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DISSOLVED ORGANIC CARBON CONTENT AND LEACHABILITY OF BIOMASS WASTE BIOCHAR FOR TRACE METAL (Cd, Cu AND Pb) SPECIATION MODELLING

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Abstract. Dissolved organic carbon (DOC) interacts with dissolved trace metal affecting their mobility and bioavailability through the formation of DOC–metal complexes. Several types of biochar (BC) produced from slow pyrolysis of wood chips (WC), lignin (LG), and digested sewage sludge at 450 and 700 °C were tested for DOC leaching via batch and up-flow percolation test methods. Trace metal (Cd, Cu, and Pb) speciation modelling in BC eluates was carried out combining measured data (i.e., DOC, pH, temperature, and dissolved trace metal concentrations) with data reported in the literature regarding fractions of DOC that are inert or active (i.e., fulvic acids (FA) and humic acids (HA)) in metal binding. BC from LG (BCLG) and WC (BCWC) at 700 °C released lower cumulative amounts of DOC compared with BC at 450 °C in the range 0.02–0.07% and 0.06–0.09% of total carbon content, respectively. For both pyrolysis temperatures, BCWC exhibited a higher tendency to release DOC compared to BCLG. Speciation modelling results showed the predominance of FA and HA complexes of Cd, Cu, and Pb in all the eluates from BCWC and BCLG irrespective of the inert fractions of DOC or the different fractions of active FA and HA considered.

Keywords: biochar, dissolved organic carbon, trace metal speciation, leaching, waste management technologies.

Introduction

In water systems, dissolved organic carbon (DOC) interacts with dissolved trace metal affecting their mobility and bioavailability through the formation of DOC–metal complexes.

Biochar-borne DOC may represent a source of impairment of aquatic environment containing organic species producing an inhibitory effect on the growth of aquatic micro-organisms (Smith *et al.* 2012) or increasing metal mobility through complexation.

Several studies have reported a decrease in metal availability and leachability from biochar (BC) as a result of physical and chemical alterations occurring during pyrolysis of waste biomass (Lu *et al.* 2011; Lin *et al.* 2012; Devi, Saroha 2014).

Feedstock selection and pyrolysis conditions (i.e., heating rate, residence time and temperature) have been reported to influence the content, composition, and extent to which DOC is released from BCs (Mukherjee, Zimmerman 2013; Alburquerque *et al.* 2014; Jamieson *et al.* 2014).

As for the influence of feedstock type on DOC content as well as the extent to which it is prone to leaching from BC, several authors have reported that BCs from wood and woody biomass generally release less DOC compared to non-wood biomass (e.g., agricultural residue and animal wastes) (Mukherjee, Zimmerman 2013; Graber *et al.* 2014). Alburquerque *et al.* (2014) reported DOC content highest in BC from woody biomass (0.8–6.7 g/kg) consisting of leaves and thin branches (i.e., olive tree pruning), followed by BC from agricultural residue, such as almond shell (0.6–2.9 g/kg) and olive stone (0.3–1.5 g/kg), and wood biomass, such as pine wood (0.3–1.4 g/kg). In the work of Graber *et al.* (2014), DOC was detected at 1.5 to 9.7 times higher concentration in the extracts of BCs from non-wood biomass (i.e., green house waste and olive pomace) than in the extracts of BC from wood biomass (i.e., wood chips). Furthermore, these authors observed

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that the influence of feedstock selection for BC production on the measured DOC concentrations was highest for BC at lower temperatures (i.e., 350 and 450 °C) compared with higher temperature (i.e., 600 °C).

Several authors have noted a decrease in DOC content with an increase in pyrolysis temperature (Gaskin *et al.* 2008; Lin *et al.* 2012; Mukherjee, Zimmerman 2013; Zhang *et al.* 2015; Alburquerque *et al.* 2014). In the work by Smith *et al.* (2016), each increase in pyrolysis temperature of 100 °C led to a decrease in DOC content in the range 41–44% in BC produced from lignin in the temperature range 300–500 °C. Al-Wabel *et al.* (2013) reported the largest decrease (from a DOC content of about 1.8 g/kg to <0.6 g/kg) for BC produced in the range 200–400 °C, probably due to the low carbonisation reached at 200 °C and the resulting higher content of organic materials suggested by the highest H/C ratio.

Conversely, some studies reported an increase in the DOC content of BC with increases in pyrolysis temperature from 350 to 400 °C (Luo *et al.* 2015) and from 350 to 450 °C (Graber *et al.* 2014).

Jamieson *et al.* (2014) suggested that pyrolysis conditions such as fast heating rates and low residence times may lead to produce BCs that are more prone to leaching DOC. These authors observed an increase of about 90% in DOC concentration in the leachate from fast pyrolysis BC at 600 °C compared with the slow pyrolysis BC at 380 °C.

DOC composition of BC is affected by the processing conditions (e.g., pyrolysis temperature) selected for producing BC (Trompowsky *et al.* 2005; Lin *et al.* 2012; Uchimiya *et al.* 2013). Trompowsky *et al.* (2005) reported a temperature trend for the yield of fulvic acids (FA) and humic acids (HA) extracted from wood BC pyrolysed in the range 300–550 °C. At the lowest pyrolysis temperature (300–350 °C), the yield of FA was highest whereas the yield of HA was the lowest. With each temperature increase of 50 °C from 350 to 550 °C, the yield of FA exhibited a steady decrease, whereas HA reached its maximum yield at 450 °C and subsequently decreased. Uchimiya *et al.* (2013) observed that DOC released by biochars in the temperature ranging 350–500 °C was enriched with carboxyl and poly(phenolic) functionalities that are able to affect metal mobilisation via complexation with metal ions.

As for the mechanism that leads to the release of DOC from BC, Jones *et al.* (2011) suggested that DOC is released at first from the surface and later from the BC matrix following a diffusion-limited mechanism. Observing a good fit between the curves showing the cumulative released quantity of DOC determined via leaching and batch methods, Mukherjee and Zimmerman (2013) suggested that the amount of DOC released from biochars depends on the ratio of the solvent to solute rather than parameters, such as contact time and energy of mixing. In column leaching tests performed by Beesley and Marmiroli (2011) and Iqbal *et al.* (2015), DOC was observed to be rapidly released from biochar with no substantial amount of DOC recorded after a few eluate samples.

