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REVIEW: DISSOLVED ORGANIC CARBON CONTENT OF BIOCHAR VARYING WITH THE TYPE OF FEEDSTOCK AND THE PYROLYSIS TEMPERATURE

Enrico MANCINELLI[1], Edita BALTRĖNAITĖ[2], Pranas BALTRĖNAS[3],
Raimondas GRUBLIAUSKAS[4], Giorgio PASSERINI[5]

[1, 5]*UNIVPM, Department of Industrial Engineering and Mathematical Sciences*
[2, 4]*VGTU, Faculty of Environmental Engineering, Department of Environmental Protection*
[3]*VGTU, Institute of Environmental Protection*

E-mails: [1]e.mancinelli@pm.univpm.it; [2]edita.baltrenaite@vgtu.lt; [3]pranas.baltrenas@vgtu.lt;
[4]raimondas.grubliauskas@vgtu.lt; [5]g.passerini@univpm.it

Abstract. The present review aims at investigating the influence of the type of feedstock and the production temperature on the dissolved organic carbon (DOC) content of biochar (BC) from slow pyrolysis. To collect data from the literature, peer reviewed articles in English published in 2007–2016 were considered. The different types of BC were classified depending on the fractions of cellulose and lignin and the type of feedstock. A linear regression (R-squared = 0.5) of the mean values of DOC content (g/kg) of BC was calculated in the range of 350–800 °C with slope (–0.005) and intercept (4.1) significant at $p < 0.05$. Irrespective of the type of feedstock, slow pyrolysis with temperatures above 500 °C would be a proper choice for limiting the mean DOC content of BC to values in the range of 0.28–1.01 g/kg.

Keywords: biochar, dissolved organic carbon content, slow pyrolysis, lignin, cellulose, manure, waste.

Introduction

Dissolved organic carbon (DOC), or water soluble organic carbon, or water extractable organic carbon represents the labile fraction of organic carbon in biochar (BC) that is prone to leaching. For the sake of simplicity, the term DOC is going to be used throughout this review.

Several studies have reported an influence of feedstock selection and pyrolysis conditions (i.e. heating rate, residence time and temperature) on the content, composition as well as on the extent to which DOC is released from BC (Lin *et al.* 2012; Mukherjee, Zimmerman 2013; Alburquerque *et al.* 2014; Graber *et al.* 2014).

Increasing pyrolysis temperature has been generally reported to result in a decrease in DOC content of BC (Liu *et al.* 2015; Zhang *et al.* 2015; Smith *et al.* 2016). However, rising pyrolysis temperatures in the range of 350–450 °C have resulted in BC with higher DOC content (Graber *et al.* 2014; Luo *et al.* 2015).

A relatively limited fraction of carbon has been reported to be released from BC as DOC (Liu *et al.* 2016), representing between 0.10 and 2.65% of the total carbon (TC) in the BC at 300 °C (Liu *et al.* 2015), between 0.04 and 0.24% of the TC in the BC at 450 °C (Mancinelli *et al.* 2017; Jones *et al.* 2011), between 0.1 and 0.4% of the TC in the BC at 650 °C (Mukherjee, Zimmerman 2013), and between 0.016 and 0.35% of the TC in the BC at 700 °C (Mancinelli *et al.* 2017).

To limit the content of DOC in BC, Smith *et al.* (2016) has suggested to adopt pyrolysis temperatures above 400 °C.

The environmental implications of BC-derived DOC have not been well elucidated, and further studies are on demand (Luo *et al.* 2015). BC-derived DOC may increase metal mobility through complexation as well as alter redox reactions and speciation of metals (Qu *et al.* 2016). In the work by Beesley *et al.* (2010), Cu concentration in soil pore water was related to changes in DOC concentration following BC application to soil. A ten-fold increase (from 3 to 30 mg C/l) in the concentration of dissolved organic matter (DOM) from BC resulted in an increase in Cr(VI) reduction from 20 to 100% in the ice phase and from 2.5 to 83% in the aqueous phase (Dong *et al.* 2014).

