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Micropollutants in biochar produced from sewage sludge: A systematic review on the impact of pyrolysis operating conditions

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ABSTRACT

Biochar obtained from sewage sludge serves as a valuable soil amendment in agriculture, enhancing soil properties by increasing the nutrient content, cation exchange capacity, water retention, and oxygen transmission. However, its utilisation is hampered by the presence of micropollutants such as polycyclic aromatic hydrocarbons (PAHs), polychlorinated dibenzodioxins (PCDDs), polychlorinated dibenzofurans (PCDFs), polychlorinated biphenyls (PCBs), and volatile organic compounds (VOCs). Previous studies indicate that the type and amount of micropollutants can be significantly adjusted by selecting the right process parameters. This literature review provides an overview of how (1) pyrolysis temperature, (2) carrier gas flow and type, (3) heating rate, and (4) residence time affect the concentration of micropollutants in biochar produced from sewage sludge. The micropollutants targeted are those listed by the European Biochar Certificate (EBC) and by the International Biochar Institution (IBI), including PAHs, PCDD/Fs, PCBs and VOCs. In addition, per- and poly-fluoroalkyl substances (PFAS) are also considered due to their presence in sewage sludge.

The findings suggest that higher pyrolysis temperatures reduce micropollutant levels. Moreover, the injection of a carrier gas (N_2 or CO_2) during the pyrolysis and cooling processes effectively lowers PAHs and PCDD/Fs, by reducing the contact of biochar with oxygen, which is crucial in mitigating micropollutants. Nevertheless, limited available data impedes an assessment of the impact of these parameters on PFAS in biochar. In addition, further research is essential to understand the effects of carrier gas type, heating rate, and residence time in order to determine the optimal pyrolysis process parameters for generating clean biochar.

1. Introduction

Ocean and sea dumping of industrial and municipal wastewater are known to contaminate waters with high concentrations of harmful pollutants (United States Environmental Protection Agency (EPA), 2021). Thus, wastewater treatment plays a key role in ensuring clean waters, but the optimal disposal of the resulting sewage sludge (SS) remains unresolved. Due to its rich nutrient and carbon content, more than 43 % and 48 % of sewage sludge are applied on agricultural land in the USA (2021) and Europe (2021) (European Federation of National Associations of Water Services, 2021; United States Environmental Protection Agency (EPA), 2023). However, pollutants like toxic heavy metals (HM), dioxins (PCDD), furans (PCDF), polyaromatic hydrocarbons (PAH) and polychlorinated biphenyls (PCB) are present in the sewage sludge (Fijalkowski et al., 2017; Singh et al., 2021). Through land application, organisms are exposed to these compounds, and bioaccumulation and biomagnification occur, resulting in the incorporation of these pollutants into the food chain (Clarke and Smith, 2011). Recently, long-lasting and widespread per- and polyfluoroalkyl substances (PFAS) have been detected in sewage sludge (Bossi et al., 2008; Johnson, 2022). Due to their high toxicity and their resistance to degradation within wastewater treatment facilities (Kim Lazcano et al., 2019; Kim Lazcano et al., 2020; Letcher et al., 2020), regulations have been enacted to prohibit the agricultural use. A case in point is the state of Maine in the USA, which implemented such regulations in 2022 (State of Maine, 2022). This exacerbates the difficult disposal, as landfilling does not provide a sustainable alternative since it leads to multiple environmental issues, including methane emissions, formation and

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leaching of toxic compounds including a mixture of high-concentration organic and inorganic contaminants like ammonia nitrogen, xenobiotics, humic acids, inorganic salts and heavy metals, and spontaneous uncontrolled fires and explosions (Costa et al., 2019; Kjeldsen and Fischer, 1995; Vaverková, 2019; Wiszniowski et al., 2006). Therefore, sustainable strategies for sewage sludge management must be developed which reduce the amount of sludge, recover nutrients, remove micropollutants and ideally provide a sustainable energy source.

In this regard, the pyrolysis process represents a promising approach for converting sewage sludge into valuable end products including a gas, a liquid, and a solid phase, so-called biochar (Oladejo et al., 2019; Vali et al., 2021; Zaharioiu et al., 2021). Although pyrolysis seems to be a sustainable solution for sewage sludge disposal, the reuse of the generated end products is challenging. The produced biochar can still contain micropollutants, like HMs, PAHs, PCDD/Fs, PCBs, VOC, and PFAS, which prevent its safe application in agriculture (Brtnicky et al., 2021; Clarke et al., 2010; Wang et al., 2019; Zielińska and Oleszczuk, 2015).

The composition and properties of the resulting biochar are determined by several factors, including the feedstock used, the specific pyrolysis process employed, and the associated process conditions (Inguanzo et al., 2002; Qadeer et al., 2017; Tripathi et al., 2016). Among these factors, the feedstock and the pyrolysis system are unalterable elements within the existing facilities. Consequently, an adjustment of the process parameters has the potential to mitigate pollutant levels in the biochar without necessitating supplementary treatment steps. Nevertheless, as far as our knowledge extends, there is presently no comprehensive review consolidating and evaluating the results obtained by the different research investigating the influence of different operational conditions on the pollutant content in biochar. However, this knowledge is essential for generating a clean and safe biochar for a sustainable agricultural application.

This work performs a systematic review of studies addressing the influence of the pyrolysis operating conditions on the presence of micropollutants in the biochar produced, focusing on the treatment of sewage sludge. Our aim is to set the state-of-the-art in this field and determine the research gaps to be addressed by future works. Since the feedstock significantly impacts the biochar composition, the scope of this work is limited to sewage sludge pyrolysis. The list of micropollutants considered is based on the International Biochar Certificate (IBC, 2015) (International Biochar Initiative, 2015) and the European Biochar Certificate (EBC, Version 10.3, April 2023) (European Biochar Certification, 2023). These guidelines are frequently referenced for developing environmental regulations. However, it must be mentioned that IBI and EBC are not regulating documents but provide recommendations for specific thresholds. The micropollutants of concern specified by the IBI and EBC encompass PAHs, PCDD/Fs, PCBs and VOC. Additionally, PFAS are addressed in the review due to the growing concern of its high concentration in sewage sludge. After discussing the impact of each process parameter, i.e. temperature, heating rate, residence time and gas flow on the specific pollutant levels, the results are compared to determine the key process parameters affecting the total amount of pollutants in biochar. Finally, we propose further research perspectives to produce clean biochar.

2. Characterization of biochar

Depending on the pyrolysis process, the biochar yield typically falls within the range of 10–50 wt% based on the feedstock (Wang et al., 2017; Zheng et al., 2019). The carbon-rich biochar encompasses nearly all inorganic constituents present form the original waste (Haghighat et al., 2020) and shows a high porosity, water-holding capacity, and cation exchange capacity (CEC) (Onosedeba Ehidiamhen, 2020).

