



## Differential release of sewage sludge biochar-borne elements by common low-molecular-weight organic acids

Danielle Vause, Natalie Heaney, Chuxia Lin\*

School of Environment and Life Science, University of Salford, Greater Manchester M5 4WT United Kingdom



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### ABSTRACT

Biochar materials originated from sewage sludge may contain elevated levels of potentially toxic elements. There was a lack of information on the mobility of biochar-borne elements, as driven by low-molecular-weight organic acids (LMWOAs) contained in plant root exudates. A batch experiment was conducted to examine the effects of three common LMWOAs on the release of major elements and trace elements with a focus on various potentially toxic trace elements. The results showed that substantial amounts of Al, Mn, Fe, K, Na and Mg were extracted from two sewage sludge-derived biochar materials by the LMWOAs. A much higher release rate of potentially toxic trace elements was observed in the presence of LMWOAs, as compared to reported data using extractants not encountered in root exudates. The LMWOA-driven releasability of various potentially toxic trace elements was in the following decreasing order: Zn > Ni > Pb > Cu > Cr > Co = Cd. Other trace elements that are subject to mobilization in the presence of LMWOAs included B, Ba, In, Li and Sr except Ba under oxalic acid extraction. Among the three LMWOAs, oxalic acid showed a generally stronger capacity to mobilize these metals. The findings obtained from this study provides new information that can be used for better evaluating the phyto-availability of trace elements bound to sewage sludge-originated biochar materials.

### 1. Introduction

Pyrolysis is viewed as an effective approach for the treatment of sewage sludge while simultaneously generating bio-energy (Hossain et al., 2011; Agrafioti et al., 2013). The residue of sewage sludge pyrolysis could also be used as biochar for various environmental applications (Yuan et al., 2015). However, owing to generally elevated concentration of trace elements in the feedstock (Van Wesenbeeck et al., 2014), biochar materials originated from sewage sludge may be a source of trace elements to the environments (Hossain et al., 2010), which may limit its beneficial utilization given the potential environmental impacts from the release of the biochar-borne trace elements of potential toxicity. Therefore, it is important to understand the releasability of sewage sludge biochar-borne trace elements in order to evaluate the environmental risk associated with a given beneficial application. The mobility of sewage sludge biochar-borne trace elements was tested by various researchers using different extractants e.g. distilled water (Gondek et al., 2014; Gondek and Mierzwa-Hersztek, 2017), NH<sub>4</sub>NO<sub>3</sub> (Mierzwa-Hersztek et al., 2018), dilute H<sub>2</sub>SO<sub>4</sub> and HNO<sub>3</sub> (Zhou et al., 2017), DTPA-CaCl<sub>2</sub>-TEA (Liu et al., 2014; Lu et al., 2016; Huang et al., 2017). Liu et al. (2014) found sewage sludge biochar had an elevated level of total Cd, Cr, Cu, Pb and Zn but only a

small fraction of these metals was available for plant uptake and their bioavailability was in the following decreasing order: Zn > Cr > Cu > Pb > Cd. Similarly, Gondek and Mierzwa-Hersztek (2017) reported low bioavailable forms of trace elements to the order of Zn > Pb > Cd > Cu. A TCLP study by Agrafioti et al. (2013) revealed that leachate from sewage sludge biochar contained < 0.74 mg/kg of heavy metals.

Soil application represents a major avenue for biochar utilization (Ahmad et al., 2014). In the rhizosphere, various low-molecular-weight organic acids (LMWOAs) are present due to root exudation (Jones and Darrah, 1994). Therefore, the added biochar materials are likely to be exposed to these naturally occurring LMWOAs, which may cause liberation of the biochar-borne elements through acidification, reduction or/and complexation (Onireti and Lin, 2016; Alozie et al., 2018). While mobilization of trace elements from sewage sludge biochar materials has been investigated using chemical reagents that are not encountered in rhizosphere as mentioned above, there is currently no information on the chemical behaviour of sewage sludge biochar-borne elements in the presence of LMWOAs that are frequently encountered in rhizospheric soils. This represents a knowledge gap for understanding the phyto-availability of sewage sludge biochar-borne elements in the soils amended with sewage sludge biochar materials. Here, we report the

\* Corresponding author.