Table 1. Resume of the characteristics of the slow pyrolysis processes reported in the articles retrieved from the literature following the above-mentioned search criteria

	Temperature, °C	Heating rate, °C/min	Residence time, h	Pressure	References
Slow pyrolysis	350–900	2.5–30	0.25–48	N.R./atmospheric	See Fig. 1

N. R. – not reported

Reduction in carbon sequestration is a potential negative effect associated with leachability of organic matter from BC utilized in land application as soil amendment or as carbon sequestration agent (Wu *et al.* 2011; Liu *et al.* 2015; Qu *et al.* 2016).

The aims of the present review were:

- to investigate the influence of the type of feedstock and production temperature on the DOC content of BC from slow pyrolysis;
- to assess the temperature trend of DOC content of BC from slow pyrolysis.

Given the potential environmental implications of DOC deriving from BC applications, understanding the parameters (i.e. feedstock type and pyrolysis temperature) that affect the amount of DOC released by BC could be an effective tool for designing ad hoc BC production and application.

Methods

In the present review, DOC from BC is operationally defined as the organic carbon determined after filtering the biochar extract through a filter with pore size ≤ 0.45-μm.

For BC from slow pyrolysis of biomass, we considered BC produced in the range of 350–1000 °C in a low-oxygen thermal process (EBC European Biochar Certificate 2015) characterised by a low heating rate (up to 100 °C/s) and a residence time of hours (Spokas *et al.* 2011) (Table 1).

BC has been produced from a large variety of types of feedstock. BC classification depending on the feedstock was adapted from Aller (2016) and fell in the following categories:

- BC from lignin-rich feedstock (e.g. lignin, tree part, wood, sawdust, almond and cacao shells, and olive stones);
- BC from cellulose-rich feedstock (e.g. cellulose, grasses, straws, grains, stalks, peanut hulls and shells, sugarcane leaves and bagasse, and maize silage);
- BC from manure and waste (e.g. manures, bio-solids, green waste (e.g. mushroom residue, greenhouse waste, and olive pomace)).

With the aim of collecting data related to the present review, the search platform Engineering Village^{TM} (Elsevier, Netherlands) having access to 12 engineering literature and patent databases was used. The search criteria were adopted following Gurwick *et al.* (2013):

- articles published in peer reviewed journals in the period from 2007 to 2016, including relevant articles cited by recent studies or published in the afore mentioned time span;
- articles published in languages other than English were discarded;
- if either the pyrolysis parameters (e.g. temperature, heating rate or residence time) or the type of thermochemical process (e.g. slow pyrolysis) for producing BC were not clearly reported, data were not considered for calculations;
- for the search query the following terms were entered: “biochar”, “dissolved organic carbon”, “water soluble organic carbon”, “water extractable organic carbon”;
- for determining DOC content of BC, we considered only data referring to samples obtained through one-step extraction or stirring methods filtered through a filter with pore size ≤ 0.45-μm.

As for the 29 articles retrieved from the literature following the above-mentioned search criteria, the exact temperature (86%), residence time (72%) and pressure/atmosphere (59%) were the most reported characteristics of pyrolysis process, whereas the heating rate (24%) was the least reported parameter.

If DOC value was reported as carbon (C) mmol/l, the DOC concentration (mg/l) was calculated multiplying the DOC values expressed as C mmol/l by the average atomic weight of carbon (12.0107 g/mol) obtained from the average atomic mass of the isotopes of carbon given in the periodic table.

As for DOC analysis of BC via batch test method, if the measured concentration of DOC in the samples (C) were reported as mg/l, the DOC content (g/kg) of BC was calculated considering the mass (M, g) of BC and the volume (V, l) of extractant reported in the materials and