To ensure consistent quality and safety standards for biochar, both the EBC and IBI have devised biochar guidelines. These guidelines serve as valuable reference points for regulatory purposes. The EBC divides the biochar into four classes: EBC-Feed, EBC-Material, EBC-AgroOrganic, and EBC-Agro, whereby only the last two include biochar application to the field (Schmidt et al., 2023). The EBC-Agro and EBC-AgroOrganic consider the new EU fertilizer regulation (2019) (European Parliament, 2019). Additionally, EBC-AgroOrganic incorporates the EU Commission guidelines pertaining to organic production (EU-Comission Regulation (EU), 2019).

Table 1 provides a list of properties recommended by the EBC and IBI guidelines, including a comprehensive overview of the various biochar properties derived from sewage sludge.

The IBI (Version 2.1) and EBC (Version 10.2G) guidelines also recommend determining 16 PAHs, 17 PCDD/Fs and 7 or 12 PCBs, which are all known for their toxic, negative impact on the immune system and partly carcinogenic properties (Fenton et al., 2021; Patel et al., 2020; Pelch et al., 2019; Safe, 1994; World Health Organization (WHO), 2010). The detailed list is provided in the appendix and includes a comparison of the required compounds based on EBC or IBI. In this regard, two points should be emphasized: 1) VOCs are addressed differently in the IBI and EBC guidelines. Under EBC, it is mandatory to measure the total VOC concentration (European Biochar Certification, 2023), while IBI just recommends its determination (International Biochar Initiative, 2015). Paragraph 3 elucidates the reasons for including VOC in the analysation to ensure a safe application. 2) PFAS are not covered by the EBC and IBI guidelines. Nevertheless, given their considerable stability and toxicity (Fenton et al., 2021; Podder et al., 2021; Xu et al., 2022), it is expected that these guidelines will undergo revisions in the near future.

Table 2 summarizes the specific thresholds for biochar according to the IBI and EBC guidelines, where the thresholds refer to the total concentration of the respective pollutant group. A wide variation of IBI values can be observed since it is based on the regulations of different countries (United Kingdom, Australia, Canada, United States, etc.), while the EBC determines the threshold values on the basis of the Swiss Ordinance on Risk Reduction related to Chemical Products, the EU Organic Farming Ordinance, and the soil protections in force in Switzerland's and Germany's regulations (European Biochar Certification, 2023).

3. Potential risks of using biochar as a soil amendment

The application of biochar has gained increasing attention due to its beneficial effects on soil properties and agricultural yields (Hossain et al., 2010; Liu et al., 2018; Yue et al., 2017; Zornoza et al., 2016). However, its value is often criticized due to the potential presence of pollutants like HMs, PCDD/Fs, PCBs, PAHs, PFAS, and VOCs. Some pollutants are already present in sewage sludge and are concentrated during the pyrolysis process, as it is the case for heavy metals. Others, such as PAHs, PCDD/Fs, PCBs, and VOCs, can either be present in the sludge or get formed during the pyrolysis process (Buss et al., 2015; Madej et al., 2016).

A fraction of pollutants like PAHs are leachable (Chen et al., 2019b; Wang et al., 2019). The soil organic carbon–water partitioning coefficient (K_{OC}), used to predict the behaviour of chemicals in aquatic environments, varies with the molecular weight. Specifically, high molecular weight PAHs exhibit higher K_{OC} values compared to low molecular weight PAHs (Buss et al., 2015). This leads to lower water solubility and stronger binding to organic surfaces. As a result, LMW-PAHs primarily enter plants and, therefore, can be transmitted into the food chain (Li et al., 2017). Consequently, the accumulation of PAHs (especially HMW-PAHs) in soil and their adsorption by roots due to organic material should not be neglected (Buss et al., 2015). Also, VOCs can have an adverse impact on the microbial process within plants and hinder their growth (Buss and Mašek, 2014; Dutta et al., 2017; Kwon et al., 2015; Spokas et al., 2011). Buss et al. (2015) consider VOC compounds to pose an even higher risk to the environment than PAHs due to their phytotoxic effects.

PCDD/Fs and PCBs show low water solubility and strong lipophilic

Table 1
Comparison of the EBC (version 10.3, 2023) and IBI (version 2.1, 2015) guidelines, including value ranges for biochar out of sewage sludge.

			EBC-Agro	IBI	Value range	Source
Element analysis	Total Ash	[% wt]	x	x	49–79	(Pituello et al., 2015; Zielińska et al., 2015; Zielińska and Oleszczuk, 2015)
	C_{tot}	[% wt]	x	x	8–31	(Xu et al., 2018; Zhang et al., 2022; Zielińska et al., 2015)
	C_{org}	[% wt]	x	≥ 60 % [a]	18	(Fachini et al., 2021a)
				≥ 30 % [b]		
	H	[% wt]	x	x	0.2–6.9	(Xu et al., 2018; Zhang et al., 2022; Zielińska et al., 2015)
	N_{tot}	[% wt]	x	x	0.5–5.0	(Xu et al., 2018; Zhang et al., 2022; Zielińska et al., 2015)
	O	[% wt]	x	–	2.4–18.4	(Xu et al., 2018; Zhang et al., 2022; Zielińska et al., 2015)
	S	[% wt]	x	–	0.3–1.47	(Xu et al., 2018; Zhang et al., 2022; Zielińska et al., 2015)
Total nutrient	N	[g/kg]	x	Opt.	0.4–6	(Ghorbani et al., 2022)
	P	[g/kg]	x	Opt.	10–96	(Ghorbani et al., 2022; Yuan et al., 2016; Zielińska et al., 2015)
	K	[g/kg]	x	–	1–16	(Ghorbani et al., 2022; Yuan et al., 2016; Zielińska et al., 2015)
	Mg	[g/kg]	x	Opt.	1–17	(Ahmad et al., 2022; Ghorbani et al., 2022; Zielińska et al., 2015)
	Ca	[g/kg]	x	Opt.	1–52	(Ahmad et al., 2022; Ghorbani et al., 2022; Yuan et al., 2015)
	Fe	[g/kg]	x	–	4–132	(Ahmad et al., 2022; Yuan et al., 2015; Zielińska et al., 2015)
Physical properties	Moisture	[% wt]	–	x	0.1–8	(; (Fachini et al., 2021a; Yuan et al., 2015)
	Volatile Matter	[% wt]	–	Opt.	6–47	((Fachini et al., 2021a; Yuan et al., 2015; Fachini et al., 2021b)
	Bulk density	[kg/m^3]	x	–	750–767	(Jain et al., 2019; Mośko et al., 2021b)
	pH	[-]	x	x	6.4–13.1	(Yuan et al., 2015; Zhang et al., 2022; Zielińska et al., 2015)
	Electrical conductivity	[dS/m]	x	x	0.3–7.8	(Ghorbani et al., 2022; Yuan et al., 2015)
	Total surface area	[m^2/g]	Opt.	Opt.	20–89	(Mośko et al., 2021b; Singh et al., 2020; Zhao et al., 2023)

x = Verification required; Opt. = Optional; [a] = IBI biochar class 1; [b] = IBI biochar class 2.

character, which decreases with increasing halogenation degree (Canadian Council of Ministers of the Environment, 2002). Due to their high octanol–water partition coefficient, PCDD/Fs and PCBs tend to accumulate highly in sediments and solids, with the potential for uptake by plants (Canadian Council of Ministers of the Environment, 2002; World Health Organization (WHO), 2021). Although PCBs have been banned since 1979 due to their high toxicity (US EPA, 2015), they are still detected in the environment due to their high chemical and thermal stability, and ability to accumulate in organisms. Their environmental availability and exposure pathways are impacted by the number and the placement of containing chlorin atoms, whereby 12 out of 209 are considered to have a similar toxicity like PCDD/Fs (Bhalla et al., 2016; Moško et al., 2021a).

