

The Impact of Different Extraction Conditions on the Concentration and Properties of Dissolved Organic Carbon in Biochars Derived from Sewage Sludge and Digestates

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ABSTRACT

This study aimed to determine the quantity and quality of dissolved organic carbon (DOC) released from sewage sludge-derived biochar and digestion-derived biochar under different extraction conditions (deionised water, hot water, 0.1 mol·L⁻¹ NaOH) using TOC analyser, UV-vis spectroscopy. Biochars were produced through the pyrolysis process at temperatures of 400, 500, 600, and 800 °C. The objective of this article was to examine the influence of diverse extraction solutions on the amount of dissolved organic carbon (BDOC) released from biochars and to delineate alterations in the composition and characteristics of DOC contingent on the extraction parameters. The findings demonstrated that elevated pyrolysis temperatures resulted in a notable reduction in DOC concentration, with fractions extracted using NaOH exhibiting the highest DOC concentrations. SUVA₂₅₄ analysis and the E2/E3 ratio indicated that biochars produced at higher temperatures contained a greater proportion of aromatic and hydrophobic substances. These results indicate that pyrolysis temperature, feedstock type and extraction conditions are of significant importance for the properties of DOC in biochar. This has important implications for their potential applications in soil management and carbon sequestration strategies.

Keywords: biochar, dissolved organic carbon, extraction, SUVA₂₅₄.

INTRODUCTION

The utilization of biochar, a soil amendment abundant in carbon generated by the thermal decomposition of diverse forms of biomass, has been the subject of considerable interest due to the numerous benefits it offers in the context of agricultural practices and environmental sustainability. The study showcases its ability to augment the physicochemical and biological characteristics of soil, enhance nutrient retention, stimulate plant development, and alleviate climate change through the reduction of greenhouse gas emissions (Xu, 2022). The research demonstrates its capacity to enhance the physicochemical and biological attributes of soil, improve nutrient retention, promote plant growth, and mitigate climate change by reducing greenhouse gas emissions. These processes can result in the accumulation and subsequent

release of environmental contaminants, contingent on the biomass employed (Davis, 2023). In recent years, research on dissolved organic carbon derived from biochar (BDOC) has attracted considerable scientific interest due to its multifaceted impact on soil ecosystems and environmental processes (Feng et al., 2021). BDOC in biochars plays a pivotal role in soil ecosystems, influencing soil properties and microbial communities.

Dissolved organic carbon (DOC) found in soil constitutes a small yet significant portion of the overall carbon content, participating in a multitude of crucial interactions with flora, fauna, and inorganic substances. DOC consists of a myriad of compounds with diverse sizes, reactivity, and roles, all of which are essential in the processes of soil development. The level of reactivity exhibited by DOC towards mineral surfaces hinges on the types of functional groups present in individual

molecules, alongside the structural characteristics and composition of the soil solution. The process of DOC adsorption in soil is reversible, thereby facilitating the release of older DOC from the soil and its subsequent transport to groundwater and rivers (Goss and Oliver, 2023).

BDOC, the highly activated fraction of biochar carbon, plays a pivotal role in modifying soil microbial functioning, enhancing soil fertility, and influencing the mobility and toxicity of contaminants. The properties of BDOC are susceptible to alteration by a multitude of factors, including the pyrolysis temperature, the type of atmosphere employed, and the biomass feedstock from which the biochar is produced. BDOC can contain humus-like substances, affect the mobility of contaminants in the soil, and interact with soil microorganisms, thus impacting microbial functioning and enzymatic activity (Yue et al., 2023; Zhang et al., 2023; Azeem et al., 2023). Moreover, the DOC fraction in biochars is inherently unstable and exhibits greater susceptibility to photodegradation and biodegradation than bulk biochar (Lee et al., 2021). Sequential extraction methods are employed to investigate the composition of dissolved organic carbon (DOC) fractions, which have been found to contain a diverse range of aromatic and hydrophilic compounds with varying degrees of stability and bioavailability (Liu et al., 2022). Consequently, an understanding of the labile fraction of dissolved organic carbon (DOC) in biochars is of paramount importance for the assessment of their stability, environmental impact, and potential applications in soil management and carbon sequestration strategies.

