



Characterization of cadmium removal from aqueous solution by biochar produced from *Ipomoea fistulosa* at different pyrolytic temperatures



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ABSTRACT

Water contamination by Cadmium (Cd) from industrial wastewater discharge is a critical global predicament. Its remediation creates an imperative need for the development of low-cost adsorbents with high efficiency. We demonstrate use of a novel adsorbent- *Ipomoea fistulosa* biochar as a low cost, efficient method to remove Cd from aqueous solution with minimum contact time period and adsorbent dose. Biochars produced from the abundantly available *Ipomoea* plant by slow pyrolysis at 350, 400, 500 and 550 °C were found to be effective in removal of Cd from aqueous solution. The efficiency of Cd removal has been considerably improved by activation of biochar using KOH which subsequently enhanced the porous structure resulting in more adsorption sites being exposed to Cd. The process of adsorption followed pseudo-second-order kinetic model. The mechanism for high adsorption efficiency is attributed to surface complexation of biochar particles with Cd(II) ions. Considering the excellent Cd sorption ability of our present adsorbent, there is heightened scope of using *Ipomoea* biochar as a new potential sorbent for removal of other toxic elements.

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1. Introduction

Water pollution caused by toxic heavy metals is a major cause of worldwide concern. Heavy metals like Cadmium (Cd) become environmental hazards by their toxicity at remarkably low concentrations, non-biodegradability and widespread occurrence in natural and anthropogenic environments (Friberg et al., 1986; Kumar et al., 2009, 2010, 2013; Tofiqhy and Mohammadi, 2011). Processes of erosion, abrasion of rocks, soils and volcanic eruptions furnish the natural sparse levels of Cd (Friberg et al., 1986; Kalderis et al., 2008). Cd is primarily introduced into the environment from anthropogenic point sources such as discharges from mining, batteries, metal plating, plastic and paper industries (Friberg et al., 1986; Grayson and Othmer, 1978; Cheung et al., 2000). Serious health hazards are posed by the toxic and carcinogenic nature of Cd. At elevated levels, it causes renal damage, hypertension, anaemia,

and is infamous for its association with the itai–itai disease. (Bodek et al., 1998; Kalderis et al., 2008). The World Health Organization (WHO), therefore, has established very strict maximum contaminant level (MCL) of 5 µg L⁻¹ in natural waters which is much lower than the level prescribed for other toxic elements (WHO, 2007). The implementation and maintenance of this designated low Cd standard in drinking water generates an ever-growing requirement for improved materials, technologies and high efficiency systems to remove Cd.

Established water treatment technologies, such as precipitation, membrane filtration, electro-coagulation, ion exchange and packed-bed filtration have been found to be fairly effective in reducing Cd concentrations (Mohan and Singh, 2002; Kim et al., 2013; Inyang et al., 2012). Unfortunately, many of these technologies were found to have high operational costs and/or sludge disposal problems (Sud et al., 2008). These significant drawbacks have stimulated the development of alternative cost effective and more efficient treatment technology for removal of Cd. In this context, biosorbents have emerged as the most suitable adsorbent for removal of Cd from aqueous solution (Demirbas, 2009).

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Biochar, which is co-produced from thermal degradation of organic materials such as forest residue, crop residue, wood, manure, and other materials, is a carbon rich material that is porous with oxygen functional groups and aromatic surfaces (Abdel Salam et al., 2011). Owing to its high surface-to-volume ratio and strong affinity for non-polar substances such as PAHs, dioxins, furans, and other compounds, biochar can be a potential sorbent for various environmental contaminants (Mohan et al., 2014; Ahmad et al., 2012; Cao et al., 2009; Chen et al., 2008; Yu et al., 2009). The ability of biochar to adsorb organic compounds has been demonstrated in previous several studies (Ahmad et al., 2012; Chen et al., 2008) whereas others have reported a good adsorption capacity for metal contaminants such as arsenic, mercury and lead (Inyang et al., 2012; Liu and Zhang, 2009). Biochars from slow pyrolysis and hydrothermal treating of rice husks, olive pomace, orange wastes, and compost were used for the remediation of Cu(II) ions (Pellera et al., 1999, 2012). Further, Lead (Pb) adsorption by slow (600 °C) pyrolysis biochars from raw and anaerobically digested sugarcane bagasse has been reported. (Inyang et al., 2011). Switchgrass biochar produced by hydrothermal carbonization process was effectively used for the removal of Cu(II) and Cd(II) (Regmi et al., 2012). Adsorption of radioactive materials like uranium using hydrothermally treated switchgrass biochar has been reported by Kumar et al. (2011b).

It is significant that activation of biochar using chemical or physical methods increases its surface area and improves the porous structure; consequently resulting in faster rate of adsorption. In the activated form, individual particles are strongly convoluted with micro porosity in the range of a few nanometers. These microscopic pores are exquisitely engineered to offer binding sites for a number of chemicals (Budinova et al., 2006) which is a major advantage of using activated biochar in remediation applications.