PFAS are used in multiple industries, like metal plating, coating, waterproof materials, fire-fighting foams, and leather and textile production, due to their versatile chemical and physical properties (Gao et al., 2020; Glüge et al., 2020). However, these compounds raise concerns about their impact on the environment due to their persistence, high bioaccumulation and leachability (Interstate Technology and Regulatory Council, 2020; Kundu et al., 2021; Longpré et al., 2020). They pose a serious risk to human health and ecological life, including negative effects on the liver, kidneys, blood, immune system, and increased cholesterol, endocrine disruption and immunosuppression are associated with PFAS (Andersen et al., 2021; Bonefeld-Jørgensen et al., 2014; Cui et al., 2020; Fenton et al., 2021; Gao et al., 2020). They are marginally or not degraded during wastewater treatment, whereby sewage sludge counts as a sink for PFAS (Lenka et al., 2021; Zhang and Liang, 2021). Aerobic and anaerobic digestion does not lead to any significant reduction; instead, new PFAS compounds are even formed (Zhang and Liang, 2021). The US EPA (2001) reported that the average PFAS load from sewage sludge in the US ranges from 2749 to 3450 kg/year, resulting in an annual release of 1370 to 2070 kg of PFAS to agricultural land (Zhang and Liang, 2021).

Heavy metals are strongly bound to biochar, and their immobilization and availability for plants are closely tied to the soil pH (Qadeer et al., 2017). However, it is known that biochar undergoes a modification of its properties due to the ageing process (Cheng et al., 2006; Godlewska et al., 2021; Sorrenti et al., 2016). Thereby, oxidation and microbial degradation occur, leading to a change in biochar structure and an increase in the polarity and CEC, whereas the pH and hydrophobicity decrease (Oleszczuk and Kołtowski, 2018). These alterations influence the interactions between biochar, organisms, and soil, influencing the accessibility and leaching of pollutants.

The ambiguity surrounding the presence of pollutants in biochar has prompted the European Commission to implement legislation prohibiting the application of biochar derived from sewage sludge as of early 2022 (Huygens et al., 2019). The decision was justified with the statement: “The lack of ensured emerging contaminant removal [during pyrolysis] is especially problematic for specific input material streams [like sewage sludge] that may contain a broad range of emerging contaminants,…[which] may lead to a reduced level of environmental and human health safety relative to the counterfactual scenario of waste incineration.” (Huygens et al., 2019). Hence, it is imperative to comprehend how pyrolysis process parameters impact pollutant levels in order to ensure an alternative and sustainable technology converting sewage sludge into a valuable end-product.

4. Review methodology focusing on micropollutants in biochar

The scope of this article is to give a systematic view of sewage sludge pyrolysis and the impact of process parameters on the pollutant amount in biochar. Fig. 1 shows the schematic approach for collecting the information, whereby the following steps were assessed:

Table 2
Thresholds for polycyclic aromatic hydrocarbons (PAHs), dioxins and furans (PCDD/Fs), and polychlorinated biphenyls (PCBs) following the IBI and the EBC regulations, and volatile organic compounds (VOC) in biochar (BC) based on EBC (Version 10.3) and IBI regulations (Version 2.1) (International Biochar Initiative, 2015; Schmidt et al., 2023); DM = dry matter.

		∑ PAHs [ng/g DM]	∑ PCDD/Fs [ng/g DM]	∑ PCBs [ng/g DM]	∑ VOC [% DM]
IBI		6 000–300 000	0.017	200–1 000	Optional
EBC	**AgroBio (Class II)**	4 000 ± 2 000	0.02	200	N.S.*
	Agro (Class III)	6 000 ± 2 200	0.02	200	N.S.*

∑ = Total concentration * N.S. = Maximum amount not specified but measurement is required.

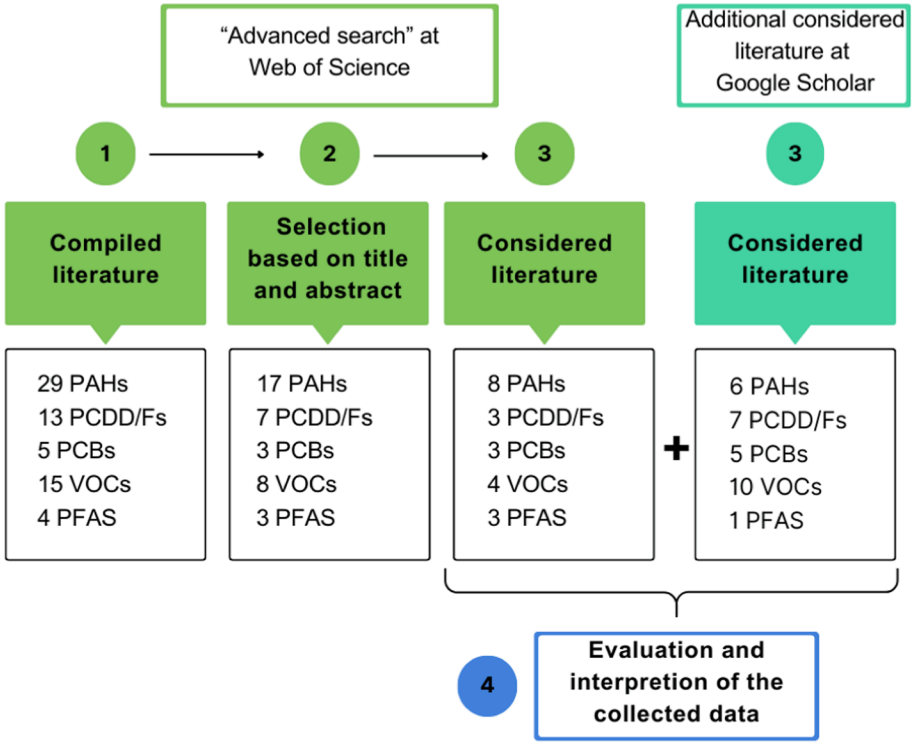

Fig. 1. Selection pathway for the systematic literature review for answering the research question: Which process parameters impact the amount of micropollutants in the generated biochar out of sewage sludge.