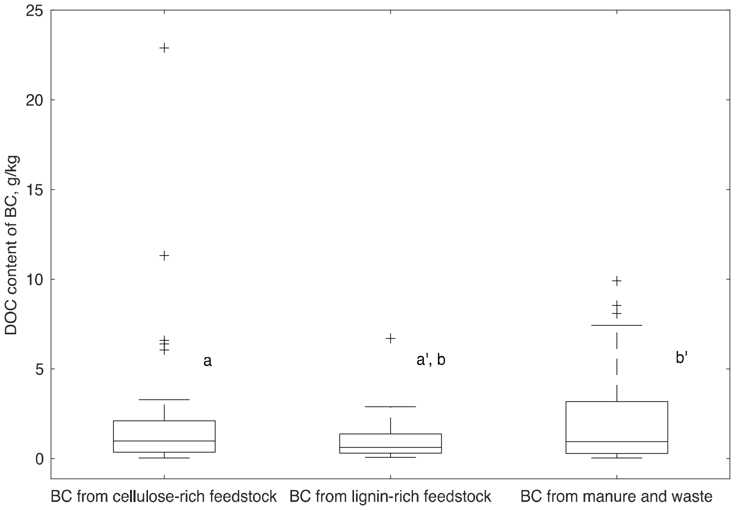

Fig. 1. Box-and-whisker plot for dissolved organic carbon (DOC) content of biochar (BC) from slow pyrolysis (350–900 °C) of cellulose-rich feedstock, of lignin-rich feedstock, and of manure and waste. Letters with a punctuation mark denote significant differences at $p < 0.05$ with the mean of the group with same letter with no punctuation mark. n = 109

Box-and-whisker plots show median (i.e. central line), upper (25%) and lower (75%) quantile (i.e. lower and upper bounds of the box, respectively) of the data, maximum and minimum values outside the central box that are not outliers (vertical lines), and outliers (cross symbols)

Data were retrieved from the following sources: Alburquerque *et al.* 2014; Baltrėnaitė *et al.* 2016; Cabrera *et al.* 2011; Cabrera *et al.* 2014; Gaskin *et al.* 2008; Graber *et al.* 2014; Hass *et al.* 2012; Heitkötter, Marschner 2015; Jeong *et al.* 2016; Jones *et al.* 2011; Khan *et al.* 2015; Liu *et al.* 2015; Lou *et al.* 2015; Lu *et al.* 2014; Mancinelli *et al.* 2017; Mukherjee, Zimmerman 2013; Peng *et al.* 2016; Rodríguez-Vila *et al.* 2016; Sáez *et al.* 2016; Smebye *et al.* 2016; Smith *et al.* 2016; Spokas *et al.* 2014; Sun *et al.* 2016; Tang *et al.* 2016; Yue *et al.* 2016; Zeng *et al.* 2015; Zhang *et al.* 2015; Zhang *et al.* 2016; Zheng *et al.* 2012

methods sections of the retrieved articles using the following formula:

$$DOC = \frac{V \times C}{M}. \quad (1)$$

Statistical analysis was performed using MATLAB R2016b (Mathworks, USA). The descriptive statistics of the collected data were defined by calculating mean, maximum, minimum, standard deviation, and coefficient of variance.

To test for the null hypothesis that data come from a normal distribution, the Chi-square goodness of fit tests for a normal distribution at the 5% significance level were calculated for the data grouped depending on the pyrolysis temperature or the type of feedstock. The Levene's test was performed at the 0.05 significance level for homogeneity of variance between the groups of data (e.g. DOC content of BC depending on the pyrolysis temperature or the type of feedstock) versus the alternative hypothesis that at least one group has a different variance.

Welch's t-test evaluated for the groups of data with unequal variances for the null hypothesis that two groups have equal means at 5% significance level.

To evaluate the temperature-related trend of DOC content of BC, DOC contents of BC were plotted versus the respective pyrolysis temperatures and a relation was determined by step-wise linear regression with slope and intercept considered significant at $p \leq 0.05$. The relation between pyrolysis temperatures and the mean values of DOC content, that were calculated for each temperature with $n \geq 2$, was determined by step-wise linear regression with slope and intercept considered significant at $p \leq 0.05$.

Results and discussion

Figure 1 shows box-and-whiskers plot of the DOC content of BC from slow pyrolysis grouped depending on the types of feedstock.

Differences between the mean DOC content of lignin-rich (0.92±1.06 g/kg) and of cellulose-rich (2.31± 4.14 g/kg) feedstock, and of lignin-rich feedstock and of manure and waste (2.40±3.06 g/kg) were statistically significant at $p < 0.05$. The high coefficient of variance (>100%) calculated for the means of all the three groups of feedstocks may be due to the wide range of pyrolysis temperatures considered, specifically, BC from cellulose rich feedstock (400–750 °C), BC from lignin-rich feedstock (350–800 °C), and for BC from manure and waste (350–900 °C).

