



Effect of modified coconut shell biochar on availability of heavy metals and biochemical characteristics of soil in multiple heavy metals contaminated soil

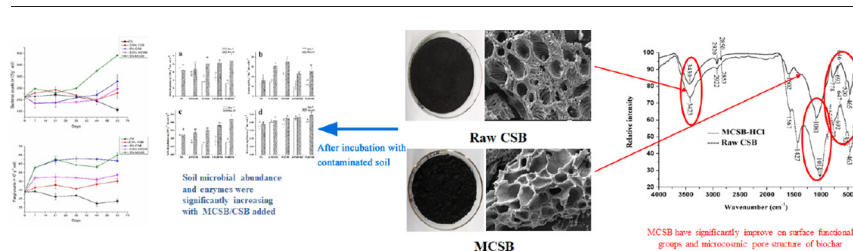
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HIGHLIGHTS

- CSB was modified with diluted HCl and ultrasonication.
- Modification effectively improved the microcosmic structure of CSB.
- MCSB is superior to raw coconut shell biochar in soil heavy metal remediation.
- MCSB is more effective than CSB on increasing soil biological activities.

GRAPHICAL ABSTRACT



ARTICLE INFO

Article history:

Received 14 March 2018

Received in revised form 8 July 2018

Accepted 9 July 2018

Available online xxxxx

Editor: F.M. Tack

Keywords:

Heavy metals

Availability

Modified coconut shell biochar

Immobilize

Biochemical activity

ABSTRACT

On account of the potential in immobilizing metals and improving soil environment, various biochar materials have been extensively applied in environmental remediation. The purpose of this experiment was to evaluate the effect of modified coconut shell biochar (MCSB) on the availability of metals and soil biological activity in multi-metals (cadmium (Cd), nickel (Ni) and zinc (Zn)) contaminated soil. MCSB was obtained from coconut shell biochar (CSB) by hydrochloric acid pickling and ultrasonication, which has significantly improved its surface functional groups and microcosmic pore structure. Sandy soil samples were incubated at 25 °C amended with MCSB or CSB by 0%, 2.5% and 5% addition for 63 days, respectively. The results showed that the acid soluble Cd, Ni and Zn decreased by 30.1%, 57.2% and 12.7%, respectively, in groups with 5% MCSB addition, which indicated MCSB had a better effect on immobilizing metals compared with CSB. In addition, higher soil biological activities were detected in different treatments compared with control (CK). Especially, the maximum bacterial number was found in 5% MCSB treatment, which increased by 149.43% compared with CK. Accordingly, our results suggested that MCSB could be used as an ameliorant to immobilize heavy metals in contaminated soils and improve soil physicochemical and biological properties.

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1. Introduction

Over the past few decades, multi-metals contamination of agricultural soil have become a serious and pervasive environmental problem,

as a result of both natural and anthropogenic activities such as mining, manufacturing, smelting activities and the improper application of fertilizers and pesticide in agriculture, and almost 10.18% Chinese agricultural soil has been polluted by heavy metals (Lwin et al., 2018; Xu et al., 2014). Heavy metals in soil are highly toxic, persistent and non-biodegradable (Wu et al., 2016). Excess heavy metals in the paddy soil not only diminish cereal production but also cause serious public health threat through various pathways (Zhou et al., 2014). Furthermore, heavy metals have adverse effects on the biological function (microbial

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community and soil enzymes) of soil (Song et al., 2017). Available fraction of metals should be essentially considered to estimate the ecological risk of heavy metals in contaminated soil.

Traditional remediation methods are costly and easily destructive to the soil ecological balance (Liu et al., 2018). Therefore, it is necessary to develop alternative techniques. *Situ* immobilization is an economical and environment friendly technology, which could effectively immobilize the contaminants and reduce eco-toxicity in contaminated soil by adsorption, complexation or (co-) precipitation (Jiang and Xu, 2013; Liu et al., 2018). Among various materials for remediation, carbon amendments have been widely deployed for soil and sediment remediation because for their high efficiency and low cost (Beesley et al., 2011). Biochar is a strongly sorbing carbonaceous charcoal material produced from the incomplete combustion of waste biomass (Huang et al., 2017a; O'Connor et al., 2017). As a suitable soil amendment, biochar possesses many positive properties: a) the resistance of biochar to chemical / biological degradation makes them to stay in soil for thousands of years; b) a large number of functional groups on the surface, such as phenolic, carboxylic, quinones, carbonyl, and hydroxyl; c) rich porous structure significantly affecting the basic properties of soil like pH, cation exchange capacity (CEC), retention of water and nutrients (Luo et al., 2017; Xu et al., 2014). Some researches have showed that the hardwood-derived biochar could reduce the water-soluble Cd and Zn in soil and significantly reduced their cytotoxicity (Huang et al., 2017a; Oustriere et al., 2017). Besides, biochar could increase microbial population and activities (Luo et al., 2017).

Coconut shell biochar (CSB), which has a great ability to adsorb heavy metals in contaminated soil (Jia et al., 2016; Paranavithana et al., 2016), was mixed and wrapped with impurities during its production process, which reduced its original microstructure characteristics and remediation efficiency. Therefore, it is necessary to modify the microstructure and surface properties of CSB to enhance remediation ability (Rajapaksha et al., 2016b). Researches have suggested that chemical modification could increase the number of free carboxyl and hydroxyl groups on the surfaces of biochar and enhance metals adsorption capacity (Rojjanateeranaj et al., 2017; Wu et al., 2017). Washing biochar with strong acids (such as H_3PO_4 , H_2SO_4 , HNO_3 , and HCl acid) has been studied for aqueous oxidation, which could modify porous structure and enhance adsorption effect on heavy metals (Peng et al., 2017). However, the effectiveness of HCl and ultrasonic modifying biochar and their implications for contaminated soil remediation processes have not yet been systematically studied. So, in this study, acid pickling and ultrasonic treated methods were selected to improve the physicochemical properties of CSB, which could improve immobilization effect of heavy metals and soil microenvironment.