As for trace metal speciation in water, dissolved organic matter (DOM) can be subdivided into an active fraction comprising humic substances able to complex metals (i.e., fraction of DOM active for metal binding) and other biogenic molecules (e.g., amino acids, proteins and carbohydrates) not affecting metal speciation (i.e., fraction of DOM inert for metal binding) (Mueller *et al.* 2012). Based on an extensive literature review, Groenenberg and Lofts (2014) reported active fractions of DOM in the range 31–100%. DOM is generally quantified as DOC (Wood *et al.* 2011) (e.g., 2 times the DOC values measured in the sample analysis) (Ahmed *et al.* 2013).

The capacity of DOC to form metal complexes depends on the presence and availability of carboxylic and phenolic functional groups. Through deprotonation, these functional groups provide DOC with negatively charged sites that are able to bind metal cations (Dee 2016). Furthermore, as these functional groups exhibit an acid-base behaviour, the metal binding ability of DOC depends on the pH of the system (Christensen, J., Christensen, T. 2000).

The chemical speciation program WHAM7 (Tipping *et al.* 2011) is based on WHAM (Tipping 1994), a mechanistic-based equilibrium model simulating the interactions of protons and metal cations with humic substances. As input data, WHAM model requires the fraction of DOM that is active for proton/metal binding and the sub-fractions that act as isolated purified humic substances (i.e., active fractions of HA or FA). The concentration of humic substances to be input in the model is often based on the measured DOC concentration and on some assumptions about DOC composition and reactivity (i.e., HA or FA) (Ahmed *et al.* 2013). For a comprehensive review about the most advanced ion-binding models (including WHAM) applied so far for predicting trace element speciation in water systems, we recommend the work by Groenenberg and Lofts (2014).

In order to avoid potential drawbacks in BC water system application (e.g., filtering media) and to ensure the durability of BC in biofiltration systems (Baltrėnas *et al.* 2016) or its application in soil (Baltrėnaitė *et al.* 2016) resulting from leaching of DOC, it is fundamental to determine the rate of carbon content that is prone to leaching as DOC and its effect on trace metal speciation and mobility.

The goals of the present research were:

1. To evaluate and compare changes in DOC leachability from BCs produced from different types of biomass feedstock (i.e., wood chips, lignin, and digested sewage sludge) at different temperatures.

2. To model trace metal (Cd, Cu, and Pb) speciation in the BC eluates.

To the best of the authors' knowledge, few studies have assessed DOC content and composition of BC from lignin (Smith *et al.* 2016) or trace metal concentration, leachability and bioavailability in BC from lignin (Mancinelli *et al.* 2016). Hence, the need for investigating DOC content and leachability of BC and for modelling trace metal speciation in the BC eluates.

1. Materials and methods

1.1. Biochar production

Wood chips (WC) from a sawmill in Kaliningrad (Russian Federation), lignin (LG) from a sugar refining industry located in Lithuania, and digested sewage sludge (DSS) from an urban waste water treatment plant in Klaipeda (Lithuania) were used as raw materials for BC production.

The pyrolysis of the raw materials was carried out according to the procedure described elsewhere (Komkiene, Baltrenaite 2016). Briefly, the slow pyrolysis was performed for 120 min with a heating rate of approximately 10 °C/min until the two desired pyrolysis temperatures of 450 and 700±5 °C were reached.

The obtained BCs were assigned the following codes:

- BCWC450 and BCWC700 for BC from wood chips (BCWC) at the pyrolysis temperatures of 450 and 700 °C, respectively,
- BCLG450 and BCLG700 for BC from lignin (BCLG) at the pyrolysis temperatures of 450 and 700 °C, respectively,
- BCDSS450 and BCDSS700 for BC from digested sewage sludge (BCDSS) at the pyrolysis temperatures of 450 and 700 °C, respectively.

1.2. Up-flow percolation test

An up-flow percolation test was performed in order to determine the leaching behaviour of BCs, as fully described in Peckyte and Baltrenaite (2015). Columns were packed with 137.5 g BCWC450, 130.8 g BCWC700, 210.5 g BCLG450, and 316.7 g BCLG700. BCs were soaked with a leachant (i.e., deionised water) for three days, and then hydrostatic pressure was applied resulting in a flow rate of approximately 12 ml/h. The first volume of eluate equal to a cumulative liquid to solid (L/S) ratio of 1 l/kg dry weight (DW) (i.e., 0.137, 0.131, 0.210, 0.317 litres of eluate from BCWC450, BCWC700, BCLG450, and BCLG700, respectively) was not analysed for DOC. At the beginning of the column leaching test (i.e., until L/S ratios of 0.1 or 0.5 l/kg DW), labile species may be rapidly released by the tested material with the resulting elution curve showing a first flush behaviour (Van der Sloot, Dijkstra 2004). In the present research, DOC measurements were taken for the eluate collected at L/S ratios equal to 1, 3, and 5 l/kg DW, thus not considering the fraction of DOC in BC that is subject to rapid washout.

1.3. Chemical analysis

The eluate collected during the up-flow percolation test of BCWC and BCLG at L/S ratios equal to 1, 3, and 5 l/kg DW was transferred to the laboratory for electrical conductivity (EC), pH and DOC measurements and trace metal analysis.

EC and pH were measured immediately after sampling. EC was measured with a conductivity meter (inoLab Cond 740 WTW) in accordance with the methodology of *ISO 7888: Water quality – Determination of electrical conductivity*. pH was measured with an electronic glass electrode (SevenMulti ion/pH/ORP module Mettler Toledo) after calibrating the pH-meter with buffer solutions at pH values of 4, 7, and 10. pH measurements were carried out in accordance with the methodology described in *ISO 10523: Water quality – Determination of pH*. For DOC analysis, samples of about 40 ml volume were taken from the eluate collected during the up-flow percolation test. After filtering the samples through a syringe (pore size 0.45 μm, Pall Corporation), DOC was determined by employing a total organic carbon (TOC) analyser (Shimadzu–V CSN).

DOC concentrations in BCs were also determined via a batch test method. Water extracts were prepared using a 1:20 (w:v) suspension after shaking (RS12 Rotoshake, Gerhardt) for 180 min according to a method described by Dias *et al.* (2010). Samples were filtered through a syringe (pore size 0.45 μm, Pall Corporation), and DOC was determined by a TOC analyser (Shimadzu-V CSN).

DOC concentrations were corrected by subtracting the mean concentration (20.4 mg/l), as measured in the blank samples. Samples were stored at 4 °C in the dark until DOC analysis was accomplished.