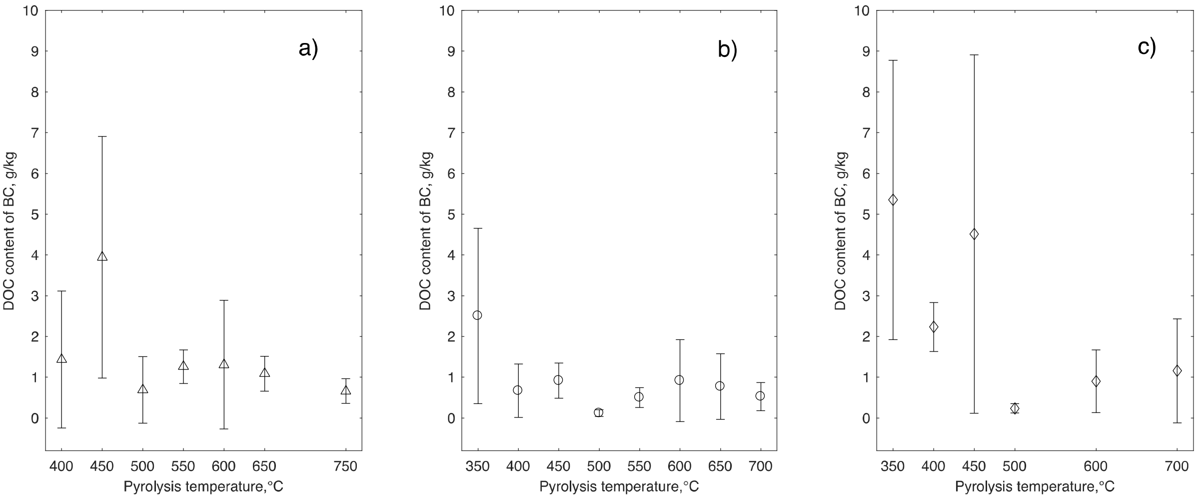

Fig. 2. Mean dissolved organic carbon (DOC) content of biochar (BC) from slow pyrolysis of cellulose-rich feedstock (a), of lignin-rich feedstock (b), and of manure and waste (c). Error bars show standard deviation. n = 33 (a). n = 42 (b). n = 22 (c)

Data were retrieved from the following sources: Alburquerque *et al.* 2014; Baltrėnaitė *et al.* 2016; Gaskin *et al.* 2008; Graber *et al.* 2014; Hass *et al.* 2012; Heitkötter, Marschner 2015; Jeong *et al.* 2016; Jones *et al.* 2011; Khan *et al.* 2015; Liu *et al.* 2015; Lu *et al.* 2014; Mancinelli *et al.* 2017; Mukherjee, Zimmerman 2013; Peng *et al.* 2016; Rodríguez-Vila *et al.* 2016; Sáez *et al.* 2016; Smebye *et al.* 2016; Smith *et al.* 2016; Spokas *et al.* 2014; Tang *et al.* 2016; Yue *et al.* 2016; Zeng *et al.* 2015; Zhang *et al.* 2015; Zheng *et al.* 2012

Mean DOC content of BC from manure and waste (Fig. 2c) was the highest (2.24–5.35 g/kg) in the temperature range of 350–450 °C. Mean DOC contents of BC from manure and waste were significantly ($p < 0.05$) greater than the mean DOC contents of BC from lignin-rich feedstock at 400 and 450 °C whereas differences were not statistically significant between DOC contents of BC from manure and waste and from cellulose-rich feedstock for the range of 350–450 °C.

Mean DOC content of BC from cellulose-rich feedstock (Fig. 2a) was the highest (0.69–1.31 g/kg) in the temperature range of 500–650 °C. Mean DOC contents of BC from cellulose-rich feedstock did not significantly differ from the mean DOC contents of BC from manure and waste whereas differences were significantly ($p < 0.05$) different or greater than the mean DOC contents of BC from lignin-rich feedstock at 500 and 550 °C, respectively.