E-mail address: [C.Lin@salford.ac.uk](mailto:C.Lin@salford.ac.uk) (C. Lin).

observed release of various elements from two selected sewage sludge-originated biochar materials produced using the same feedstock but at different pyrolysis temperatures in the presence of three common root-released LMWOAs (citric acid, oxalic acid and malic acid). The objective was to gain a preliminary understanding of the role of these three common LMWOAs in mobilizing various elements from the two selected sewage sludge biochar materials, which will provide a basis for guiding further investigations to obtain insights into the biochemical mechanisms responsible for plant uptake of sewage sludge biochar-borne elements.

## 2. Materials and methods

The two sewage sludge biochar materials (labelled as SS550 and SS700 for that produced under low-oxygen conditions at a pyrolysis temperature of 550 °C and 700 °C, respectively) used in the experiment were purchased from the United Kingdom Biochar Research Centre (UKBRC). The basic physical and chemical characteristics of the biochar materials provided by the manufacturer are given in Table S1 in the Supplementary materials. The functional groups on the biochar surfaces were identified using a Fourier transform infrared spectrometer (FTIR).

A batch experiment was conducted with one control and three treatments being set for each biochar material. For the controls, 2 g of the respective biochar was extracted by 20 mL of deionized water. For the treatments, 20 mL of 0.02 M citric, malic or oxalic acid solution was used, respectively. The concentration of LMWOAs set for this study is within the concentration range encountered in rhizosphere (Jones and Darrah, 1994). Details on the experimental set-up are provided in Table S2 in the Supplementary materials.

After adding all the ingredients, the batch reactors were shaken in a rotary shaker at 150 rpm for 1 h. After shaking, the pH and electrical conductivity (EC) in the solution were measured using a calibrated pH meter and an EC meter, respectively. The solution was then filtered for determination of various elements using an inductively coupled plasma atomic emission spectrometer (Varian 720ES ICP-OES).

The experiment was performed in triplicate. All chemical reagents used in the experiment were of analytical reagent grade. Ultrapure water (18.2 MΩ/cm) was used throughout the entire course of the experiment. One-way analysis of variance (ANOVA) and Duncan's multiple range tests were used to determine the statistically significant difference between the means of the controls/treatments.

The release rate of a biochar-borne element by each of the LMWOAs is calculated using the following formula:

$$\text{Release rate (\%)} = E_{\text{LMWOA}}/E_{\text{total}} \times 100$$

where ELMWOA stands for the concentration of an extracted element by an LMWOA and Etotal denotes the total concentration of that biochar-borne element.

## 3. Results and discussion

The spectra of the two-biochar materials used for the study are shown in Fig. 1. No major changes in surface functionality for the biochar materials produced under different pyrolysis temperature were observed. This suggests that the biochar materials were relatively stable at these production temperatures in terms of functionality (Song et al., 2014). The broad stretch observed between 3200 and 3500 cm<sup>-1</sup> can be assigned to -OH stretching (Keiluweit et al., 2010) and a slight decrease in intensity can be observed with increased pyrolysis temperature which is indicative of increased dehydration of the biochar material (Kim et al., 2012). At around 2921 cm<sup>-1</sup> a small vibration can be observed for SS700 which is attributable to a CH aliphatic stretch (de Jesus et al., 2017). Another stretch, which indicates the presence of a C=O bond of amide I at ~1617 cm<sup>-1</sup> (Lu et al., 2013; Fan et al., 2016). The strong peak observed for both biochar at ~1385 cm<sup>-1</sup> is most likely aliphatic CH<sub>3</sub> deformation (Özçimen and Ersoy-Meriçboyu,