The extraction of DOC from biochar can be conducted using a variety of solvents and methods, each of which reveals different components and properties of BDOC. The extraction of BDOC can be conducted using a variety of solvents: 0.1 mol·l⁻¹ HCl, 0.1 mol·l⁻¹ NaOH, deionized water, hot water (60 °C or 80 °C), and salts (0.1 mol·l⁻¹ NaCl; 0.1 mol·l⁻¹ KCl). Liu et al. (2019) conducted an extraction of dissolved organic carbon (DOC) constituents from forty-six biochars employing deionized water, 0.1 mol·l⁻¹ HCl, and 0.1 mol·l⁻¹ NaOH solutions. The findings indicated a discernible trend whereby the concentration, aromaticity and molecular weight of DOC derived from biochar typically demonstrated an ascending order in the sequence of acid, water-, and base-extractable DOC samples (Liu et al., 2019). Wu et al. (2018) the volume and properties

of dissolved organic carbon (DOC) released from biochar obtained from wetland vegetation were examined across various extraction conditions, such as deionized water, 0.1 mol·l⁻¹ hydrochloric acid, sodium hydroxide, sodium chloride. The results revealed that the highest DOC concentration was discharged in the NaOH solution, whereas the lowest concentration was observed in H₂O (Wu et al., 2018). Differences in the composition of BDOC depend on a number of extraction process factors, including the type of extractant, extraction time and temperature. The extraction and use of BDOC requires a comprehensive approach to balance its benefits for soil and plant health with potential environmental risks. Comparative studies on the effect of various biomass and pyrolysis temperature on BDOC properties are needed, as well as the development of more accurate and efficient methods for BDOC analysis to better understand its composition and dynamics.

In the present investigation, an assessment was conducted on the DOC emanating from biochar. A total of eight biochar specimens derived from sewage sludge and digestate underwent pyrolysis at temperatures ranging from 400 °C to 800 °C for analysis. The primary aims of this research encompassed: (1) exploring the impact of varied extracting solutions (deionized water, hot water, 0.1 mol·l⁻¹ NaOH) on the DOC released from biochars; (2) characterizing alterations in the composition and properties of the DOC released from biochars.

MATERIALS AND METHODS

The study was carried out on two types of biochar. The biochars were produced from digestate (BP) and sewage sludge (BS). The dried and ground digestates/sewage sludge were placed in a fluidised quartz reactor, which is part of a pyrolysis unit (constructed at the Faculty of Chemistry, UMCS). Pyrolysis was performed in an inert atmosphere (N₂, 100 cm³·min⁻¹) by heating the system to 400, 500, 600 and 800 °C. All samples were subjected to slow pyrolysis at a heating rate of 10 °C·min⁻¹. The biochars were prepared at the Faculty of Chemistry, Maria Curie-Skłodowska University in Lublin. The biochars were labelled as follows: BS400, BS500, BS600, BS800, BP400, BP500, BP600, BP800, depending on the pyrolysis temperature. The characteristics of the biochar are presented in Table 1.

Table 1. The selected properties of biochars

Parameter	BS400	BS500	BS600	BS800	BP400	BP500	BP600	BP800
pH	8.1	10.96	10.61	10.43	11.21	12.10	11.75	10.31
EC (mS · cm ⁻¹)	64	570	657	600	25.5	17.33	21.8	29
Total organic carbon (%)	32.32	23.26	11.54	17.89	22.65	18.98	19.44	16.78
Residual organic carbon (%)	1.99	2.68	13.67	8.19	4.66	4.75	4.39	6.47
Total inorganic carbon (%)	0.18	0.09	0.07	0.09	0.11	0.12	0.11	0.28