Mean DOC content of BC from lignin-rich feedstock (Fig. 2b) was the lowest in the temperature range of 350–550 °C (0.12–2.50 g/kg) as well as at 700 °C (0.52±0.35 g/kg).

Figure 3 shows a linear relation between DOC content of BC from slow pyrolysis and the temperatures in the range of 350–790 °C. Lower temperatures (<350 °C) were not considered as it would have resulted in a strong degree of scatter (data not shown). According to EBC (EBC European Biochar Certificate 2015), biochar is produced at temperatures ≥350 °C. However, different studies labelled as biochar the carbonaceous solid product of pyrolysis process at temperatures in the range of 250–300 °C (Liu *et al.* 2015; Zhang *et al.* 2015; Smith *et al.* 2016; Yue *et al.* 2016). The linear relation in Figure 3 does not fit for temperatures higher than 790 °C, as the BC produced at these temperatures would be associated with negative DOC content.

There was a weak correlation (R-squared = 0.1) of the data with the linear model with intercept (4.136) and slope (–0.005) significant at $p = 0.000065$, with a sufficient degree of scatter at temperatures in the range of 350–450 °C (Fig. 3).

The mean values of DOC content of BC decreased or increased with each 50 °C increase in temperature in the range of 350–550 °C (Fig. 4). This may be due to a higher influence of feedstock properties on biochar with a lower carbonization. Comparing the fractions of cellulose and lignin in the feedstock (i.e. wheat straw and pig manure) and the respective BC, Zhao *et al.* (2014) observed no fractions of cellulose for the BC produced in the range of 350–650 °C as well as an increase in the fraction of lignin for the BC produced in the range of 200–500 °C.

The mean values of DOC content of BC from slow pyrolysis decreased with increasing temperatures in the range of 600–800 °C (Fig. 4). The influence of feedstock selection for BC production on DOC content is highest for BC at lower temperatures (≤450 °C) compared with higher temperature (>600 °C) (Graber *et al.* 2014).

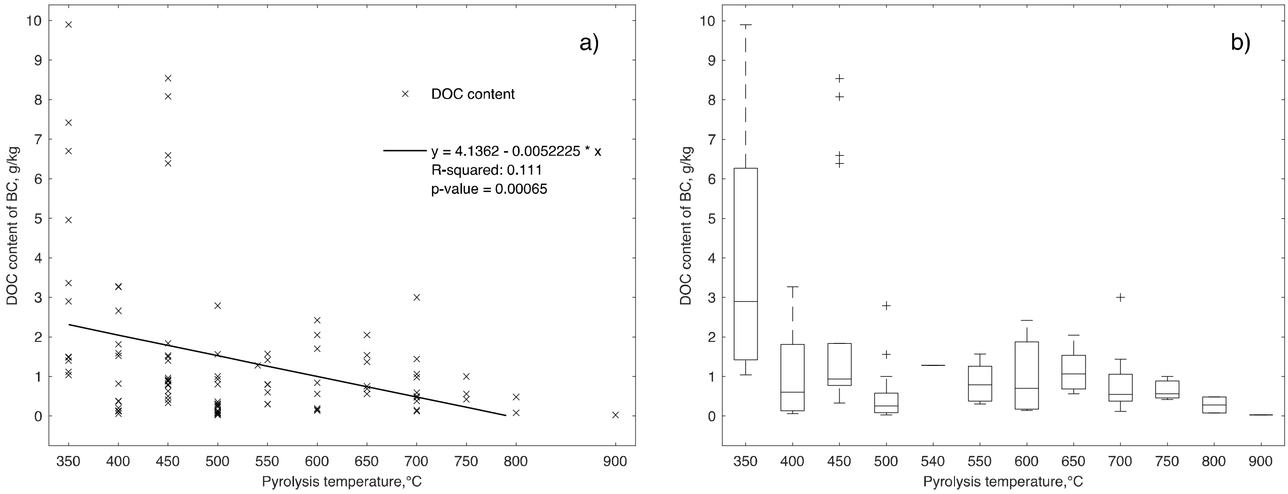

Fig. 3. Scatter plot (a) and box-and-whisker plot (b) for dissolved organic carbon (DOC) content of biochar (BC) from slow pyrolysis. n = 102