The objectives of this study were (1) to reveal the differences in the physicochemical properties between CSB and modified coconut shell biochar (MCSB) by Scanning Electron Microscope (SEM) and Fourier transform infrared spectroscopy (FTIR); (2) to investigate the immobilizing ability of CSB and MCSB in multi-metals contaminated soil; (3) to evaluate the influence of CSB and MCSB on soil physicochemical (such as pH, CEC etc.) and soil biological properties (such as microbial counts and soil enzymes activities etc.).

2. Materials and methods

2.1. Soil and biochar preparation

The contaminated soil was collected from topsoil of paddy fields in Mianzhu County, Sichuan Province, China (31°24'N, 104°13'E). The soil sample was thoroughly mixed, air dried, and passed through a 2 mm sieve prior to initiating the experiments. CSB was obtained from Desheng Active Carbon Factory (Jiangsu, China) produced by industrial pyrolysis reactor, with a retention time of 6 h at approximately 800 °C (Tripathi et al., 2016).

MCSB was obtained by the following procedure: 5 g of CSB and 250 mL of hydrochloric acid (1 M) were mixed thoroughly in 500 mL beaker and ultrasonicated for 3 h with interval stirring. Finally, the material was filtered and washed repeatedly with ethanol and deionized water until filtrate's pH was neutral, then dried to constant weight (Li et al., 2016a; Rajapaksha et al., 2016a).

2.2. Main properties analysis of soil and biochar

The pH of biochar was measured in a biochar/water slurry at a 1:20 (w/v) ratio after stirring for 30 min. The electrical conductivity (EC) of biochar was measured at 25 °C after suspending in water (1:10 w/v). CEC was measured using a modified compulsive exchange method (Skjemstad et al., 2008; Wu et al., 2016). Soil pH was measured in a soil/water slurry at a 1:2.5 (w/v) ratio and the EC was measured at a ratio of 1:5 (w/v). Organic carbon (OC) was determined by Potassium Dichromate Oxidation Spectrophotometric Method (HJ 615-2011). Soil texture was analyzed using hydrometer measuring method (Meier, 1999). The CEC of soil was measured by the compulsive exchange method (Meier, 1999). Available phosphorus (Olsen-P) was extracted with NH_4F-HCl and determined by spectrophotometry (NY/T 1121.7-2014). Total heavy metals content in soil and biochar were determined by Flame Atomic Absorption Spectrometry (FAAS) (VARIAN, SpecterAA-220Fs) (Wu et al., 2016).

To comprehend microstructure and biochemical changes of MCSB, SEM equipped with energy dispersive X-ray spectrometry (EDS) elemental mapping (JSM-5900LV, Japan) were utilized (Li et al., 2017; Luo et al., 2018). FTIR spectrum of the CSB and MCSB were recorded by using a Nicolet Nexus6700 spectrophotometer (Thermo Fisher Scientific, USA) (Abdul et al., 2017; Tang et al., 2015).

2.3. Incubation experiments

In this study, different levels of CSB or MCSB were added into soil to evaluate their effects on heavy metals availability and microbial characteristics. Pre-prepared soil (250 g) were placed in a 500 mL beaker and thoroughly mixed with amendments and then incubated at 25 °C for 63 d. The treatments were control (CK, without remediation), 2.5% CSB (mass ratio of biochar and soil), 5% CSB, 2.5% MCSB and 5% MCSB. Each treatment was replicated three times. In the remediation process, each beaker was weighed and maintained a constant humidity (70%) by supplementing the reduced moisture every 3 d. All treatments were sampled at 7 and 63 d for soil enzyme activities measuring. At the meantime, the samples at 7, 21, 35, 49 and 63 d were collected to analysis for the indigenous microbial counts.

2.4. Sequential extraction of BCR

Heavy metals fractions in contaminated soil was performed using the modified BCR procedure (Dang and Obbard, 2006; US EPA Method 3052, 1996): Concisely, 1.0 g of soil was shaken at 25 °C, 250 rpm for 16 h with 40 mL of 0.11 M acetic acid, then centrifuged for 5 min at 8000 rpm and the supernatant was collected for assay acid extractable fraction. The above mentioned residues were shaken at 25 °C, 250 rpm for 16 h with a 40 mL mixture of 0.5 M hydroxylamine hydrochloride (pH 2.0) and 0.05 M HNO_3 , then centrifuged for 5 min at 8000 rpm and the supernatant was collected for assay reducible fraction. For oxidizable fraction, the above mentioned residues were added to 10 mL 30% H_2O_2 (pH = 2.5), placed in a water bath at 85 °C for about 1 h until the volume of liquid was <3 mL, then the residue was extracted with 10 mL 30% H_2O_2 and 50 mL 1.0 M acetic acid (pH = 2) was finally added and centrifuged for assay when the volume of liquid was <1 mL. Finally, the above mentioned residual soil was digested with a mixture of 6 mL HNO_3 , 5 mL $HClO_4$, and 4 mL HF using a microwave digestion method to extract the residual fraction. The concentration of different metals fractions in samples were

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