



Effects of ball milling on the physicochemical and sorptive properties of biochar: Experimental observations and governing mechanisms[☆]



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ABSTRACT

With the goal of combining the advantages of ball-milling and biochar technologies, a variety of ball-milled biochars (BM-biochars) were synthesized, characterized, and tested for nickel (Ni(II)) removal from aqueous solution. Ball milling increased only the external surface area of low temperature biochars, but still dramatically enhanced their ability to sorb aqueous Ni(II). For higher temperature biochars with relatively low surface area, ball milling increased both external and internal surface area. Measurements of pH, zeta potential, stability, and Boehm titration demonstrated that ball milling also added oxygen-containing functional groups (e.g., carboxyl, lactonic, and hydroxyl) to biochar's surface. With these changes, all the BM-biochars showed much better Ni(II) removal efficiency than unmilled biochars. Ball-milled 600 °C bagasse biochar (BMBG600) showed the greatest Ni(II) adsorption capacity (230–650 compared to 26–110 mmol/kg for unmilled biochar) and the adsorption was dosage and pH dependent. Compared with the unmilled biochar, BMBG600 also displayed faster adsorption kinetics, likely due to an increase in rates of intra-particle diffusion in the latter. Experimental and modeling results suggest that the increase in BM-biochar's external and internal surface areas exposed its graphitic structure, thus enhancing Ni(II) adsorption via strong cation- π interaction. In addition, the increase in acidic surface functional groups enhanced Ni(II) adsorption by BM-biochar via electrostatic interaction and surface complexation. Ball milling thus has great potential to increase the efficiency of environmentally friendly biochar for various environmental applications.

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1. Introduction

Biochar is a carbonaceous material usually produced from pyrolysis of biomass. It can be produced from a wide range of feedstock materials including agricultural and industrial wastes (Ahmad et al., 2014; Inyang et al., 2016). Because of its relatively low cost and widespread availability, biochar has been considered as an alternative sorbent for environmental remediation (Ma et al., 2014; Tang et al., 2013). Previous studies have demonstrated that biochar

has excellent ability to sorb various organic and inorganic contaminants, including polycyclic aromatic hydrocarbons, pesticides, dyes, and heavy metals (Hu et al., 2015; Liu et al., 2016; Lyu et al., 2016; Tang et al., 2015; Yao et al., 2012a). Recently, efforts have been made to engineer biochar with improved properties for environmental application, particularly as an adsorbent for contaminant removal (Liu et al., 2016; Wang et al., 2015a, 2017; Zhang et al., 2015). Various pre- and post-treatment methods including acid/base modification, chemical oxidation, impregnation with natural and engineered nanoparticles, coating, steam and CO₂ activation, etc. have improved biochar's sorptive abilities to different extents (Fang et al., 2016; Inyang et al., 2014; Rajapaksha et al., 2016; Wang et al., 2015a; Xue et al., 2012; Yao et al., 2014). Almost all of these engineering methods heavily rely on chemical

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processes, which might have the disadvantage of being expensive or producing harmful wastes. Relatively little research attention has been paid to physical modification methods to improve biochar's physicochemical and sorptive properties without these potential drawbacks.

Ball milling is a powerful non-equilibrium processing method which mechanically reduces the grain size of solids to ultrafine scale (e.g., nanoscale) particles (Lyu et al., 2017a; Soares et al., 2015; Ullah et al., 2014). Ball-milling technology has recently been applied as an alternative and cost-effective method to produce novel engineered nanomaterials including nanocomposites (Fan et al., 2016; Wang et al., 2016). Wang et al. (2016) combined ball milling with electrochemical exfoliation to produce novel ball-milled graphene oxide (GO) that had much greater sorption U(VI) ability than the unmilled GO. Their results also indicate that ball milling can increase the types and amounts of oxygen functional groups on the surface and edges of graphene sheets. Using activated carbon as one of the feedstock materials, Ramanujan et al. (2007) and Gao et al. (2015) applied ball-milling process in the synthesis of carbon-based nanocomposites with improved properties for biomedical and environmental applications. Because of the similarities between biochar and activated carbon, ball milling thus may be used as a physical modification method in the production of engineered biochar.

Current research on the application of ball-milling technology in biochar modification, however, is still in its infancy. Only few studies have applied ball-milling technology in the production of engineered biochar with improved physicochemical properties (Peterson et al., 2012; Richard et al., 2016; Shan et al., 2016; Spokas et al., 2009). Peterson et al. (2012) investigated the effects of ball-milling parameters (i.e., mass ratio of milling media to biochar and mass of solvent) on increases in biochar surface area. Shan et al. (2016) reported that ball milling can facilitate the synthesis of ultrafine magnetic biochar/Fe₃O₄ composites with good ability to sorb aqueous pharmaceuticals (i.e., carbamazepine and tetracycline). While these results are encouraging, further work is needed to explore the variations and mechanisms related to ball milling's effects on biochar.