Ipomoea is an aquatic plant found in the wetlands of tropical countries like India where it grows abundantly. It is an evergreen, flowering, shrub with height ranging from 1.1 to 3 m, and stem diameter between 1.5 and 6 cm. In India, *Ipomoea* is a common amphibious weed occurring in fields, ponds, riversides and wet places/marshy areas (Kumar et al., 2015). Generally, *Ipomoea* growth is considered obstructive as it covers of large patches of productive land, creates impenetrable masses of tangled vegetation in water bodies and drainage systems which impedes water flow, drainage and leads to flooding. On the other hand, *Ipomoea* has been utilized as a source of paper pulp (Nandkumar, 2011), chemicals (Ganaie et al., 2014), drugs (Gupta et al., 2012; Rout and Kar, 2013) energy (Kumar et al., 2015) and latex (Patel et al., 2009). Moreover, many chemical constituents are found in the plant are responsible for its use in various medicines. It was reported that *Ipomoea carnea* is a suitable raw material for the production of activated carbon (Sharma and Bachheti, 2013). This lead us to investigate *Ipomoea* as a potential agent for metal removal, by designing a study to assess the efficiency of *Ipomoea* biochar in removing Cd(II) ions from aqueous solution. The general objective of this study was to develop a method to produce biochar from *Ipomoea fistulosa* through pyrolysis and to investigate the ability of *Ipomoea*-derived biochar to remove Cd(II) ions from aqueous solution. Specific objectives were: (I) to activate the biochar to improve its porous structure and compare Cd removal efficiencies with non-activated biochars derived at different pyrolytic temperatures; (II) to gain a fundamental understanding of adsorption process by physical and chemical characterization of biochar through coupled application of Brunauer-Emmett-Teller (BET), X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FT-IR) and Scanning electron microscopy (SEM) coupled with Energy dispersive X-ray spectroscopy (EDX) techniques; and (III) finally to understand the sorption mechanism by kinetics and isotherms study.

2. Material and methods

2.1. Preparation of biochar

Fresh *Ipomoea (Ipomoea fistulosa)* biomass collected from a wetland of Assam, India was washed and sun dried then cut into small pieces (0.5–1.5 cm). The pieces were ground with a Willy mill (SECOR Scientific Eng. Co.) and subsequently allowed to pass through 0.2 mm (70 mesh) (Saikia et al., 2015). Samples were then oven dried and kept in a desiccator for further analysis and pyrolysis experiments.

2.2. Pyrolysis and activation

The samples were pyrolysed in a fixed-bed reactor at five different temperatures, 350, 400, 450, 500 and 550 °C (BC350, BC400, BC500 and BC550), respectively with a heating rate of 40 °C min⁻¹ and a nitrogen flow rate of 1.2 L min⁻¹. The samples were kept at the final temperature for at least 1 h to allow sufficient time to finish pyrolysis. The details of the pyrolysis process can be found elsewhere (Saikia et al., 2015). The biochar thus produced were stored in desiccators prior to use. Further, activation of the biochar was done by mixing KOH pellets with biochar in 3:1 ratio followed by addition of water (100 mL) until all the activating agents were dissolved. The mixture was kept at room temperature for 2 h and then oven dried overnight at 105 °C. The dried samples were placed in reactor and heated to 300 °C at 40 °C min⁻¹ and held at this temperature for 1 h. The temperature was further increased to a final activation temperature at the same heating rate and held for 2 h before cooling down under nitrogen flow. The heat treatment was performed to reach the final temperature in the range 350–550 °C. After heat treatment, the product was thoroughly washed with water, followed by washing with 0.1 M HCl and finally with distilled water. The sample was then dried at 110 °C for 12 h and characterized for its physical and chemical properties.

2.3. Product characterization

Total carbon (C), nitrogen (N), and hydrogen (H) content in the biochar (BC) and activated biochar (ABC) samples were analyzed with a CHN Elemental analyzer (Perkin Elmer, 2400 Series-II). Oxygen was determined as the weight difference between the raw dried biochar and sum of C, H, N, and other non-volatile elements. Proximate analysis (determination of moisture content, ash content, volatile matter and fixed carbon) of biochar samples were carried out as per ASTM standard (ASTM, 2013). The pH of the BC and ABC (*i.e.* equilibrium pH of biochar/water suspension) was measured in a biochar (dry sample)/deionized water mass ratio of 1:20 with a pH meter (Fisher Scientific Accumet Basic AB15). Total surface area was measured using N₂ sorption on a NOVA 1200 analyzer and calculated using Brunauer-Emmett-Teller (BET) method. Scanning electron microscope (SEM) images were obtained with a JEOL, JSM-6290LV Scanning Microscope. Subsequently Energy Dispersive X-ray (EDX) microanalysis of adsorption of behavior of Cd(II) ions on the biochar was performed to confirm metal sorption on the biochar surface. A qualitative elemental analysis of the biochar after sorption was carried out using an accelerating voltage of 20 kV and a magnification of 500×. Surface crystallinity was determined using X-ray diffractometer (XRD) and the powder X-ray was recorded on a Rigaku miniflex diffractometer (Cu-K α radiation, $\lambda = 1.5406 \text{ \AA}$) in 2θ range of 10–70° at a scanning rate of 20° min⁻¹. The IR spectra were recorded in KBr pellets by a Nicolet (Impact 410) FTIR spectrometer in the diffuse reflectance mode at 4 cm⁻¹ resolution.

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