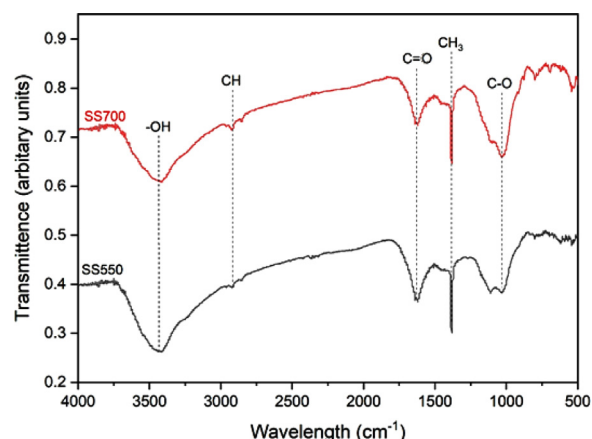


Fig. 1. FTIR spectra of the two biochar materials (SS550 and SS700) used in the experiment.

2010). A broad stretch present between 1000 and 1200 cm<sup>-1</sup> can be assigned as aliphatic ether C-O and alcohol C-O stretching (Özçimen and Ersoy-Meriçboyu, 2010), which becomes more pronounced with increased pyrolysis temperature. There was only a slight difference in absorbance between the pre-extraction and post-extraction FTIR spectra (data not shown), indicating that no removal or formation of new functional groups on the reacted biochar materials. Scanning electron micrographs revealed a porous structure with substantial amounts of the precipitates for both biochar materials (Fig. S1).

After the reaction, the mean pH in the control for SS550 was 7.20, which was significantly ( $P < 0.05$ ) lower than that (pH 8.87) in the control for SS700 (Table 1). This was expected because the former was less alkaline, as compared to the latter (Table S1 in the Supplementary materials). For each biochar material, the mean pH in any LMWOA treatment was always lower than that in the respective control. This was due to acidification by the added organic acids. EC in the solutions also generally increased after addition of citric and malic acids (Table 1), reflecting the dissolution of the biochar-borne compounds.

Values are means  $\pm$  standard error of the mean ( $n = 3$ ). Means in each column with different letters indicate statistical significance ( $P < 0.05$ )

In both controls (SS550 and SS700), no Al was detected. For Fe, only a trace amount was detected for SS700 and none was detected for SS550. These are expected since the solubility of aluminium and iron oxides is low at neutral and slightly alkaline pH (Schwertmann, 1991). Significantly more ( $P < 0.05$ ) K was released from SS550, as compared to SS700. For both biochar materials, oxalic acid tended to extract more Al, as compared to either citric acid or malic acid. More Al tended to be extracted from SS550, as compared to SS700 (Table 2). It is likely that the biochar-borne Al was mainly in hydrolysed form. Given that the solution pH was above 6, the mobilization of biochar-borne Al was likely to be driven by complexation rather than acidification. Under the set experimental conditions, oxalic acid is more effective in terms of solubilizing aluminium hydroxides. The same trend (oxalic acid

Table 1  
pH and electrical conductivity in the solution.

Treatment	pH	EC ( $\mu\text{S}/\text{cm}$ )
SS550	7.20 $\pm$ 0.01b	259 $\pm$ 1.67ab
SS700	8.87 $\pm$ 0.06a	185 $\pm$ 6.84b
SS550-C	6.44 $\pm$ 0.12f	411 $\pm$ 69.55a
SS550-M	6.43 $\pm$ 0.04f	385 $\pm$ 116.43a
SS550-O	6.57 $\pm$ 0.02ef	258 $\pm$ 6.00ab
SS700-C	6.66 $\pm$ 0.02de	254 $\pm$ 0.67ab
SS700-M	6.77 $\pm$ 0.03d	258 $\pm$ 9.60ab
SS700-O	7.02 $\pm$ 0.02c	144 $\pm$ 1.76b

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