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Changes in availability of Ca, K, Mg, P and S in sewage sludge as affected by pyrolysis temperature

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Abstract: Pyrolysis is a promising technology for sewage sludge (SS) treatment providing several improvements of SS properties for soil application. However, information on the influence of pyrolytic temperature on the availability of nutrients in resulting biochar (BC) is limited. In this study, anaerobically stabilised SS was pyrolysed in a laboratory fixed-bed reactor at 220, 320, 420, 520, and 620 °C for 30 min in the N₂ atmosphere. Pyrolysis resulted in a higher total content of all studied nutrients in BCs. Aromaticity and hydrophobicity of BCs increased with increasing temperatures while solubility decreased. Relative availability (% from total content) of nutrients in BCs was in order: Ca > Mg ~ K > S > P. Pyrolysis at 220 °C produced acidic BC with a higher content of acetic acid-extractable nutrients compared to non-pyrolysed control. An increment in pH and a significant drop in the content of available Ca, Mg, K and S were found at temperature 320 °C. Pyrolysis at 320 °C increased the content of available P by 28 % compared to non-pyrolysed SS. At the temperature of 420 °C and higher, available contents of all studied nutrients were lower than in non-pyrolysed SS.

Keywords: torrefaction; biosolids; disinfection; plant nutrition; phosphorus; recycling

The majority of phosphorus (P) within the European Union is wasted through sewage sludge (SS) disposal (Van Dijk et al. 2016). Recycling of SS on agricultural soil is, therefore, one of the approaches for closing the P loop. Direct soil application of stabilised SS is currently common practice bringing several other benefits for soil quality. The dry SS usually contains 50–70 mass % of organic matter (Kacprzak et al. 2017), which is considered to be relatively bioavailable and is easily mineralised after application to soil (Hattori and Mukai 1986, Kacprzak et al. 2017). Even so, soil application of SS results in an increment of cation exchange capacity (CEC) of soil (Singh and Agrawal 2008), higher soil content of humic and fulvic acids (Urbaniak et al. 2017) and total content of organic carbon (C_{org}) (Albiach et al. 2001). Consequently, soil water holding capacity and soil aggregate stability

increase while the bulk density of soil decreases after the application of SS. However, soil application of SS also represents a significant risk of soil contamination by a wide range of organic and inorganic pollutants and/or pathogens commonly contained in SS (Clarke et al. 2017, Kacprzak et al. 2017, Száková et al. 2019). Pyrolysis of SS may overcome these problems, and therefore it represents a promising technology for SS pre-treatment before soil application. During pyrolysis, SS and resulting BC become disinfected due to high processing temperatures, and, at the same time, SS organic matter undergoes a variety of chemical transformations resulting in its higher stability and resistance against microbial degradation (Mašek et al. 2013, De Rosa et al. 2018). It has been well demonstrated that BC application to soil may result in a significant increment of soil pH, CEC

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(Hailegnaw et al. 2019a), and may decrease NO_3^- and dissolved organic carbon leaching (Hailegnaw et al. 2019b). However, it is still not well understood, how the temperature of SS pyrolysis influences the content and availability of nutrients contained in resulting BC. Without such knowledge, it is impossible to sufficiently predict the influence of produced BC on plant nutrition, and also to design a cost-effective technology for SS pyrolysis and subsequent nutrient recycling in soil.

The main goal of this study is to determine the changes in total and available contents of Ca, K, Mg, P, and S during the pyrolysis of anaerobically stabilised sewage sludge under a wide range of the applied pyrolysis temperatures.

MATERIAL AND METHODS

Sample collection and pre-treatment. The SS sample was collected from a wastewater treatment plant (WWTP) in the Czech Republic. The construction capacity of the WWTP is 29 000 population equivalent (PE), and $\text{Fe}_2(\text{SO}_4)_3$ is used for P precipitation. The used SS was anaerobically stabilised (mesophilic anaerobic digestion) and dewatered by decanter centrifuge (dry matter content 24 wt. %). After collection, the sample was air-dried in thin layers at 105 °C until constant mass. Dried SS was milled and passed through a 1 mm stainless sieve prior to analyses or pyrolysis, it served as a control and is denoted as SS-con.

Pyrolysis. Pyrolysis of samples was carried out in an inert atmosphere of N_2 (99.99%) using an electric laboratory tube furnace (GHA 12/600, Carbolite Gero Ltd., Hope, UK). Samples of known weight placed

in ceramic holders were introduced into a quartz tube previously connected to the stream of N_2 . The inlet flow was set to 100 L/h giving a calculated cold linear flow velocity within the empty reactor tube of 1.9 cm/s. Once the whole inner space of the tube was filled with N_2 , the tube was inserted into the preheated furnace. Samples were pyrolysed for 30 min at a given temperature (measured from the moment of reaching the target temperature inside the quartz tube). Pyrolysed samples are denoted as BC-220; BC-320; BC-420; BC-520 and BC-620 corresponding to the target pyrolytic temperatures of 220; 320; 420; 520 and 620 °C, respectively. The scheme of the fixed-bed pyrolysis reactor used is shown in Figure 1.