Sequential extraction

The scheme of the extraction procedure is shown in Figure 1. The extraction of the DOC involved the use of three distinct extraction solutions: deionized water, hot water, and 0.05 mol·l⁻¹ NaOH, leading to the categorization of water-extractable DOC (WEOC), hot-extractable DOC (HEOC), and base-extractable DOC (BEOC) correspondingly. The concentrations of dissolved organic carbon (DOC) were analyzed using a TOC-L Shimadzu analyzer (Shimadzu, Japan) and the UV-VIS spectrophotometer Hitachi (Hitachi, Japan). Dissolved organic carbon

in biochar (BDOC) (mg·g⁻¹) calculated according to the Equation 1 (Liu et al., 2022):

$$BDOC = \frac{V \cdot c}{M} \quad (1)$$

where: *V* – volume of solvent (L), *c* – concentration of total organic carbon in solvent (TOC mg·l⁻¹), *M* – mass of the biochar (g).

Cumulative *BDOC* concentration is the sum of concentrations of individual *BDOC* forms in extracted solutions. The spectral absorption ratio of (*E*₂/*E*₃) calculated according to the Equation 2 (Yamashita and Ikeda, 2010):

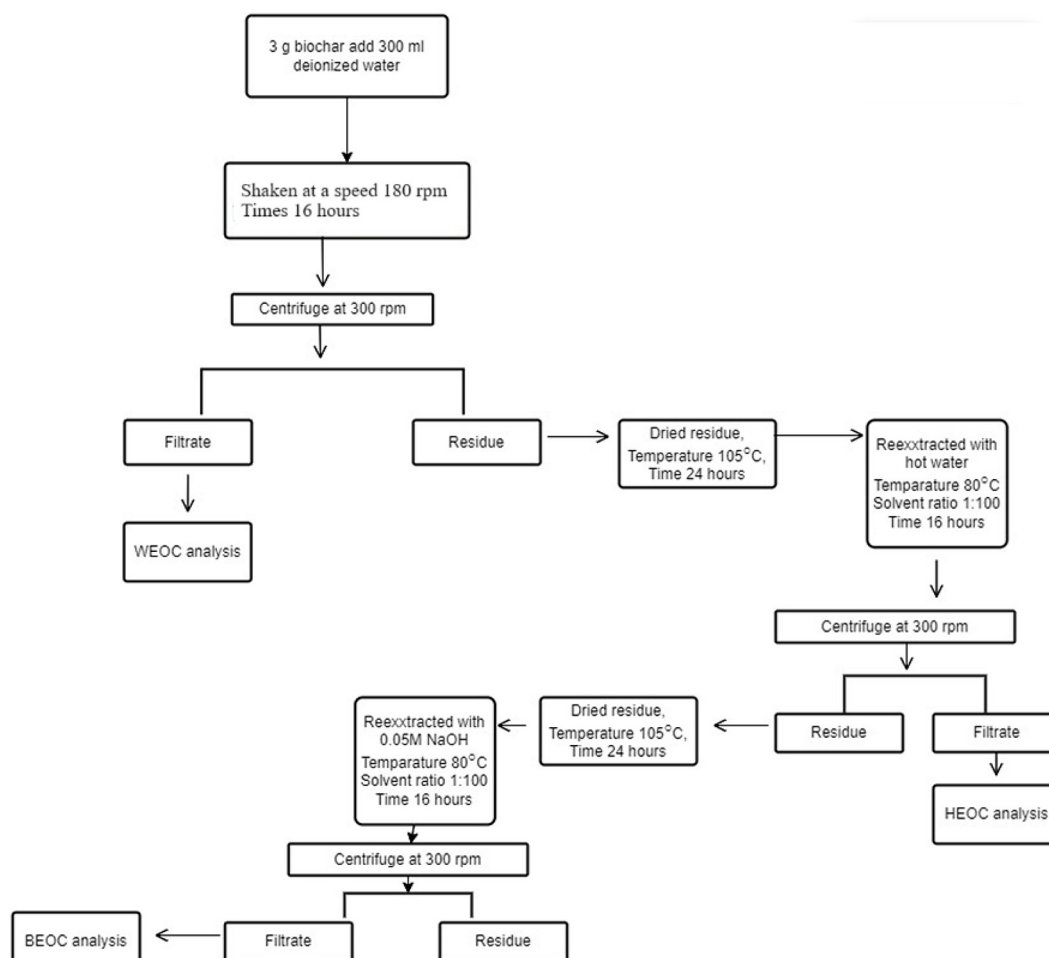


Figure 1. Flow chart for extraction of biochars

$$\frac{E_2}{E_3} = \frac{A_{254}}{A_{365}} \quad (2)$$

where: A_{254} and A_{365} – absorbance marked with wavelength 254 nm and 365 nm.