The overarching goal of this work was to promote the BM-biochar technology through improving current understanding of the fundamental processes and mechanisms that govern the physicochemical and sorptive properties of BM-biochar. Nine types of pristine biochars derived from different biomass and produced at three pyrolysis temperatures, were ball milled under optimal conditions. A range of laboratory experiments were conducted to characterize the physicochemical properties of the resultant BM-biochars. In addition, nickel (Ni(II)) was used as a model metal contaminant compound in batch experiments to compare the sorptive properties of the unmilled and milled biochars. Ni can pose a serious risk to public health (e.g., dermatitis, coughing, and lung cancer) due to its toxicity and carcinogenicity (Kurniawan et al., 2006). In the United States EPA sets maximum drinking water Ni concentration at 0.1 mg/L (EPA National Primary Drinking Water Regulations). The specific objectives were as follows: (1) determine and compare physicochemical properties of BM-biochars and their corresponding unmilled biochars; (2) measure and compare the sorption characteristics of Ni(II) onto the biochars; (3) shed light on the mechanisms that govern adsorption of heavy metals by BM-biochars; and (4) evaluate the effect of presence of additional cations on sorption of Ni from wastewater.

2. Materials and methods

2.1. Materials

All chemicals used in the present study were of analytical grade

and deionized (DI) water (18.2 MO) (Nanopure water, Barnstead) was used to make all the solution. Nickel (II) nitrate hexahydrate [Ni(NO₃)₂·6H₂O], sodium nitrate (NaNO₃), Magnesium nitrate hexahydrate [Mg(NO₃)₂·6H₂O], calcium nitrate [Ca(NO₃)₂·4H₂O], potassium chloride (KCl), concentrated hydrochloric acid (HCl), and sodium hydroxide (NaOH) were purchased from Thermo Fisher Scientific International Inc. (Waltham, Massachusetts, USA) and used as received. Feedstock biomass for pristine biochar, sugarcane bagasse (BG), bamboo (BB), and hickory wood chips (HC) were air-dried and sieved to 0.5–1 mm particles.

2.2. Preparation of pristine biochars

Biochar was prepared following the procedures of previous study (Creamer et al., 2014; Sun et al., 2014; Yao et al., 2012b). In brief, BG, BB, and HC were oven dried at 80 °C and placed in a quartz tube furnace (Olympic, 1823HE) to produce biochar through slow pyrolysis in a N₂ environment. Peak temperatures of 300, 450, and 600 °C were held for 2 h. The samples were then rinsed with DI water several times to remove impurities, oven-dried at 80 °C, and stored in air-tight containers prior to ball milling. The nine pristine biochar samples were labeled as BG300, BG450, BG600, BB300, BB450, BB600, HC300, HC450, and HC600, based on the feedstock type and peak pyrolysis temperature.

2.3. Preparation of BM-biochars

Biochars were placed in a planetary ball mill machine (PQ-N2, Across International, New Jersey, USA) within agate jars (500 mL) with balls (diameter = 6 mm, 180 g in each jar) and operated at 300 rpm in ambient air and with rotation direction altered every 0.5 h. To test biochar-to-agate ball mass ratio effect, various amounts of BG600 (1.8 g, 3.6 g, and 9 g) were mixed with 180 g agate balls (i.e., biochar-to-balls mass ratio = 1:100, 1:50, and 1:20) for 12 h. To investigate ball milling time effect, 1.8 g BG600 biochar was mixed with 180 g agate balls (biochar-to-ball mass ratio = 1:100) for 3, 6, 12, and 24 h, respectively. Following these experiments, all biochars were ball milled under the determined optimal milling conditions.

2.4. Biochar characterization

The specific surface area and pore volume of the pristine and BM-biochars were measured following the multipoint N₂ adsorption Brunauer-Emmett-Teller (BET) method (Autosorb-1, Quantachrome Instruments, Florida, USA). Samples were de-gassed under vacuum at least 24 h at 180 °C prior to analysis. Surface area was calculated according to BET theory using adsorption data in the 0.01–0.3 relative pressure range. Pore volumes were calculated using Barrett-Joyner-Halenda (BJH) theory using desorption isotherm leg data. Dynamic light scattering (DLS) tests were performed on a Malvern Zeta sizer Nano Instrument (Nano-S, Malvern Instruments, Worcestershire, UK) to test the hydrodynamic radius of the BM-biochars. Zeta potential of selected samples was determined using a ZetaPlus (Brookhaven Instrument Co., Holtsville, NY). Biochar pH values were measured with a ratio of 1.0 g of biochar in 20 mL DI water after equilibrium for 1.5 h using a AB-250 pH meter (Accumet, Fisher Scientific, America) (Rajkovich et al., 2011). The structure and surface morphology of the samples were characterized by scanning electron microscopy (SEM) (JSM-6400 Scanning Microscope, JEOL, Tokyo, Japan). The Boehm's titration method was used to quantify surface functional groups of selected biochar samples (Boehm, 1994).

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