Linear regression (a) for the values of DOC content of BC in the temperature range of 350–790 °C with slope and intercept significant at $p \leq 0.05$

Box-and-whisker plots (b) show median (i.e. central line), upper (25%) and lower (75%) quantile (i.e. lower and upper bounds of the box, respectively) of the data, maximum and minimum values outside the central box that are not outliers (vertical lines), and outliers (cross symbols)

Data were retrieved from the following sources: Alburquerque *et al.* 2014; Baltrėnaitė *et al.* 2016; Cabrera *et al.* 2011; Gaskin *et al.* 2008; Graber *et al.* 2014; Hass *et al.* 2012; Heitkötter, Marschner 2015; Jeong *et al.* 2016; Jones *et al.* 2011; Khan *et al.* 2015; Liu *et al.* 2015; Lu *et al.* 2014; Mancinelli *et al.* 2017; Mukherjee and Zimmerman 2013; Peng *et al.* 2016; Rodríguez-Vila *et al.* 2016; Sáez *et al.* 2016; Smebye *et al.* 2016; Smith *et al.* 2016; Spokas *et al.* 2014; Tang *et al.* 2016; Yue *et al.* 2016; Zeng *et al.* 2015; Zhang *et al.* 2015; Zheng *et al.* 2012

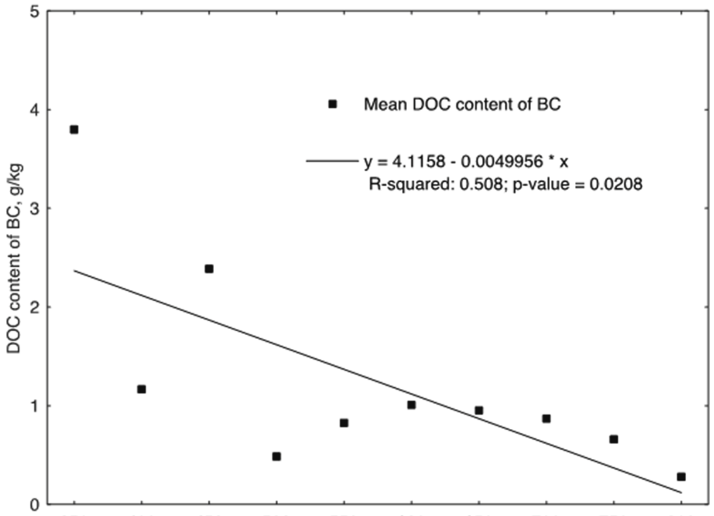

Fig. 4. Scatter plot and linear regression for the mean values of dissolved organic carbon (DOC) content of biochar (BC) from slow pyrolysis in the temperature range of 350–800 °C. n = 100

Data were retrieved from the following sources: Alburquerque *et al.* 2014; Baltrėnaitė *et al.* 2016; Gaskin *et al.* 2008; Graber *et al.* 2014; Hass *et al.* 2012; Heitkötter, Marschner 2015; Jeong *et al.* 2016; Jones *et al.* 2011; Khan *et al.* 2015; Liu *et al.* 2015; Lu *et al.* 2014; Mancinelli *et al.* 2017; Mukherjee, Zimmerman 2013; Peng *et al.* 2016; Rodríguez-Vila *et al.* 2016; Sáez *et al.* 2016; Smebye *et al.* 2016; Smith *et al.* 2016; Spokas *et al.* 2014; Tang *et al.* 2016; Yue *et al.* 2016; Zeng *et al.* 2015; Zhang *et al.* 2015; Zheng *et al.* 2012

At temperatures in the range 500–650 °C, the fraction of lignin of BC was characterised by a stable behaviour or a slight decrease compared to the lignin fraction of the feedstock (Zhao *et al.* 2014).

Secondary reactions as well as transformations of organic fractions (e.g. cellulose) into recalcitrant forms (e.g. lignin) (Wu *et al.* 2011; Lin *et al.* 2012; Zhao *et al.* 2014), occurring with rising pyrolysis temperatures (e.g. >350 °C) results in lowering the content of labile organic carbon in biochar that may be quantified as DOC.