1. Compilation of the literature using Web of Science and Google Scholar:
   a. Definition of the research question: "Which process parameters impact the amount of micropollutants in the generated biochar out of sewage sludge?"
   b. Determination of the keywords for the selection of the relevant literature, which includes: "pyrolysis" AND "sewage sludge" AND "biochar" AND "name of the specific pollutant".
2. Selection of the relevance of the obtained literature: The selection of documents is performed based on the search terms in the article title, followed by the relevance of the article abstract and a detailed reading of the publication focusing on the scope of this literature review.
3. Evaluation of the quality of the selected literature and extraction of the data: Literature with a lack of precision on how the experiments were carried out or the tests were analyzed has not been considered for inclusion in this review.
4. Interpretation of the collected data: The extracted data were collected in Excel files to see trends or differences in the studies. The obtained insights were then interpreted and evaluated in comparison with other literature.

5. Micropollutants in biochar out of sewage sludge and the impact of process parameters

5.1. Polycyclic aromatic hydrocarbons

Polycyclic aromatic hydrocarbons (PAHs) are the most frequently occurring contaminants in biochar (Godlewska et al., 2021). They can be present in sewage sludge, but they can also be formed during the incomplete combustion of the sludge during the pyrolysis process (Dutta et al., 2017; Hemlin and Lalangas, 2018; Schmidt et al., 2023; Wang et al., 2017). PAHs are a class of semi-volatile compounds characterized by the presence of two or more aromatic rings.

5.1.1. Influence through variation of process conditions

The formation of PAH is a complex reaction pathway involving multifaceted reaction pathways such as cyclization, dealkylation, dehydrogenation, and aromatization. The extent of PAH formation is tied to the concentration of free radicals, whose formation is facilitated by the presence of oxygen (Serban C. Moldoveanu, 2019). That is an important factor which should be considered more in detail since biochar can get in touch with oxygen during the cooling process or later on during the storage. This means that, although a pyrolysis process may ensure a PAH-free biochar, the cooling process and storage have to be controlled.

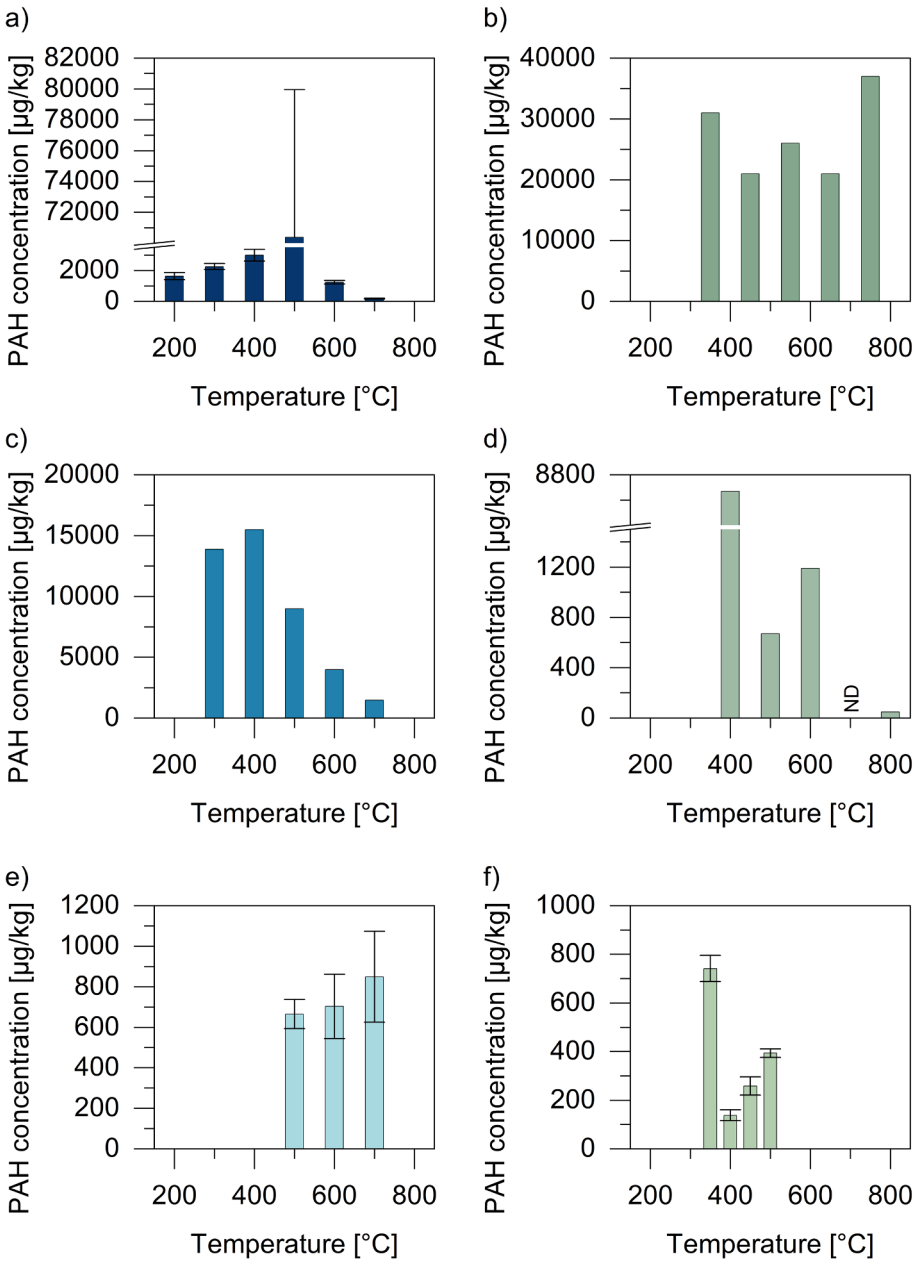

Fig. 2. PAH concentration as a function of temperature under different pyrolysis operating conditions. a) Tube furnace with a residence time of 6 h, N_2 gas supply of 0.3 L/min, heating rate of 5 °C/min (Luo et al., 2014), b) Rotary kiln with a residence time of 20 min, a gas supply of 10 L/min (Buss et al., 2016), c) Tube furnace with a residence time of 3 h and a N_2 gas supply of 0.3 L/min at a heating rate of 10 °C/min (Chen et al., 2019b) d) Quartz fixed-bed reactor with a residence time of 2 h, a He gas supply of 0.15 L/min (Moško et al., 2021a) e) Tube furnace with a residence time of 5 h, a N_2 supply of 0.63 L/min and a heating rate of 25 °C/min (Zielińska and Oleszczuk, 2015) f) Muffle furnace with a residence time of 2 h, no gas supply by a heating rate of 10 °C/min (Raj et al., 2021). ND = Below detection limit.