The specific UV absorption at 254 nm ($SUVA_{254}$) calculated according to the Equations 3 and 4 (Wang et al., 2013):

$$a_{254} = \frac{2.303 \cdot A_{254}}{L} \quad (3)$$

$$SUVA_{254} = \frac{A_{254}}{(L \cdot c)} \quad (4)$$

where: A_{254} – absorbance factor (m^{-1}), L – the length of the quartz tray 4.5 cm, $SUVA_{254}$ – the specific UV absorption ($L \cdot mg^{-1} \cdot m^{-1}$).

STATISTICAL ANALYSIS

Each trial was repeated thrice, and the results were presented as averages with corresponding

standard deviations. Statistical analysis was carried out using Statistica version 13.3. The multivariate analysis of variance (MANOVA) model was constructed to evaluate the effects of biochar types and extract types on BDOC. In addition, the effect of the intercept was included to assess the baseline level of BDOC without the influence of the studied factors. A significance level of 0.05 ($p < 0.05$) was used to assess the statistical significance of the results.

RESULTS

Table 2 presents the concentration of BDOC in all the biochar studied in the extraction solutions examined, i.e. deionized H_2O , hot H_2O and sodium hydroxide solution. Figure 2 illustrates the distribution of BDOC in the various extraction solutions, demonstrating that the predominant component is sodium hydroxide, with a

Table 2. BDOC concentrations ($mg \cdot g^{-1}$) of BS, and BP biochars were measured in various extraction solutions, with a sample size of three

Type of biochar	BDOC concentration ($mg \cdot g^{-1}$)			Cumulative BDOC Concentration ($mg \cdot g^{-1}$)
	WEOC	HEOC	BEOC	
BS400	6.5±0.11	10.22±0.23	24.99±2.57	41.71±2.91
BS500	1.53±0.03	1.25±0.17	5.2±0.24	7.98±0.44
BS600	1.67±0.11	0.68±0.02	3.03±0.03	5.38±0.16
BS800	0.41±0.02	0.19±0.03	4.93±0.51	5.53±0.56
BP400	1.5±0.2	0.61±0.19	17.9±2.48	20.01±2.87
BP500	0.15±0.04	0.11±0.07	1.57±0.14	1.83±0.25
BP600	0.21±0.02	0.1±0.02	1.8±0.01	2.11±0.05
BP800	0.13±0.01	0.14±0.02	0.78±0.07	1.05±0.1

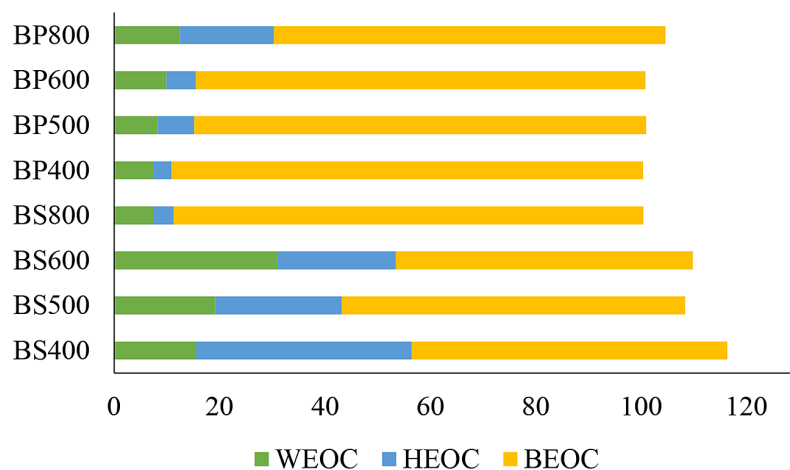


Figure 2. The distribution of dissolved organic carbon concentration in water (WEOC), hot water (HEOC) and a sodium hydroxide (BEOC) solution