As for the batch test and up-flow percolation test, the dissolved organic carbon (DOC; g/kg) contents of the BCs were calculated using the following equation, adapted from Jamieson *et al.* (2014):

$$\mathrm{DOC} = \frac{V \times C}{M}, \tag{1}$$

where V – volume of deionised water utilised as eluate or extractant, l; C – measured concentration of DOC in the sample, mg/l; M – dry mass of biochar, g.

As far as the up-flow percolation test is concerned, the cumulative released quantity $\left(\sum_i \mathrm{DOC}_i\right)$ of DOC, g/kg, was calculated for analysis in the eluate fraction as described below:

$$\sum_i \mathrm{DOC}_i = \sum_i \frac{V_i \times C_i}{M}, \tag{2}$$

where M – dry mass of biochar, g; i – index of the eluate fraction from 1 to 3; C_i – measured concentration of DOC in the sample, mg/l; V_i – volume of eluate fraction varying with the L/S ratios set in the up-flow percolation test, l.

The cumulative released quantity $\left(\sum_i \mathrm{DOC}_i\right)$ of DOC expressed as a percentage of total carbon content of the biochar was calculated as follows:

$$\sum_i \frac{\mathrm{DOC}_i}{\mathrm{TC}} \times 100 = \sum_i \frac{V_i \times C_i}{M} \times \frac{1}{\mathrm{TC}} \times 100, \tag{3}$$

where DOC_i – dissolved organic carbon calculated as in equation (2), g/kg; TC – total carbon content of biochar, g/kg; i – index of the eluate fraction; V_i – volume of deionised water utilised as eluate, l; C_i – measured concentration of DOC in the sample, mg/l; M – dry mass of biochar, g.

For trace metals (Cd, Cu, and Pb) analysis, 50 ml of the eluate from the collected sample was filtered through sterile Acrodisc syringe filters with a Supor (hydrophilic polyethersulfone) membrane and pore size 0.45 μm (Pall Corporation) (Mancinelli *et al.* 2015). Measurements were taken by flame atomic absorption spectrophotometry (FAAS) or graphite furnace (GFAAS) analysis according to the procedure described by Baltrėnaitė *et al.* (2016).

1.4. Quality assurance

For quality assurance, measurements were repeated three times. All samples were prepared in duplicate. For DOC determination in BCs, two blank samples were prepared following the entire sequence of steps, as described for DOC analysis.

1.5. Calculation and statistical analysis of the experimental data

Statistics were accomplished using Excel (Microsoft). Analysis of Variance (ANOVA) was used to test differences between the data using Excel (Microsoft). Assuming the truth of the null hypothesis (i.e., the population means are all equal), the p-value is the probability of observing an event at least as extreme as the one in the tested sample data. In the present study, the differences were significant with $p \leq 0.05$ (i.e., 5% risk of rejecting the null hypothesis when it is true).

Comparisons between the DOC concentrations in the water extracts of BC (i.e., measurement value) determined via the batch test method, pyrolysis temperature and the feedstock (i.e., the nominal variables chosen to organise the measurements into groups) were carried out using the two-way ANOVA with replications. The two-way ANOVA with replications was calculated within each feedstock type in order to determine any statistical differences between the pH or EC values of the eluates collected from the BC up-flow percolation test (i.e., the measurement value), L/S ratios, and the pyrolysis temperatures (i.e., the nominal variables).

Comparisons between the DOC concentrations of the eluates collected from the BC up-flow percolation test (i.e., measurement value), pyrolysis temperatures, and the *L/S* ratios (i.e., the nominal variables chosen to organise the measurements into groups) were carried out using the two-way ANOVA with replications.

1.6. WHAM modelling

Trace metal speciation modelling was done using WHAM/Model 7 (Tipping *et al.* 2011) for waters. The WHAM model determines metal binding to DOM, such as metal binding to HA and FA. In order to define the binding parameters that represent the nature and number of sites available for metal binding with organic ligands, input data WHAM model requires the fraction of DOM that is active for proton/metal binding and sub-fractions that act as HA or FA. In the present study, DOM was considered colloidal, and it was assumed to consist of 50% *DOC*, which is 2 times the DOC values measured in the sample analysis (Ahmed *et al.* 2013). Since DOC composition and reactivity (i.e., active fractions of HA and FA) were not determined in the present study, a range of fractions of DOC acting as active HA and FA according to values reported in the literature (Table 1) was assumed. We also assumed that the active fraction of DOC in BC has the binding properties of FA and HA, as these fractions were detected in the BC extract in a study by Trompowsky *et al.* (2005).

Table 1. Summary of the input values for trace metal speciation modelling: fractions of dissolved organic carbon (DOC) considered inert and active (i.e., colloidal fulvic acid (FA) and humic acid (HA)) to metal complexation

	Inert, %	Active FA, %	Active HA, %	References
DOC	0	90	10	Unsworth *et al.* 2006
	35	65	0	Ahmed *et al.* 2013
	35	55	10	

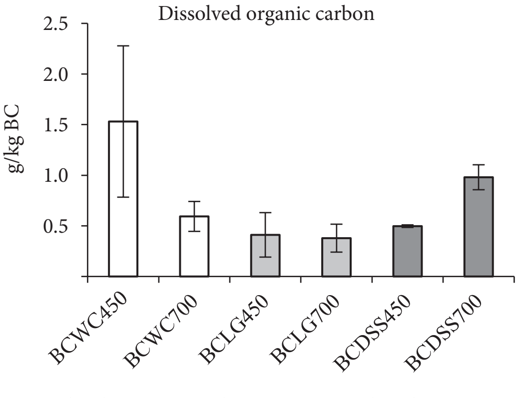


Fig. 1. Dissolved organic carbon content in biochars (BCs) determined via batch method. Values are shown as the mean of two observations (n = 2) ± standard deviation. BCs at 450 and 700 °C from wood chips, lignin and digested sewage sludge are BCWC450, BCWC700, BCLG450, BCLG700, BCDSS450, and BCDSS700, respectively

The values of Cd, Cu, and Pb concentration were input as measured in the BC eluates (Figs S1–3 in supplemental material). Regarding the solute type adopted for the modelling, the concentration in BC eluates of trace metals in soluble form (syringe filtered water, 0.45 µm pore size) was identified as total dissolved concentration.

2. Results and discussion

2.1. Dissolved organic carbon in a water extract of biochars via batch test method

Figure 1 shows the results of the DOC analysis in the water extract of BCs via the batch test method. Several studies have observed that wood and woody biomass feedstock tend to release less DOC than non-wood biomass feedstock (Mukherjee, Zimmerman 2013; Alburquerque *et al.* 2014; Graber *et al.* 2014).