However, the first question to be answered is if the pyrolysis process parameters impact the amount of PAH contained in the biochar. Hence, six publications (Buss et al., 2016; Chen et al., 2019b; Luo et al., 2014; Moško et al., 2021a; Raj et al., 2021; Zielińska and Oleszczuk, 2015) analyzed the PAH concentration as a function of the operating temperature. Their process parameters are listed in the appendix. The concentration of PAHs in biochar varies significantly between studies as a consequence of the different sewage sludges. Nevertheless, it is possible to compare the different publications by considering the trends of each study, which are shown in Fig. 2.

We observe a similar trend of PAH concentrations in Fig. 2a and 2c. Both show a PAH peak between 400 and 500 °C, which drops with increasing temperature. This suggests that lower temperatures support the formation process, while temperatures above 500 °C volatilize the formed PAHs. Nevertheless, this tendency was not consistent in the case of Fig. 2b, 2e, and Fig. 2f. In Fig. 2b, no discernible temperature dependence is evident, whereas in Fig. 2e) and 2f), the PAH concentration increases with increasing temperature. Consequently, establishing a clear trend related to the influence of temperature on the total PAH concentration in biochar remains elusive. These findings align with the research conducted by (Hale et al., 2012). They concluded that a clear relationship between temperature and total PAH concentration could not be established (Hale et al., 2012). This leads to the assumption that other process parameters are the main drivers for the concentration of PAH in biochar from sewage sludge.

Comparing the process conditions of Buss et al. (2016), Fig. 2b, with

the other studies in Fig. 2, shows that besides the use of a different reactor, a shorter residence time was used. Buss et al. (2016) applied only 20 min, whereas the other studies used residence times of at least two hours. This leads to the assumption that a too-short residence time promotes the PAH concentration in the biochar. Moreover, a comparison of slow and fast pyrolysis supports this assumption since lower PAH concentrations generally can be seen when using slow pyrolysis instead of fast pyrolysis (Hale et al., 2012; Wang et al., 2017; Zielińska and Oleszczuk, 2015).

Closely related to the temperature and residence time is the heating rate. Ko et al. (2018) analyzed the formation of PAHs at different heating rates (5, 10 and 20 °C/min) during a slow pyrolysis process of sewage sludge. The total PAH concentration was three times higher at 20 °C/min than at 5 °C/min. This observation was related to surface cracking due to the quick temperature change, which releases a high amount of volatile compounds (Thomas et al., 2015). The combination of a high temperature and a high amount of volatiles leads to frequent collisions and the formation of new PAHs (Hale et al., 2012). Therefore, the concentration of PAHs might be reduced through the use of low heating rates.

The works in Fig. 2a, 2c and 2e were performed using the same reactor type, gas type and residence time over three hours. Nevertheless, the total concentration in Fig. 2e is between 1.8 (Fig. 2a) and 5.7 (Fig. 2c) times lower at 600 °C. This might be due to the carrier gas flow rate, which is two times higher in Fig. 2e than in Fig. 2a and 2c. Also, Ko et al. (2018) verified a significant impact of the PAH concentration on the gas flow rate. An increase in the gas flow rate from 200 up to 400 ml/min reduces the detected PAH concentration by a factor of 2.3, and a further increase in the gas flow rate up to 800 ml/min leads to a PAH reduction by a factor of 5.4. A comparison carried out by La Rosa et al. (2019) between a batch reactor (no removal of the generated vapour phase) and a rotary kiln (constant removal of the generated vapour phase) leads to the same observation. Also, the used type of carrier gas seems to have an impact on the PAH concentration, since using CO_2 instead of N_2 leads to a 50–86 % higher decrease in PAHs (Kwon et al., 2012; Lee et al., 2017), although it should be noted that these studies used styrene-butadiene rubber and printed circuit boards as feedstock. Thereby, CO_2 acts as a carbon scavenger to mitigate air pollutants by blocking reaction pathways towards benzenes and PAH formation (Kwon et al., 2012). However, it is still unknown whether the same results can be achieved using sewage sludge.

5.2. Polychlorinated dibenzodioxins (PCDDs) and polychlorinated dibenzofurans (PCDFs)

The term “dioxins” is often used to describe polychlorinated dibenzo-p-dioxins (PCDDs) and polychlorinated dibenzofurans (PCDFs), which are persistent environmental pollutants. Both chlorinated groups show a triple-ring structure that consists of two benzene rings connected by one or two oxygen atoms.

PCDD/Fs are formed during incomplete combustion in the presence of chlorine (Altarawneh et al., 2009). Thereby, the chlorine concentration significantly influences the formation of PCDD/Fs (Hale et al., 2012), whereby chlorine concentration exceeding 1 % (e.g. $CaCl_2{\cdot}6H_2O$ or polyvinylchloride) promotes the formation of PCDD/Fs (Han et al., 2022). This underlines the importance of a careful selection of the additional feedstock if sewage sludge is to be mixed with another material before it is added to the pyrolysis process. The formation process of PCDD/Fs occurs in the gas phase (homogenous route) as well as on the surface of the char particle (heterogeneous route) (Lopes et al., 2015). Thereby, the homogenous route occurs in the pyrolysis process at a temperature around 500 to 800 °C (Lopes et al., 2015). HCl is released into the gas phase and forms highly chlorinated compounds like polychlorophenols, polychlorobenzenes or PCBs (Garcia-Perez, 2008; Lopes et al., 2015; Stanmore, 2004). These compounds are the precursors to the formation of PCDD/Fs by polycondensation catalysed by metals (Garcia-Perez, 2008; Hu et al., 2006). Thereby, four principal pathways are so far identified in the formation of PCDD/F: 1) cyclization of polychlorobiphenyls, 2) cyclization of polychlorobiphenyl ethers, 3) chlorination of dibenzofuran and 4) dichlorination of PCDFs (Stanmore, 2004). The heterogeneous route takes place at a temperature of 200 to 450 °C, whereby two different formation pathways exist (Garcia-Perez, 2008; Lopes et al., 2015; Stanmore, 2004): 1) chloroaromatic precursors, formed in the gas phase by multistep reaction, condense on the biochar or fly ash and are converted by a catalytic reaction to PCDD/Fs. Copper and iron, which are integrated on the carbon surface, act as a catalyser. 2) In the de-novo reaction, the carbon matrix burns, whereby oxidation and chlorination occur simultaneously on the surface of the biochar or ash particles. The formation process is driven by the amount of oxygen, whereby the reaction rate is determined by the carbon breakdown (Stanmore, 2004; Wielgosiński, 2011). Metals, mainly copper, catalyse the oxidation of carbon and chlorination and dichlorination of organic products and act as a “shuttle” for chlorine between the gas and solid phases (Shadrack and Slawo, 2017; Stanmore, 2004). Comparing the heterogeneous and homogenous pathways, the heterogeneous pathway is expected to be the dominant phase, generating the main PCDD/Fs (Altarawneh et al., 2009). In addition to the chemical formation, PCDD/Fs in biochar can occur due to volatilization followed by adsorption or re-condensation of PCDD/Fs on the biochar (Han et al., 2022).