range of 56% to 86%. Treatment with sodium or potassium bases promotes the dissociation of carboxyl and phenyl groups and the breaking of ester bonds, leading to an increase in the concentration of dissolved organic carbon. This phenomenon has been observed in several studies where alkaline conditions facilitate the cleavage of ester bonds (Ding et al., 2012).

The water-extracted fraction ranged from 7% to 31%. This is the most mobile fraction, easily penetrating various environmental components (Tfaily et al., 2017). The lowest values were obtained for the hot water fraction, which ranged from 3 to 41%. HEOC is considered a heterogeneous pool due to the fact that hot water (≥ 70 °C) eradicates vegetative cells of microorganisms and leaches various components from microbial biomass, along with numerous nonmicrobial organic substances. Consequently, HEOC exhibits significantly greater biodegradability rates compared to WEOC (Hamkalo and Bedernichek, 2014). Regardless of the type of biochar (BS or BP) and the production temperature, the BEOC fraction always has the largest percentage of the BDOC concentration.

Biochar has different concentrations of BDOC depending on the pyrolysis temperature. Higher biochar production temperatures result in lower BDOC concentrations in all extraction solutions. This is evident for both sludge and digestate biochars. For the WEOC fraction, the BDOC concentration in BS800 is more than 10 times lower than in BS400 biochar. BS800 biochar had the lowest BDOC concentrations of all sewage sludge biochars, with the highest concentration in BEOC ($4.93 \text{ mg}\cdot\text{g}^{-1}$) and a cumulative concentration of $5.53 \text{ mg}\cdot\text{g}^{-1}$. A similar trend is observed for digestate biochars, with the lowest BDOC concentrations found in BP800, where the BEOC is 0.78 mg/g and the cumulative concentration is $1.05 \text{ mg}\cdot\text{g}^{-1}$.

Sewage sludge biochar is characterized by a higher BDOC content in all fractions investigated

compared to digestate biochar. The BDOC concentration in the WEOC and HEOC fractions in BS biochar is more than 10 times higher than the BDOC concentration in digestate biochar (BP) at pyrolysis temperatures of 400 °C and 500 °C. The differences in BDOC values between sludge and digestate biochars decrease with increasing temperature.

Sodium hydroxide-extractable fractions always have the highest BDOC concentration compared to the deionized water-extractable and hot water-extractable fractions. For example, in BS600 biochar the BDOC concentration in the BEOC fraction is twice as high as in the WEOC fraction, while in BP600 biochar it is more than eight times higher. Table 3 presents the results of the MANOVA statistical analysis. The intercept is statistically significant ($p < 0.05$), indicating that the mean BDOC value, without taking other factors into account, is significantly different from zero. The MANOVA analysis confirms that both biochar type and extract type have a statistically significant effect on BDOC.

These findings align with those of Liu et al., who discovered a significant correlation between the amounts of DOC extracted from biochar and both temperature and extraction (Li et al., 2017). The research results and statistical analysis confirm that both the production temperature and the type of biochar significantly influence the concentration of DOC in biochar. When biochar is applied to soil, these differences may be important in relation to the availability of organic carbon to soil microorganisms. The environmental significance of WEOC in biochars has been emphasized in previous literature (Lehmann and Kleber, 2015), prompting a comparison of WEOC levels under various pyrolysis settings and feedstock varieties.