At the pyrolysis temperature of 450 °C, BCWC450 showed the highest DOC content which was about 3 and 4 times higher than the DOC contents of both BCLG450 and BCDSS450. At the pyrolysis temperature of 700 °C, BCDSS700 exhibited the highest DOC content, which was about 2 and 3 times higher than the DOC contents of both BCWC700 and BCLG700.

With regard to the observed differences in DOC content of BC from different types of feedstock, transformations of organic structure into recalcitrant forms (Wu *et al.* 2011) as well as secondary reactions (Lin *et al.* 2012) occurring during pyrolysis process may have resulted in lowering the content of DOC in the batch extracts of BCWC and BCLG.

During the pyrolysis process, physical alterations (e.g., formation of larger pores and increases in surface area) occurring in the char matrix (Liaw, Wu 2015) or volatilisation of organic volatiles (Zhang *et al.* 2015) could lead to opening blocked pores, resulting in a higher concentration of DOC in the batch extracts of BCDSS700.

The DOC temperature trend varied significantly ($p < 0.05$) depending on the feedstock. With the increase in pyrolysis temperature, BCWC and BCLG showed a significant ($p < 0.05$) decrease in DOC concentration in the water extract by about 61 and 7%, respectively. A similar trend in a decrease in the amounts of DOC with an increase in pyrolysis temperatures has been reported by several authors over a wide range of biomass feedstock and pyrolysis temperatures (Gaskin *et al.* 2008; Mukherjee, Zimmerman 2013; Graber *et al.* 2014; Smith *et al.* 2016). Conversely, the amount of DOC in BCDSS increased by 96% with the increase in pyrolysis temperature. The observed trend was inconsistent with the results reported by Zhang *et al.* (2015), who noted a decrease in DOC content associated with each increase in temperature of 100 °C in BC from sewage sludge pyrolysed in the range 300–900 °C.

The observed differences in the DOC temperature trend may be related to differences in molecular weight distribution of DOC between the different types of BC. With increases in pyrolysis temperatures the DOC content of BC decreases because of the decomposition of DOC with a lower molecular weight (Luo *et al.* 2015), resulting in a lower concentration of DOC in the batch extracts of BC from wood chips and lignin at 700 °C. The observed increase in DOC content of BC from digested sewage sludge with an increase in pyrolysis temperature may result from the thermo-stable behaviour of DOC with higher molecular weight and the concurrent decrease in BC yield (Luo *et al.* 2015).

2.2. Results of the up-flow percolation test method

Due to time and resource constraints, DOC measurements were taken only in the eluates collected from the up-flow percolation test performed on BCWC450, BCWC700, BCLG450, and BCLG700. These BCs were selected for

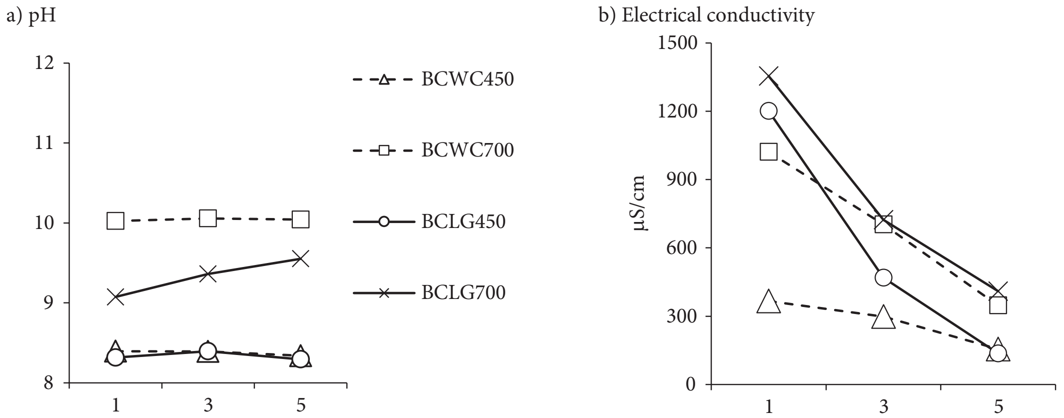


Fig. 2. Mean values of (a) pH and (b) electrical conductivity of biochar (BC) eluates collected at various liquid to solid (L/S) ratios. Values are shown as the mean of two observations (n = 2). Error bars of each data point would be smaller than size of symbol. DW – dry weight. BCs at 450 and 700 °C from wood chips and lignin are BCWC450, BCWC700, BCLG450, and BCLG700, respectively

testing DOC leaching behaviour because of the TC analysis results reported in a previous study by Mancinelli *et al.* (2016). These authors measured the highest TC content in BCWC and BCLG for both the applied pyrolysis temperatures of 450 and 700 °C which were between 54 and 68%, respectively, higher than the content of TC in BCDSS.

The pH and EC of the eluates collected from BCWC and BCLG up-flow percolation test increased significantly ($p < 0.05$) with increases in pyrolysis temperatures (Fig. 2). For each feedstock type, EC values of the eluates significantly ($p < 0.05$) decreased with increases in liquid to solid ratios. EC provides information about the presence of total soluble salts in the eluate. For all the tested BCs, with increasing volume leached through the columns, BCs released decreasing quantities of soluble salts.

For each feedstock type, pH values of the eluates showed significant ($p < 0.05$) differences for liquid to solid ratios and pyrolysis temperatures. BCWC showed the highest rise in alkalinity, with the pH of eluates from BCWC700 being between 1.6 and 1.7 units higher with each increase in liquid to solid ratio compared to the respective pH levels from BCWC450. The pH levels of the eluate samples from BCLG700 were between 0.8 and 1.3 units higher than the respective levels from BCLG450. For all the liquid to solid ratios, BCs at 450 °C from wood chips and lignin showed a similar alkaline pH in the range 8.3–8.4.

Figure 3 shows the DOC concentrations determined in the eluates collected at various liquid to solid ratios in the up–flow percolation test of BCWC and BCLG. Irrespective of the temperature adopted for pyrolysis treatment, BCWC and BCLG showed a similar release trend characterised by a significant ($p < 0.05$) decrease in DOC concentrations of the eluates collected at increasing liquid to solid ratios.