5.2.1. Influence through variation of process conditions

The general amount of chlorine in sewage sludge is low and contains around 0.05–0.18 % on dry matter basis (Chen et al., 2019a; Yu et al., 2017). Based on the low chlorine concentration, the biochar produced out of sewage sludge shows a low PCDD/F concentration of up to 11 ng/kg (Dai et al., 2018; Weidemann et al., 2018).

Studies on the determination of PCDD/Fs in biochar from sewage sludge are very limited. However, expanding the scope to studies using other raw materials, it can be observed that pyrolysis temperature can affect PCDD/F concentration in biochar. Thus, the highest PCDD/F peak can be measured at a temperature of 200 to 400 °C for feedstock, which was a mixture of municipal solid waste and sawdust, whereas at a temperature above 400 °C most PCDD/Fs transfer into the gas phase (Hale et al., 2012; Lyu et al., 2016). Dai et al. (2018) achieved a reduction of more than 95 % by using wet sewage sludge as feedstock at a temperature $>$ 500 °C. The final concentration was between 9 and 11 ng/kg. Lyu et al. (2016) significantly reduce the total PCDD/F in biochar (out of sawdust) at the pyrolysis temperature $>$ 700 °C. This observation substantiates the presumption that the heterogeneous formation pathway is the predominant formation mechanism of PCDD/Fs during pyrolysis. This heterogeneous pathway, as previously mentioned, involves the formation of PCDD/Fs in the gas phase, followed by their condensation onto the biochar. The condensation process can be prevented by the supply of a carrier gas, which ensures a rapid removal of the vapour phase and, therefore, avoids the condensation of chloroaromatic precursors (Dai et al., 2014; Hu et al., 2006). The provision of a carrier gas also prevents the intrusion of oxygen into a pyrolysis process, which is an important consideration since the presence of oxygen supports the formation of PCDD/Fs (Hale et al., 2012). Given that the pyrolysis process operates in the absence of oxygen, the detected amount of PCDD/F in biochar is generally low. However, this can change if the cooling process runs under an oxygen atmosphere, as shown by Hu et al. (2006). The PCDD/F concentration increases 103 times compared to the starting material if the cooling process occurs under an oxygen atmosphere. Thereby, PCDD/Fs are formed, and re-condensation on the biochar occurs (Buss, 2021). In contrast, a 99.99 % reduction was achieved by ensuring oxygen-free pyrolysis and applying a constant carrier gas (N_2) during the pyrolysis and cooling processes (800 °C, PCDD/F removal of sediments) (Hu et al., 2006). The minimum amount of carrier gas flow for a sufficient removal of PCDD/Fs was not determined. Nevertheless, this aspect is interesting from an economic perspective in

order to optimize the process and reduce operating costs. Furthermore, it is unknown if heating rate and residence time impact the PCDD/F concentration; hence further research is required.

5.3. Polychlorinated biphenyls (PCBs)

The general structure of PCBs is $C_{12}H_{10-x}Cl_x$, where x denotes the number of chlorine atoms in the range of 1 to 10 (United Nations, 1999). Its toxicity is influenced by both the quantity and the placement of chlorine atoms (Moško et al., 2021a). The formation of PCBs runs a similar mechanistic pathway like PCDD/Fs and requires the presence of chloride in the feedstock (Huygens et al., 2019), which highlights again the careful selection when mixing sewage sludge with another feedstock for pyrolysis. The HCl is the starting material, which is transformed into chloride radicals (Liu et al., 2001). Metals, mainly copper, act thereby as a catalyst as well as a transmitter of heterogeneous chlorines (Stanmore, 2004). The generated chlorine radical triggers a chain reaction in which benzols are transformed into PCBs. The formation process starts at temperatures around 300 °C, and the adsorption of PCB occurs on the biochar through the presence of elementary carbon or soot particles (Huygens et al., 2019). In total, 209 different compounds exist, whereby the melting point and lipophilic character increase with increasing degree of chlorination, whereas the vapour pressure and water solubility decrease. Moško et al., (2021a) detected 21 different PCBs in sewage sludge with a total PCB concentration of 274.2 ng/g. The detected amount in biochar derived from sewage sludge falls either below the detection limit or is found to be ≤26.6 ng/g (Alipour et al., 2022; Bleuler et al., 2020; Moško et al., 2021a). Consequently, there is limited available information in this regard.

5.3.1. Influence through variation of process conditions

The formation of PCBs is temperature dependent (Huygens et al., 2019; Moško et al., 2021a). Temperature around 350 °C maximizes the PCB formation (Huygens et al., 2019), whereas temperature above 600 °C leads to a reduction of over 97 % (14.9 ng/g) (Moško et al., 2021a). Increasing the temperature up to 700 °C or even higher achieves a reduction of up to 99.8 % to 99.9 % (Moško et al., 2021a). The reduction of PCBs might be thereby explained by volatilization followed by a decomposition of PCBs during the pyrolysis process, which is why a combustion of the volatile phase after the pyrolysis process is recommended. Alipour et al. (2022) achieved a high reduction of PCB during sewage sludge pyrolysis at 450 °C and 600 °C. For both biochars, the PCB value was below the detection limit.

A variation of the residence time from 30, 60 up to 90 min has almost no impact on the PCB concentration, whereby it must be mentioned that these tests were carried out with contaminated sediments at 800 °C (Hu et al., 2006). Studies analysing the impact of the residence time on the amount of PCBs in the biochar generated out of sewage sludge were not found.

Besides the impact of the residence time, Hu et al. (2006) also analysed the impact of the cooling process on the PCB concentration in contaminated sediments. They determined a significant impact of the atmospheric conditions on the PCB as well as on the PCDD/F concentration. The TEQ value was zero for both pollutant groups by applying N_2 (1 L/min) in the cooling process. By running the test by the same pyrolysis condition but exposing the sediments to oxygen during their cooling process, the TEQ value for PCBs and PCDD/Fs increased to 29.0 and 1743.4, respectively. This suggests using a carrier gas during the cooling process ensures a constant removal of the vapour phase and an oxygen-free atmosphere, reducing the pollutants. Although similar results can be expected for sewage sludge pyrolysis, this has not yet been proven.