Higher pyrolysis temperatures result in lower dissolved organic carbon concentrations for both types of biochar. For sewage sludge biochar, WEOC values decrease significantly from 6.5 at 400 °C to 0.41 at 800 °C. A similar trend

Table 3. Univariate tests of significance for the BDOC

Parameter	Sigma-constrained parameterization Decomposition of effective hypotheses				
	SS	Degrees of freedom	MS	F	p
Free word	642.24	1	642.24	35.44	0.000001
Type of biochar	682.93	7	97.56	5.38	0.000241
Ekstrakt	352.17	2	176.078	9.71	0.000390
Error	688.53	38	18.11		

is observed for digestate biochar, where WEOC decreasing from 1.5 at 400 °C to 0.13 at 800 °C.

At lower temperatures (400 °C), sewage sludge biochar (SSB) has significantly higher BDOC concentrations in the WEOC fraction compared to digestate biochar (6.5 mg·g⁻¹ vs. 1.5 mg·g⁻¹). With increasing temperature, the difference in WEOC between the studied biochars decreases, with both reaching low values at 800 °C (0.41 for BS800 and 0.13 for BP800). The observation suggests that the pyrolysis temperature plays a crucial role in influencing the quantity of dissolved organic carbon found in biochar, as

elevated temperatures lead to the production of a more persistent and less soluble type of organic carbon. The type of feedstock (sewage sludge vs. digestate) has a significant effect on WEOC at lower temperatures but becomes less significant at higher temperatures. The assessment of the aromatic abundance and molecular characteristics of dissolved organic carbon (DOC) components derived from BS and BP and introduced into H₂O, hot H₂O, and NaOH solutions was conducted by analyzing the SUVA₂₅₄ value (Figure 3) and the E2/E3 ratio (Figure 4). The specific ultraviolet absorbance at 254 nm (SUVA₂₅₄) is a

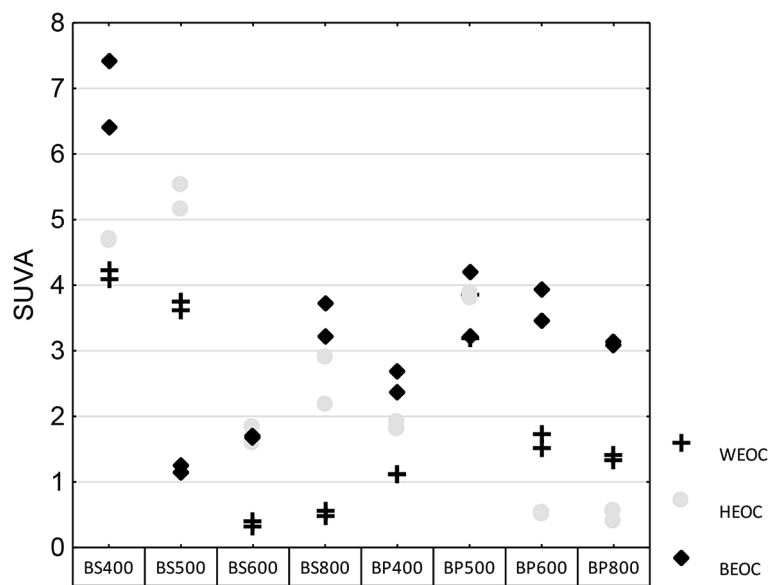


Figure 3. The specific UV absorbance (SUVA₂₅₄) of BDOC released from the biochar in various extracts