An increase in L/S ratio from 1 to 3 l/kg DW produced the highest decreases in the DOC concentrations of the eluates collected from BCLG700 (50%) and BCWC450 (49%), followed by BCLG450 (30%) and BCWC700 (21%). A further increase in L/S ratio from 3 to 5 l/kg DW showed a substantial decrease (71%) in the DOC concentration of the eluate collected from BCLG450, followed by BCWC700 (49%), BCWC450 (18%), and BCLG700 with the slightest decrease (1%). In the works by Beesley and Marmiroli (2011) and Iqbal *et al.* (2015), DOC was observed to be rapidly released from BC with no substantial amount of DOC recorded after a few eluate samples.

Low temperature (450 °C) BCLG and BCWC significantly (p < 0.05) released greater amounts of DOC compared with the respective high temperature (700 °C) BC. A decrease in DOC content with an increase in pyrolysis temperature was reported in a previous study on BC leaching trends by Mukherjee and Zimmerman (2013). A decrease in water leachability of organic matter (quantified as total organic carbon as percentage of TC) in BC is related to the transformation of organic structure into recalcitrant forms occurring during pyrolysis (Wu *et al.* 2011).

In order to analyse the fraction of carbon that is released from BC, the DOC content of BCs was expressed as a percentage of the respective TC content (Table 2).

With regard to the comparison between the DOC results of the batch test and the TC content of biochars, our results were comparable with those reported by Jones *et al.* (2011), who calculated DOC in water extracts of BC from mixed hardwood at 450 °C, which was 0.04% of the TC content of BC. We calculated the highest percentage of DOC for BCWC450 (0.24% TC), followed by BCDSS450 (0.17% TC), and BCLG450 (0.05% TC).

Regarding the up-flow percolation test, the calculated cumulative released quantity of DOC at the cumulative liquid to solid ratio of 9 l/kg DW was highest for BCWC450 (0.09% of the TC content), followed by BCWC700 (0.07% of the TC content), BCLG450 (0.06% of the TC content), and BCLG700 (0.02% of the TC content) (Table 2).

For both pyrolysis temperatures, BCWC exhibited a higher tendency to release DOC compared with BCLG. The cumulative released quantity of DOC of BCWC expressed as a percentage of TC content was 1.5 times higher at 450 °C and 3.5 times higher at 700 °C than the respective quantity of DOC calculated for BCLG. In the work by Li *et al.* (2014), BC from wood and lignin pyrolysed at 400 and 600 °C had different surface properties, with BC from wood exhibiting higher micropore structure (specific surface area, micropore area and volume) compared with BC from lignin at both pyrolysis temperatures. Closed or blocked pores that were formed in BC during pyrolysis open due to physical rearrangement occurring during thermochemical treatment (e.g., partial steam gasification)

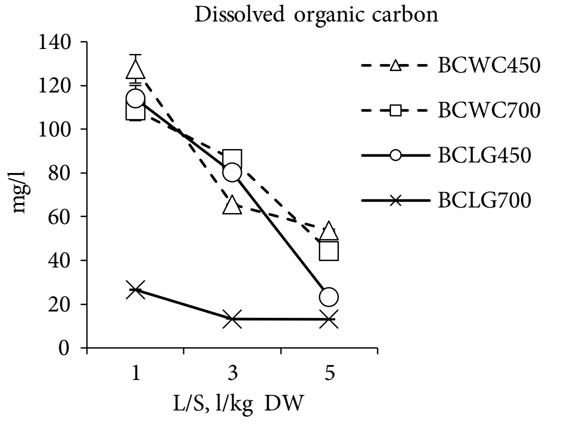


Fig. 3. Mean values of dissolved organic carbon (DOC) of biochar (BC) eluates collected at various liquid to solid (L/S) ratio. Values are shown as the mean of two observations ($n = 2$) ± standard deviation. DW – dry weight. BCs at 450 and 700 °C from wood chips and lignin are BCWC450, BCWC700, BCLG450, and BCLG700, respectively

Table 2. Dissolved organic carbon (DOC) content in biochars (BCs) determined via batch test and up-flow percolation test expressed as a percentage of the respective total carbon (TC) content

	Batch test		Up-flow percolation test			
	TC[1], g/kg BC	Amount of DOC as a % of TC	Amount of DOC as a % of TC			Cumulative amount of DOC as a % of TC
		(1:20) w:v	L_1/S	L_2/S	L_3/S	$\Sigma L_i/S$
BCWC450	629.2 ±44.1	0.24 ±0.14	0.02 ±0.0	0.03 ±0.0	0.04 ±0.0	0.09
BCWC700	876.9 ±67.2	0.07 ±0.02	0.01 ±0.0	0.03 ±0.0	0.03 ±0.0	0.07
BCLG450	839.2 ±19.0	0.049 ±0.03	0.01 ±0.0	0.03 ±0.0	0.01 ±0.0	0.06
BCLG700	717.4 ±49.2	0.053 ±0.02	0.00 ±0.0	0.01 ±0.0	0.01 ±0.0	0.02
BCDSS450	290.4 ±2.2	0.17 ±0.00	N.D.	N.D.	N.D.	N.D.
BCDSS700	279.4 ±1.8	0.35 ±0.04	N.D.	N.D.	N.D.	N.D.

[1] Values of total carbon (TC) concentration in the studied BCs were determined in a study by Mancinelli *et al.* (2016)

BCs at 450 and 700 °C from wood chips, lignin and digested sewage sludge are BCWC450, BCWC700, BCLG450, BCLG700, BCDSS450, and BCDSS700, respectively

DOC – dissolved organic carbon. DW – dry weight. N.D. – not determined. w:v –weight to volume ratio. L_i/S – liquid to solid (L/S) ratio equal to 1, 3, and 5 *l*/kg DW for *i* equal to 1, 2, and 3, respectively. $\Sigma L_i/S$ – cumulative L/S ratio

(Liaw, Wu 2015) or due to the swelling of BC in water (Ahmad *et al.* 2014). Therefore, organic matter, which was entrapped in the char matrix, may become more accessible to leaching. It is likely that the leaching condition applied in the up-flow percolation test enhanced the swelling of BCWC in water, thus resulting in more DOC as a percentage of TC released with increasing liquid to solid ratios compared with BCLG.

As regards the temperature-related trend of the cumulative released quantity of DOC expressed as a percentage of the respective TC content, with an increase in pyrolysis temperature BCWC and BCLG showed a decrease of about 22 and 67%, respectively (Table 2).