5.4. Volatile organic compounds

VOCs result from thermochemical transformation and rearrangement of chemical compounds like cellulose, lignin and hemicellulose (Buss and Mašek, 2016). They are organic chemicals formed during pyrolysis, particularly between 250 and 350 °C, where a significant solid mass loss occurs due to the volatilization of VOCs (Buss and Mašek, 2014). Based on their low boiling points, VOCs typically remain in the vapour and later in the bio-oil phase (Ghidotti et al., 2017; Spokas et al., 2011), which is why the detected VOCs in the biochar are mainly due to re-condensation of VOCs during or after the pyrolysis process (Buss and Mašek, 2016; Han et al., 2022; Spokas et al., 2011; Xiao et al., 2018). The ten most frequently observed VOCs in biochar are benzene, toluene, propanol, acetone, methyl ethyl ketone, methyl acetate, octanal, 2,3-butadiene, pentanal and 3-methyl butanal (Buss et al., 2015; Ghidotti et al., 2017; Godlewska et al., 2021), which can exceed 100 mg/kg in total if the pyrolysis process is not optimal (Han et al., 2022). As these compounds are relatively weakly bound, they are easily released into the soil and groundwater (Haubold-Rosar et al., 2016). Although knowing the negative effects of VOCs on plants, soil microbes and algae (Zheng et al., 2019), the available literature is limited. However, the determination of containing VOCs in biochar could be used to indicate potential phytotoxic effects.

5.4.1. Influence through variation of process conditions

Contrary to PAHs, PCDD/Fs, and PCBs, the amount of VOC is less dependent on the pyrolysis feedstock (Aller, 2016; Zheng et al., 2019). Instead, the mineral content impacts the VOC concentration, whereby a correlation between temperature and VOC was seen for biochar with ash content <20 % (Aller, 2016). However, a poor linear correlation was found for ash content >20 %, as it is the case for biochar obtained from sewage sludge. Nevertheless, pyrolysis temperature influences the type of VOCs produced and, thus, the VOCs that can be sorbed on the biochar. Temperatures below 350 °C lead to the formation of short carbon chains such as ketones, aldehydes and furans, whereas temperatures above 350 °C mainly produce VOCs with long carbon chains (Spokas et al., 2011). Thereby, short VOCs (i.e., less than 11 carbon atoms) exhibit phytotoxic effects, whereas long-chain hydrocarbons appear to have no toxic effect on plants (Huygens et al., 2019). Another parameter of interest is the H/C ratio, since a H/C ratio > 0.7 increases the probability that benzene, ethylbenzene, toluene, phenols, polycyclic aromatic hydrocarbons, xylenes and volatile fatty acids are present (Aller, 2016; Ghidotti et al., 2017). Consequently, biochar with a high degree of carbonization is recommended to ensure carbon stability, which reduces the leaching effect. However, a higher degree of carbonization leads to a reduction in cation exchange capacity, which is why other ways have to be determined to reduce the VOC concentration.

Since sorption of evaporated VOCs is a source of contamination of biochar, the implementation of a carrier gas stream in both the pyrolysis process and the cooling process might be advisable. However, further studies are needed to confirm this statement. Furthermore, the choice of carrier gas seems to play a role in the formation of VOCs, as demonstrated by Zhang et al. (2011). The use of different gases, like N_2, CO_2, CO, CH_4 and H_2 results in variations of the VOC profile. Specifically, when CO or CO_2 atmospheres are used, methoxy-containing compounds decrease, and monofunctional phenols increase. Furthermore, the type of carrier gas also impacts the surface area and porosity of the biochar (Kim et al., 2019). Both properties were doubled using CO_2 instead of N_2 during the pyrolysis of oak-wood. This effect assumes that the CO_2 supply releases more VOCs from the feedstock, which reinforces the necessity to ensure a quick removal of the generated vapor phase through the adequate selection of carrier gas, as it can impact both VOC composition and biochar properties.

5.5. Per- and polyfluoroalkyl substances

Per- and polyfluoroalkyl substances (PFAS) are highly fluorinated aliphatic substances which contain a linear or a branched alkyl chain and at least one fully fluorinated methyl or methylene carbon atom - C_nF_{2n+1} (Buck et al., 2011; Cui et al., 2020; Winchell et al., 2022). More than 12,000 different PFAS are listed by the US EPA, which can be divided into two major classes: non-polymers (including perfluoroalkyl acids (PFAAs), perfluoroalkyl carboxylic acids (PFCAs), perfluoroalkane sulfonic acids (PFSAs) or perfluoroalkane sulfonamides (FASAs)) and polymers (including fluoropolymers and perfluoropolyethers (PFPE)) (Interstate Technology and Regulatory Council, 2020). The major PFAS are perfluorooctanesulfonic acid (PFOS) (403 ± 127 ng g^{-1} dry weight) and perfluorooctanoic acid (PFOA) (34 ± 22 ng g^{-1} dry weight).

5.5.1. Influence through variation of process conditions

Up to now, information on the behaviour of PFAS during the pyrolysis process is limited, as their occurrence in sewage sludge has only recently become known (Kundu et al., 2021). Thoma et al. (2022) analysed the PFAS behaviour during the pyrolysis of sewage sludge at a full-scale pyrolysis process, operating at an average temperature of 617.5 °C and a residence time of 19.1 min. The pre-dried sewage sludge contains 21 different PFAS compounds, with concentrations ranging from 2 to 85 µg/kg. After pyrolysis, the detected PFAS concentration in biochar was below the detection limit, resulting in a removal efficiency of ≥ 81.3 %. This suggests that PFAS are released from the feedstock. This assumption is reinforced since organofluorine compounds like tetrafluoromethane (CF_4), hydrogen fluoride (HF) and hexafluoroethane (C_2F_6) were detected in the flue gas after passing the thermal oxidizer. Additionally, Williams et al. (2021) achieved the removal of 28 PFAS below the detectable level (≤0.005 mg/kg) during their bench-scale pyrolysis experiments of sewage sludge at 700 °C. The authors also assume that PFAS evaporate from the feedstock. In contrast to these results, no reduction was verified by Jin Hyo et al. (2015) when pyrolyzing sewage sludge at 300 and 700 °C with a heating rate of 7 °C/min and a residence time of 3 h. The PFOA and PFOS concentration of the sewage sludge was 11.51 µg/kg and 4.82 µg/kg, respectively, while the biochar generated at 300 °C had a concentration of PFOA and PFOS of 12.03 µg/kg and 4.86 µg/kg, respectively. Through the temperature increase up to 700 °C, the PFOA and PFOS concentration was 10.64 µg/kg and 6.28 µg/kg, respectively. However, the authors do not specify the reactor and process flow used, so that a re-condensation of vaporised PFAS cannot be ruled out.

Due to the limited data available up to now, it is impossible to determine the key factors that ensure a reduction or even total removal of PFAS in the biochar. In this regard, Kundu et al. (2021) mention that the pyrolysis temperature, the oxygen content, gas residence time, water vapour and the alkali and alkaline minerals (like Mg, Na, Ca and K) in the gas phase have an influence on the PFAS concentration in biochar. However, further research is needed to confirm this statement.