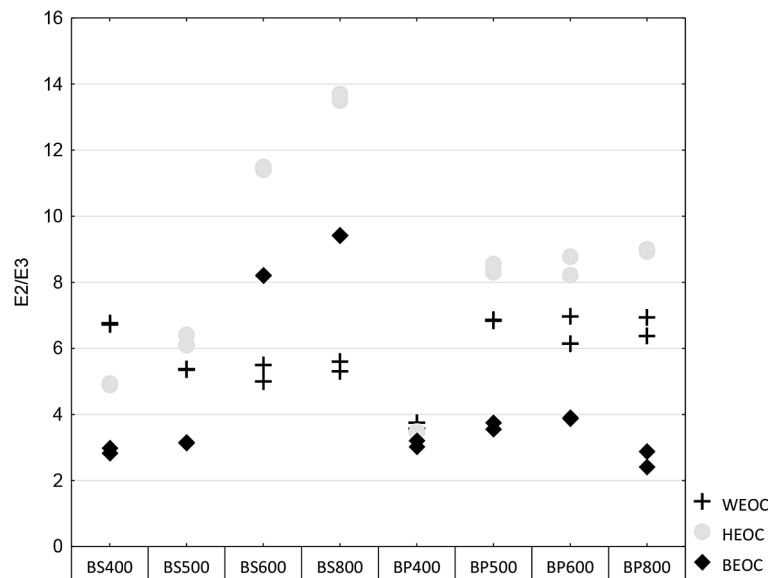


Figure 4. The ratio (E2/E3) from UV absorbance of BDOC released from the biochar in various extracts

useful parameter for assessing the aromaticity of dissolved organic carbon (DOC). $SUVA_{254}$ value greater than $4 \text{ L} \cdot \text{mg}^{-1} \cdot \text{m}^{-1}$ typically indicates the presence of higher levels of aromatic and hydrophobic substances in the DOC, while a value less than $3 \text{ L} \cdot \text{mg}^{-1} \cdot \text{m}^{-1}$ indicates the presence of more hydrophilic substances (Uchimiya et al., 2016). This correlation is significant because aromatic compounds tend to absorb more UV light due to their conjugated double bond structures, $SUVA_{254}$ is often used as a surrogate indicator for the study of aromatic compounds in soil and water (Pradhan et al., 2014).

BS400, BS500 and BP500 have higher $SUVA_{254}$ values in the WEOC fraction ($3.4\text{--}3.7 \text{ L} \cdot \text{mg}^{-1} \cdot \text{m}^{-1}$), indicating more aromatic and hydrophobic substances. BS600, BS800, BP400, BP600, and BP800 show lower $SUVA_{254}$ values ($0.437\text{--}3.470 \text{ L} \cdot \text{mg}^{-1} \cdot \text{m}^{-1}$), indicating more hydrophilic substances. The BEOC fraction tends to have higher DOC concentrations ($1.220\text{--}6.910 \text{ L} \cdot \text{mg}^{-1} \cdot \text{m}^{-1}$) compared to WEOC and HEOC, especially for BS400, BP500, BP600, and BP800.

The E2/E3 ratio, which refers to the absorption ratio at 254 nm and 365 nm, is an absorbance coefficient in the range of 254 to 365 nm. This ratio can characterize the degree of humification of organic matter and indicate the source of the organic matter (Wang et al., 2020). The E2/E3 ratio is inversely proportional to the molecular weight of the organic matter (Curtin et al., 2011).

The biochar produced from sewage sludge (BS) at higher temperatures (BS600, BS800) exhibits lower E2/E3 values in the HEOC and BEOC fractions when compared to the biochar produced from digestate (BP) at the same temperatures. The E2/E3 values observed in the WEOC and HEOC fractions of digestate biochar (BP) indicate a potential reduction in the molecular weight of the organic matter present.

The application of elevated pyrolysis temperatures (BS600, BS800) to sewage sludge biochar (BS) has been observed to result in a notable reduction in E2/E3 values within the HEOC and BEOC fractions. This phenomenon is indicative of a heightened molecular weight of organic matter. In the case of digestate biochar (BP), higher pyrolysis temperatures also result in a decrease in E2/E3 values in the BEOC fraction, though not to the same extent as observed in sewage sludge biochar.

It has been demonstrated that the HEOC and BEOC fractions are characterized by higher molecular weights of organic matter, particularly in

the case of sewage sludge biochar. The production temperature of biochar and the type of feedstock (sewage sludge vs. digestate) have a significant impact on the E2/E3 ratio and the molecular weight of organic matter in various fractions. It has been demonstrated that sewage sludge biochar exhibits a higher molecular weight of organic matter at higher production temperatures, particularly in the HEOC and BEOC fractions.