With an increase in pyrolysis temperature, results of the up-flow percolation test and batch test showed a similar trend for BCWC with a decrease by about 22 and 71%, respectively (Table 2). Comparison of batch and up-flow percolation test results showed an opposite trend for BCLG, with an increase by about 8% and a decrease by about 67%, respectively (Table 2).

2.3. Trace metal speciation biochar eluates predicted by WHAM

Trace metal speciation predictions were obtained assuming various active DOC fractions of FA (0.55–0.90) and HA (0–0.1) covering a range of assumptions reported in the literature about WHAM modelling for trace metal speciation in waters regarding active and inert fractions of DOM.

Speciation model results of Cd, Cu and Pb showed their affinity to form complexes in all the eluates collected at various L/S ratios from BCWC and BCLG, being the sum of the fractions of these trace metals bound to colloidal FA and HA equal to the respective total dissolved concentrations for all the assumptions taken in consideration for WHAM modelling (i.e., various fractions of DOC considered as active FA and HA). According to Unsworth *et al.* (2006), Fe, Al, and Mn can compete with Cd, Cu, and Pb for binding with HA and FA, thus affecting their speciation.

The high proportion of metal-FA and metal-HA complexes predicted for all the modelled eluate types may be due to the observed characteristics, such as alkaline pH levels, high DOC concentrations and the absence of elements that can compete for binding with HA and FA, such as major cations (i.e., Ca^{2+} and Mg^{2+}), Fe, Al, and Mn.

Comparisons between the free ion activity of Cd, Cu, and Pb considering 35% inert fractions of DOC and no inert fractions of DOC (Table 1) showed between 2.3 and 3 times higher free ion activity of the analysed trace metals (Figs 4–6).

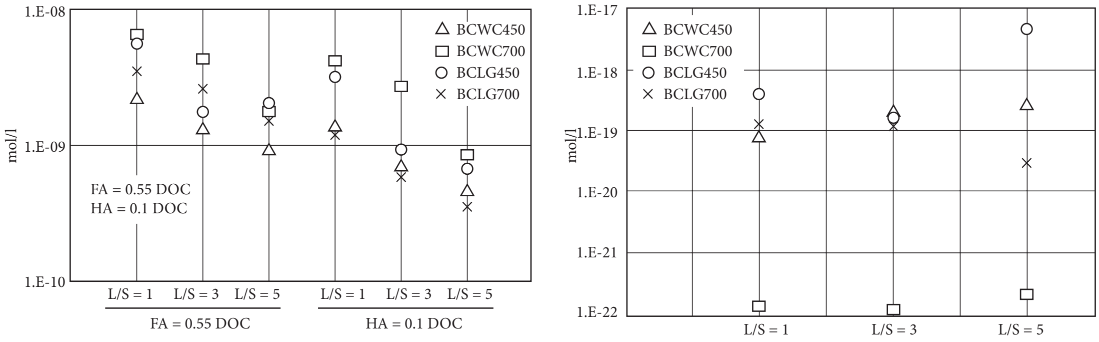
Regarding the free ion activity of the analysed trace metals considering 35% DOC as inert fractions (Table 1), the modelling results of the eluates collected from BCWC and BCLG exhibited a different trend. Regardless of the L/S ratios, the modelling results of the eluates collected from BCWC exhibited higher free ion activity for Cd (between 11.4 and 23.3%), Cu (between 13.6 and 25.8%), and Pb (between 11.6 and 23.3%) considering 65% DOC acting as active FA compared to 55% DOC acting as active FA and 10% DOC acting as active HA.

Except for the eluate collected from BCLG700 at the final L/S ratio, in all the eluates collected from BCLG, the free ion activity was higher for Cd (between 0.8 and 16.8%), Cu (between 3 and 22.4%), and Pb (between 1 and 16.9%) considering 65% DOC acting as active FA compared to 55% DOC acting as active FA and 10% DOC acting as active HA. Conversely, the eluate collected from BCLG700 at the final L/S ratio showed a decrease by about 3.4–3.7 times in free ion activity.

In the work by Chakraborty and Chakrabarti (2006), a decrease in electrical conductivity and hence in ionic strength in diluted mine effluents was associated with stronger bonds in metal-DOC complexes resulting from a higher electrostatic attraction between positively charged metal ions, such as Cd^{2+} and Cu^{2+}, and the negatively charged sites of DOC.

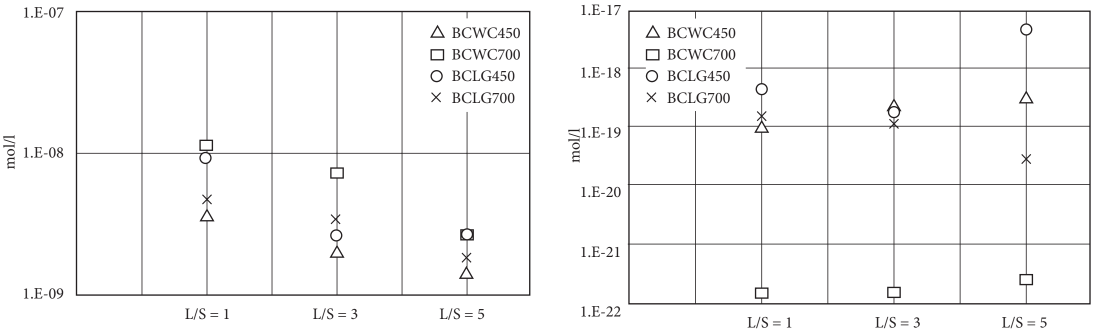
In the present study, pH values of all the BC eluates were in the alkaline range. Furthermore, electrical conductivity greatly decreased with increasing L/S ratios. In addition, for all the modelled eluate types a strong affinity of Cd, Cu, and Pb to form DOC complexes was predicted. Therefore, it is likely that the trace metals released from BCWC and BCLG formed a strong bond with DOC, thus not representing a risk of impairment for the receiving water bodies.

a) Cd bound to FA (0.55 DOC) and HA (0.10 DOC) in BC eluates b) Cd free ion activity in BC eluates



c) Cd bound to FA (0.65 DOC) in BC eluates

d) Cd free ion activity in BC eluates



e) Cd bound to FA (0.90 DOC) and HA (0.10 DOC) in BC eluates f) Cd free ion activity in BC eluates