Table 3
Overview of pyrolysis operating conditions and their impact on micropollutants (polyaromatic compounds (PAHs), dioxins and furans (PCDD/Fs), polychlorinated biphenyls (PCBs) and volatile organic compounds (VOCs)).

	PAHs	PCDD/Fs	PCBs	VOCs
Process type	Fast pyrolysis: ↑ Slow pyrolysis: ↓	n/a	n/a	n/a
Temperature	No clear tendency	< 400 °C: ↑ (1)	≈ 350 °C:	< 350 °C: ↑ of short-chain VOCs
		> 400 °C: ↓	> 600 °C: ↓	> 350 °C: ↑ of longer-chain VOCs
		T > 1000 °C PCDD/Fs are destroyed		
Presence of a carrier gas	↓	↓	n/a	n/a
Gas type (O_2, CO_2, N_2)	O_2: ↑	O_2: ↑ (1)	O_2: ↑	Selection of gas impacts the type of formed VOCs
	CO_2 reached a higher reduction compared to N_2 atmosphere (1)	n/a	n/a	
Heating rate increase	↑	n/a	n/a	n/a
Residence time	Short residence time: ↑(1)	n/a	No impact(1)	n/a

↑ = Increase; ↓ = Reduction; n/a = No data available; (1) Result not achieved by SS pyrolysis.

6. Comparison of the process parameters on the specific pollutant amount

In order to obtain a clean and safe biochar with adequate parameters, it is not sufficient to consider only one contaminant. Instead, it is necessary to adopt a holistic approach considering all pollutants simultaneously to determine which operating conditions reduce the total amount of pollutants. Table 3 provides an overview of the process parameters and their impact on the pollutants studied. PFAS is not included in the table since the available information is too limited for drawing conclusions.

The analysis reveals that the selection of process temperature can influence both the type and quantity of pollutants detected in biochar. As a general guideline, temperatures exceeding 600 °C tend to reduce the concentrations of PCDD/F and PCB. Moreover, it seems that high pyrolysis temperature effectively reduces PFAS in biochar and forms long-chain VOCs, which show a lower toxicity to plants (Huygens et al., 2019). However, it should be mentioned that studies are inconsistent regarding the potential harm of VOCs, since various factors, such as soil properties, biochar ageing, and the presence of microorganisms, can influence the effects of VOCs. Consequently, further research is essential to comprehensively assess the impact of specific VOCs on crop growth and composition, in order to specify them in the IBI and EBC guidelines.

During the pyrolysis process, a gas phase is formed that contains evaporated pollutants from the feedstock or serves as a source for forming new pollutants. Adding a carrier gas reduces the PAH and PCDD/F content in the biochar, and it is assumed that the carrier gas also leads to the reduction of PCBs, PFASs and VOCs. This conclusion is based on the idea that a carrier gas supply shortens the contact time between the biochar and the gas phase, which can prevent the adsorption of contaminants onto the biochar. The data evaluation also leads to the assumption that the control of the cooling process plays an important role in producing a clean biochar. The same principles that apply to the pyrolysis process can also be applied to this context, whereby the carrier gas prevents the adsorption of possible contaminants onto the still-warm biochar. In addition, the carrier gas prevents oxygen entering during the cooling process, which is an essential factor, as oxygen can promote the formation of PCDD/Fs and PCBs. Research has also shown that oxygen plays a supporting role in the formation of PAHs, whereby its formation is temperature dependent (Shiju and Mary, 2008). It is important to note that comprehensive studies are needed to understand better the influence of carrier gas in the pyrolysis process and cooling processes. Particular attention should be given to the cooling process, as it appears to significantly impact the cleanliness of the biochar.

In this context, it is crucial to analyse the impact of the type of carrier gas, as it has been shown to affect both the pollutant levels as well as the properties of the biochar. The injection of CO_2 instead of N_2 as a carrier gas achieved a higher pollutant reduction while simultaneously increasing the surface area. This triggers a far-reaching idea of how to recycle the waste gas stream generated in a wastewater treatment plant,

such as from an anaerobic digester, and use it as a carrier gas stream in the pyrolysis and cooling process. Furthermore, synergies can be created between manufacturing companies and pyrolysis plants in order to recycle the waste gas stream. By considering that, it is necessary to take the calorific value of the synthesis gas produced into account to ensure that its heating rate is not negatively affected.

Furthermore, it is still too early to determine the behaviour of PFAS during the pyrolysis process. Considering the high stability of PFAS compounds, it is essential also to analyse the other generated phases, including the aqueous pyrolysis liquid, the pyrolysis oil and pyrolysis gas, to avoid transferring the pollutants from one phase to the next. This aspect is also a limitation of this present review, which focuses exclusively on biochar. However, in order to develop a sustainable process for the utilisation of sewage sludge, it is imperative to analyse all phases with regard to pollutant content. This allows the determination of the required post-treatment steps, which ultimately enables a global evaluation of the optimal process parameters, including economic considerations. In this regard, it is recommended first to consider the end use of the products and the associated requirements so that the desired properties can be targeted by setting the process parameters. Ideally, these assessments should be carried out in pilot plants as they provide results that accurately simulate real-world conditions and take into account factors such as heat exchange and mixing. In addition, it is crucial to analyse the feedstock for contaminant content as this enables the calculation of a removal efficiency factor. Using a removal efficiency as a benchmark facilitates meaningful comparisons between different studies, ultimately leading to informed decisions about the optimal process parameters for producing clean biochar.

7. Conclusion

This review studied existing research on how process parameters affect the levels of micropollutants in biochar, aiming to identify the optimal pyrolysis conditions to generate a clean biochar. The study shows that a temperature above 600 °C leads to the removal of PCDD/Fs and PCBs, and the first publications on PFAS indicate a trend where higher temperatures favour the production of PFAS-free biochar. A carrier gas supply during the pyrolysis process as well as at the cooling process, supports the generation of a clean biochar, but information is lacking to understand if and how the variation of the carrier gas type impacts the restraining pollutants.

Regarding the effect of the residence time on the concentration of micropollutants, the information available is limited and inconclusive, and thus, additional efforts are needed to determine the link between the pyrolysis residence time and the presence of micropollutants in biochar. Ideally, these tests should be carried out with a pilot or large-scale plant, as this better reflects reality. Research focusing on the effects of process parameters on PFAS content in biochar is still scarce and further work is needed in this area. In this regard, it is advisable to carry out comprehensive analyses of all resulting end products in order to prevent a shift of the highly stable PFAS compounds into another phase. This requires the use of cost-effective standard methods that allow comparison between different studies. In this process, it is crucial to consistently include the feedstock in the analysis in order to determine the removal efficiency and thus enable meaningful comparisons between studies. This leads to the conclusion that, in order to minimize the presence of micropollutants in the pyrolysis products, all generated end-products should be considered for the optimization of the pyrolysis conditions to prevent the transfer of micropollutants between the phases to achieve an actual removal.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.wasman.2023.12.036.

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