DISCUSSION

The study evaluated the impact of different extraction conditions on the concentration and properties of DOC in biochars derived from sewage sludge and digestates at different pyrolysis temperatures. The principal objective was to ascertain the impact of disparate extraction solutions (deionized water, hot water, $0.05 \text{ mol} \cdot \text{L}^{-1}$ NaOH) on the quantity and quality of DOC released from biochar. The concentration of DOC was found to significantly decrease with an increase in temperature, with the greatest reduction observed at $600 \text{ }^\circ\text{C}$ and $800 \text{ }^\circ\text{C}$, in comparison to temperatures of $400 \text{ }^\circ\text{C}$ and $500 \text{ }^\circ\text{C}$. This phenomenon was observed in both sewage sludge and digestate biochars. For instance, the WEOC concentration in BS800 was approximately 10 times lower than in BS400. A similar pattern was observed in BP biochars.

As demonstrated by Zhang et al., the DOC derived from biochar is dependent on the pyrolysis atmosphere. Air-limited pyrolysis has been shown to produce a greater quantity of BDOC than N_2 and CO_2 flows, and to contain a higher proportion of humus-like substances (Yue et al., 2023). The concentration and properties of DOC in biochar are significantly influenced by the type of feedstock. The concentration of DOC in sewage sludge biochars was found to be higher than that in digestate biochars, particularly at lower pyrolysis temperatures. These differences diminished with increasing pyrolysis temperature. Azem's studies demonstrated that plant-derived biochar produced greater quantities of aliphatic BDOC at lower temperatures, whereas higher temperatures resulted in the generation of more aromatic BDOC (Azeem et al., 2023).

The type of extracting compound also influenced the concentration of BDOC. The DOC fractions extracted using NaOH exhibited the highest concentrations, in comparison to the fractions extracted with deionized water and hot water. BEOC

accounted for a range of 56% to 86% of the total DOC concentration. In studies conducted by Liu et al., the water-extractable were found to range from 3.21% to 35.57% (Liu et al., 2019). Similarly, as observed in the aforementioned studies, over half of the DOC from biochar was released in the NaOH solution.

The analysis of $SUVA_{254}$ and the E2/E3 ratio indicated that higher pyrolysis temperatures lead to greater stability of DOC and an increase in the content of aromatic and hydrophobic substances. Biochars produced at higher temperatures exhibited lower E2/E3 values, indicative of a higher molecular weight of organic matter. It has been demonstrated that dissolved organic carbon components with higher molecular weights, such as those with elevated aromatic content, are frequently less bioavailable in comparison to smaller, more labile fractions (Peuravuori and Pihlaja, 1997).

The results of the conducted studies demonstrate the significance of meticulously selecting the extraction conditions to accurately assess the concentration and properties of DOC in biochar. This is of paramount importance for comprehending the environmental impact and potential applications of biochar.

CONCLUSIONS

A comprehensive understanding of the dynamics of BDOC is essential for the evaluation of their potential applications in agriculture and environmental protection. The findings demonstrate that the pyrolysis temperature, type of feedstock, and extraction conditions exert a significant influence on the concentration and properties of dissolved organic carbon in biochars. It is therefore of paramount importance to gain an understanding of these factors if biochars are to be effectively utilised in agriculture. The concentration of DOC in biochars derived from sewage sludge and digestates is significantly reduced by higher pyrolysis temperatures (600 °C and 800 °C). Biochars produced at lower temperatures (400 °C and 500 °C) exhibited higher BDOC concentrations, suggesting greater lability and bioavailability of these fractions in soil.

It has been demonstrated that the application of elevated pyrolysis temperatures results in the enhanced stability and aromaticity of BDOC, which is advantageous for the long-term sequestration of carbon in soil. The concentration of dissolved

organic carbon in sewage sludge biochars is higher than that in digestate biochars at lower pyrolysis temperatures. These differences become less pronounced at higher pyrolysis temperatures.

The BDOC fractions extracted with a sodium hydroxide solution exhibited the highest BDOC concentrations, in comparison with the fractions obtained through deionized water and hot water extraction. The examination of $SUVA_{254}$ and the E2/E3 ratio revealed that biochars produced at higher temperatures have a greater proportion of aromatic and hydrophobic substances, as well as a higher molecular weight of organic matter.

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