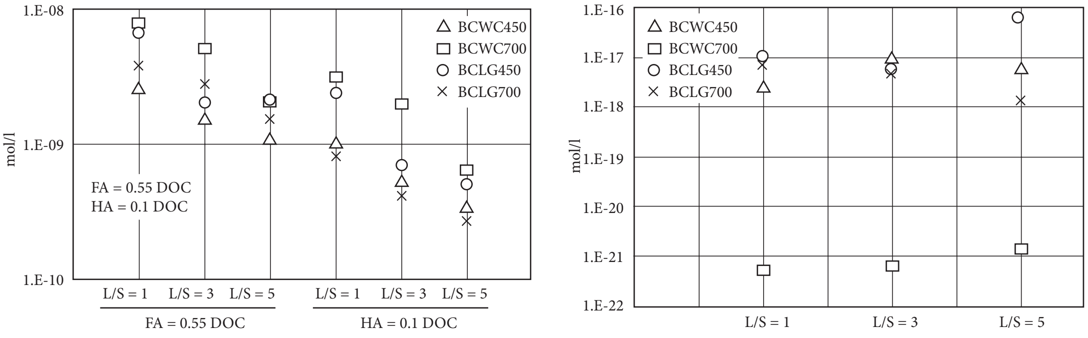


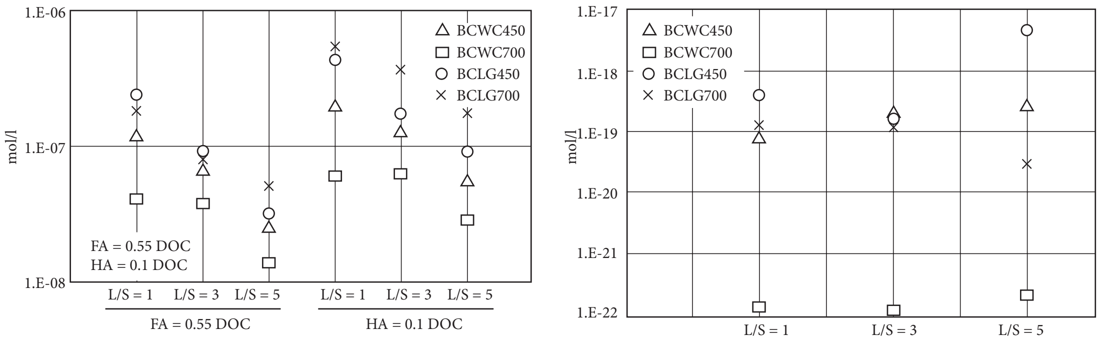
Fig. 4. Fraction bound to colloid fulvic acid (FA) and humic acid (HA) (a, c, e) and free ion activity (b, d, f) of Cd in biochar (BC) eluates collected at various liquid to solid (L/S) ratios, predicted by WHAM. DOC – dissolved organic carbon. BC at 450 and 700 °C from wood chips and lignin and are BCWC450, BCWC700, BCLG450, and BCLG700, respectively

Conclusions

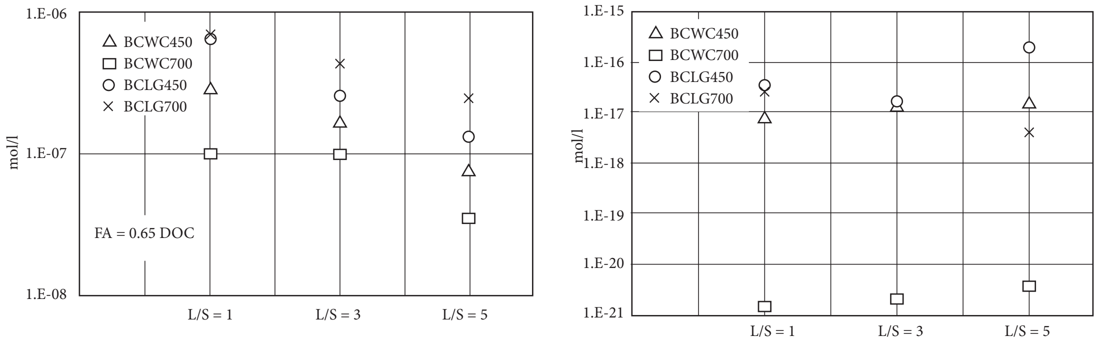
1. For each type of feedstock (i.e., wood chips (WC) and lignin (LG)), analysis of the up-flow percolation test showed the same temperature related trend in dissolved organic carbon (DOC) leachability from biochars (BCs), with high temperature (700 °C) BCs releasing lower cumulative amounts of DOC compared with low temperature (450 °C) BCs, which were in the range 0.02–0.07% and 0.06–0.09% of total carbon (TC) content, respectively. Under the adopted pyrolysis conditions, the increase in pyrolysis temperature from 450 to 700 °C was more effective in lowering DOC leachability from BCs from LG (BCLG) compared with BCs from WC (BCWC), with a decrease by about 67 and 22% in the cumulative released quantity of DOC expressed as a percentage of TC content, respectively.

2. Regarding the influence of feedstock selection for BC production on DOC leachability, for both pyrolysis temperatures, BCWC exhibited a tendency to release more DOC compared with BCLG, as it was the cumulative

a) Cu bound to FA (0.55 DOC) and HA (0.10 DOC) in BC eluates b) Cu free ion activity in BC eluates



c) Cu bound to FA (0.65 DOC) in BC eluates d) Cu free ion activity in BC eluates



e) Cu bound to FA (0.90 DOC) and HA (0.10 DOC) in BC eluates f) Cu free ion activity in BC eluates