Conclusions

1. The influence of the type of feedstock on the dissolved organic carbon (DOC) content of biochar from slow pyrolysis was investigated considering lignin-rich feedstock, cellulose-rich feedstock, and manure and waste. Differences among the mean DOC content of lignin-rich (0.92±1.06 g/kg) and of cellulose-rich (2.31±4.14 g/kg) feedstock, and of lignin-rich feedstock and of manure and waste (2.40±3.06 g/kg) were statistically significant at $p < 0.05$. However, the high coefficient of variance (>100%) calculated for the means of all the three groups of feedstock suggests that pyrolysis temperatures influenced the mean value of DOC content calculated considering only the type of feedstock and irrespective of the wide range of temperatures (350–900 °C).

2. Considering the temperature trend of the mean DOC content of biochar from the three types of feedstock, in the temperature range of 350–450 °C the mean DOC content of BC from manure and waste was the highest (2.24–5.35 g/kg). In the temperature range of 500–650 °C, the mean DOC content of BC from cellulose-rich feedstock was the highest (0.69–1.31 g/kg). Mean DOC content of BC from lignin-rich feedstock was the lowest in the temperature range of 350–550 °C (0.12–2.50 g/kg) and at 700 °C (0.52 g/kg).

3. A linear regression (R-squared = 0.5) of the mean values of DOC content (g/kg) of BC from slow pyrolysis was calculated in the range of 350–800 °C with slope (–0.005) and intercept (4.1) significant at $p < 0.05$. The application of this correlation for predictive purposes may better fit for pyrolysis temperatures above 450 °C because of a sufficient degree of scatter of the mean DOC content of BC at lower temperatures (350–450 °C).

4. Irrespective of the type of feedstock, DOC content of biochar from slow pyrolysis at temperatures between 500 and 800 °C was lower (0.28–1.01 g/kg) compared to DOC content of BC at temperatures in the range of 350–450 °C (1.17–3.8 g/kg). Therefore, slow pyrolysis with temperatures above 500 °C would be a proper choice for the applications of biochar in conditions that require caution in adding dissolved organic carbon to the pool of dissolved organic matter (e.g. remediation of metals-contaminated sites).

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Enrico Mancinelli dedicates this work in memory of beloved Lola.

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IŠTIRPUSIOS ORGANINĖS ANGLIES KIEKIO BIOANGLYJE, PAGAMINTOJE IŠ SKIRTINGŲ ŽALIAVOS TIPŲ, ESANT SKIRTINGOMS PIROLIZĖS SĄLYGOMS, LITERATŪROS APŽVALGA

E. Mancinelli, E. Baltrėnaitė, P. Baltrėnas, R. Grubliauskas, G. Passerini

Santrauka

Apžvalginiame straipsnyje nagrinėjama žaliavos tipo ir bioanglies gamybos temperatūros įtaka ištirpusios organinės anglies (IOA) kiekiui bioanglyje (BA), paruoštoje lėtosios pirolizės sąlygomis. Tyrimas paremtas literatūros duomenų iš recenzuojamų angliškų mokslinių straipsnių, kurie publikuoti 2007–2016 metų laikotarpiu, surinkimu ir apibendrinimu. Skirtingi bioanglies tipai buvo išskirti pagal celiuliozės ir lignino kiekį bei žaliavos tipą. Taikytas tiesinės regresijos modelis ($R^2 = 0,5$) ištirpusios organinės anglies kiekiui nustatyti bioanglyje, paruoštoje esant 350–800 °C temperatūrai. Nustatytos šios tiesinės regresijos charakteristikos: nuolydžio vertė –0.005, o sankirtos vertė 4.1, esant reikšmingumo vertei $p < 0.05$. Nepriklausomai nuo žaliavos tipo, lėtosios pirolizės sąlygos, esant temperatūrai, didesnei kaip 500 °C, būtų tinkamos ribojant ištirpusios organinės anglies kiekį bioanglyje iki 0,28–1,01 g/kg.

Reikšminiai žodžiai: bioanglis, ištirpusios organinės anglies kiekis, lėtoji pirolizė, ligninas, celiuliozė, mėšlas, atliekos.