting.

Hence, in the present study, surface-modified biochar was produced for the first time from a sustainable prolifera-green-tide source through hydrothermal carbonization using ammonium phosphate, thiocarbamide, ammonium chloride and urea as activity-enhancing additives. One of the positive aspects of this study is that a facile one-step route was developed to solve the above-mentioned issues. Namely, the post-treatment process was abandoned. To the best of our knowledge, research on this aspect has not been reported. Additionally, the influences of operational parameters (additive species, impregnation ratio, reaction time and temperature) on the physicochemical properties and adsorption capability of the modified biochars were assessed. The modified biochars were characterized by nitrogen adsorption–desorption, Fourier transform infrared (FT-IR), energy dispersive X-ray spectroscopy (EDX) and Scanning Electron Microscopy (SEM). Adsorption isotherm experiments were conducted to examine and compare the adsorption properties of the pristine and modified biochars using copper ions as a model adsorbate.

1. Experimental

1.1. Chemicals and materials

Enteromorpha prolifera (EP) was gathered from the coast in Qingdao, Shandong Province. All the chemical reagents were of analytical grade, and deionized water was used during the whole experiment.

1.2. Hydrothermal carbonization of EP for the preparation of biochar

Ammonium phosphate, ammonium chloride, urea or thiourea was mixed with 20 g EP. In a typical experiment, different masses of ammonium phosphate were mixed with 20 g EP at mass ratios of 0:1, 0.5:1, 1:1 and 1.5:1. The mixture was dissolved in 120 g deionized water and impregnation was carried out for 12 hr. The mixture was transferred into a hydrothermal reactor, and heated to 140°C, 180°C and 220°C for 4 hr, 8 hr and 12 hr. After that, when the system had naturally cooled to room temperature, the products were washed with hot deionized water until the wash solution was clear. Finally, the sample was dried in an oven at 100°C and stored for further use. The biochars produced by the addition of ammonium phosphate, thiocarbamide, ammonium chloride and urea were named biochars by ammonium phosphate (APBC), biochars by thiocarbamide (TUBC), biochars by ammonium chloride (ACBC) and biochars by urea (URBC), respectively. The biochars prepared under different conditions were labeled with x–y–z, where x refers to hydrothermal temperature, y refers to hydrothermal time and z refers to the impregnation ratio of modifying agent to precursor. For example, 140-8h-1-APBC represented the biochar sample prepared by adding ammonium phosphate at the hydrothermal temperature of 140°C, hydrothermal time of 8 hr and impregnation ratio of 1.

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