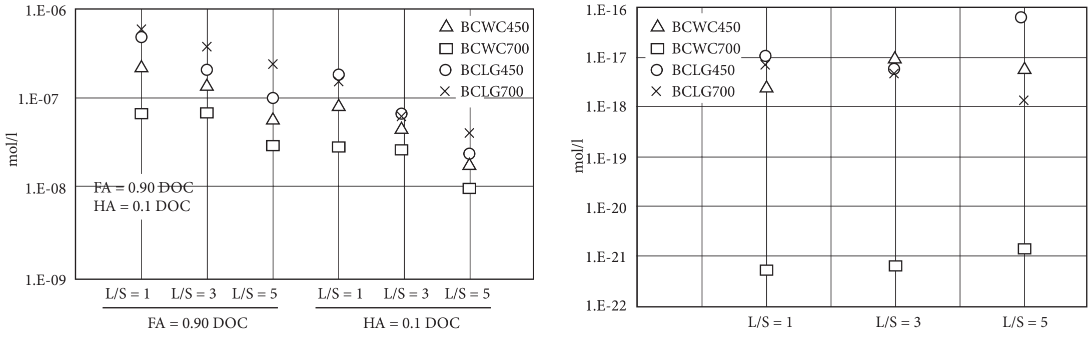


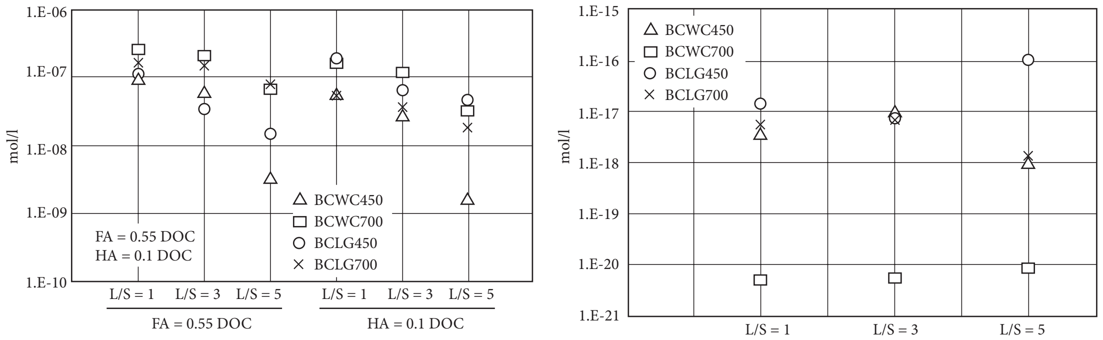
Fig. 5. Fraction bound to colloid fulvic acid (FA) and humic acid (HA) (a, c, e) and free ion activity (b, d, f) of Cu in biochar (BC) eluates collected at various liquid to solid (L/S) ratios, predicted by WHAM. DOC – dissolved organic carbon. BC at 450 and 700 °C from wood chips and lignin and are BCWC450, BCWC700, BCLG450, and BCLG700, respectively

released quantity of DOC of BCWC expressed as a percentage of TC content 1.5 times higher at 450 °C and 3.5 times higher at 700 °C than the respective quantity of DOC calculated for BCLG. It is likely that the leaching condition applied in the up-flow percolation test enhanced the swelling of BCWC in water by opening closed or blocked pores, thus increasing accessibility to leach the organic matter that was entrapped in the char matrix during the pyrolysis process.

3. Trace metal speciation modelling results showed the predominance of fulvic acid (FA) and humic acid (HA) complexes of Cd, Cu, and Pb in all the eluates collected at various liquid to solid (L/S) ratios from BCWC and BCLG irrespective of the inert fractions (0 and 35%) of DOC or the different fractions of active FA (55, 65, 90% DOC) or HA (0 and 10% DOC) considered. The high proportion of metal-FA and metal-HA complexes predicted for all the modelled eluate types may be due to the observed characteristics, such as alkaline pH values, high DOC concentrations and the absence of elements that can compete for binding with HA and FA.

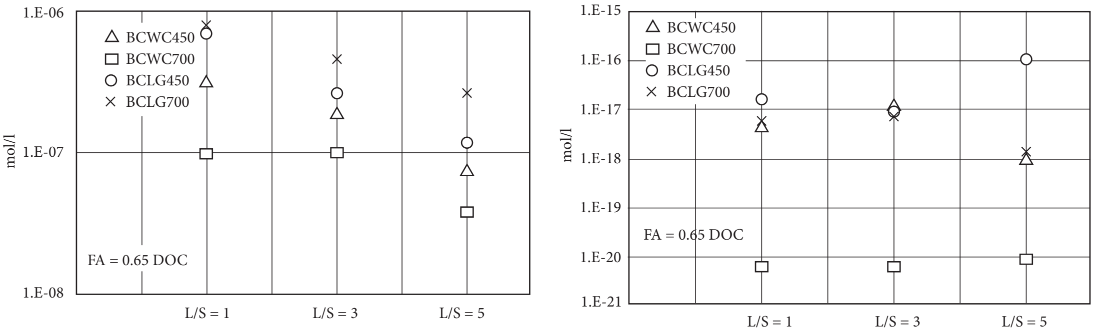
a) Pd bound to FA (0.55) and HA (0.10) in BC eluates

b) Pd free ion activity in BC eluates



c) Pd bound to FA (0.65) in BC eluates

d) Pd free ion activity in BC eluates



e) Pd bound to FA (0.90) and HA (0.10) in BC eluates

f) Pd free ion activity in BC eluates

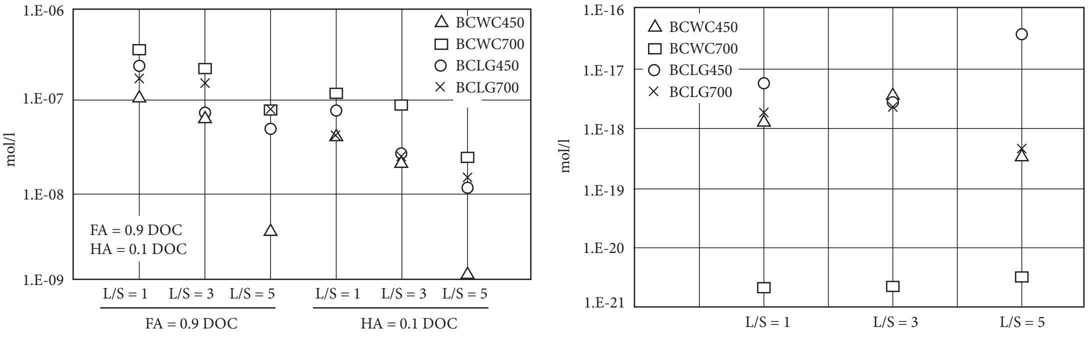


Fig. 6. Fraction bound to colloid fulvic acid (FA) and humic acid (HA) (a, c, e) and free ion activity (b, d, f) of Pd in biochar (BC) eluates collected at various liquid to solid (L/S) ratios, predicted by WHAM. DOC – dissolved organic carbon. BC at 450 and 700 °C from wood chips and lignin and are BCWC450, BCWC700, BCLG450, and BCLG700, respectively

4. If BCWC or BCLG are intended for water application, such as a filter media, the amount of DOC that may be emitted in surface water as a result of BC release in order to avoid high load of BC-borne DOC should be carefully estimated. It is likely that the trace metals released from BCWC and BCLG formed a strong bond with DOC, thus not representing a risk of impairment for the receiving water bodies.

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