Analytical methods. The values of sample pH were determined in the supernatant after 2 h shaking in 0.01 mol/L CaCl_2 (1/5; v/v). Electrical conductivity (EC) was measured in filtrated supernatant after 1 h extraction of the sample with demineralised water (1/10; w/v). The content of ash was determined gravimetrically after burning the samples at 550 °C for 4 h. Total C, H, and N contents were measured using Vario Macro cube analyser (Elementar Analysensysteme GmbH, Hanau, Germany). The content of oxygen (O) was calculated as follows: $\text{O \%} = 100 \% - (\text{C} + \text{H} + \text{N} + \text{S} + \text{ash, \%})$ (Wang et al. 2013). Total contents of Ca, K, Mg, P, and S were determined by inductively coupled plasma optical emission spectrometry (ICP-OES; Agilent 720, Agilent Technologies Inc., Santa Clara, USA) after the dry-ashing procedure (Mader et al. 1998) followed by heating (130 °C) of the residue with a mixture of HF (48%) and HNO_3 (65%) (1:2; v:v) until dryness. The dry residue was subsequently reconstituted with 3 mL of *aqua-regia*

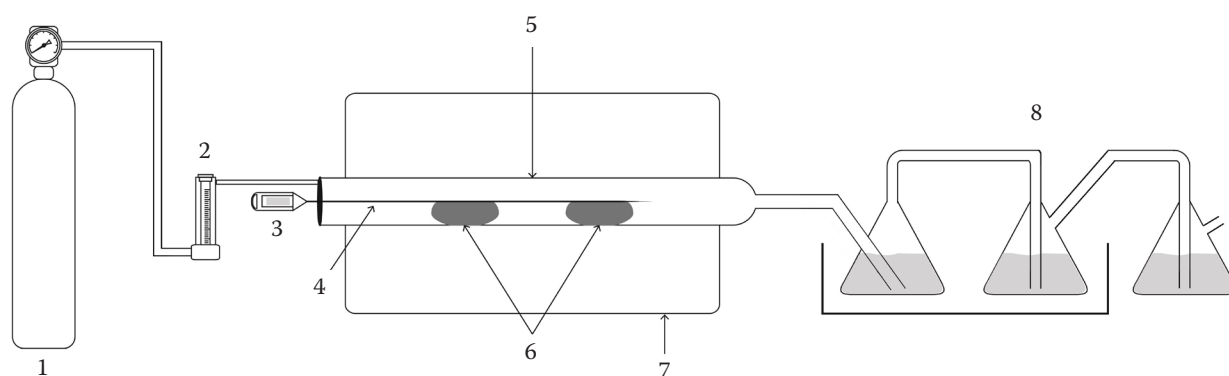


Figure 1. Schematic of the laboratory pyrolysis apparatus. 1 – the source of N_2 ; 2 – gas flow-meter; 3 – thermometer; 4 – thermometer probe; 5 – quartz tube; 6 – ceramic sample holders; 7 – electric furnace; 8 – volatiles collecting system

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and demineralised water to reach a final volume of 50 mL. The available contents of Ca, K, Mg, P, and S were determined in the supernatant after 16 h extraction in 0.11 mol/L CH₃COOH (1:100 w/v) and centrifugation (16 000 × g; 10 min). ICP-OES was used for determination (Ochecová et al. 2017).

Statistical analysis. All the analytical procedures and pyrolytic treatments were conducted in triplicates. Differences between treatments in the individual parameters were examined by one-way analysis of variance (ANOVA) followed by Tukey's honestly significant difference (HSD) post-hoc test ($P < 0.05$).

RESULTS AND DISCUSSION

Physicochemical properties. The loss of SS mass during pyrolysis increased with increasing pyrolytic temperature and followed the logarithmic trend (Table 1). The best-fitting function was found to be as follows:

$$y = -1017.1082 + 316.4617 \ln x - 23.4092 (\ln x)^2;$$

$$R^2 = 0.9997$$

where: y – loss of sample mass (wt. %); x – pyrolysis temperature.

BC-220 showed a loss of almost 9% of its mass compared to SS-con (corresponding to the BC yield 91%), while at the pyrolytic temperature of 620 °C, nearly 50% of the sample mass was released as vapours. The results are comparable with other studies of SS pyrolysis (Syed-Hassan et al. 2017), but when compared to the pyrolysis of different wood chips

(Mašek et al. 2013), the yield of SS biochar is considerably higher likely because of higher ash content in the feedstock material.

Content of ash in samples increased with increasing temperature of pyrolysis, and in BC-620, the ash content was nearly double as the content in SS-con. Pyrolysis at 220 °C led to slight sample acidification when compared to SS-con. From this point, the pH of samples increased with the increasing temperature reaching pH 8 at 620 °C. A steep increment in pH was observed between 520 °C and 620 °C (Table 1). A similar increasing trend was reported by Suliman et al. (2016) for poplar and douglas fir wood BCs between temperatures 350–650 °C, but in their study, BCs exceeded pH 8 already at temperatures around 350 °C. We assume that the overall alkalinisation with increasing pyrolysis temperature is caused by several factors. The main factors are likely the increasing content of ash in the resulting material and also the chemical transformations such as thermal decomposition of sulphates (Mu and Perlmutter 1981a), carbonates (Mu and Perlmutter 1981b), and/or decreasing content of acidic functional groups in the organic part of the material (Collard and Blin 2014). The amount of readily dissolvable salts, as determined by EC of water extracts, decreased with increasing temperature of pyrolysis in overall. A significant drop in EC was observed between temperatures 220 °C and 320 °C, and then a relative increment in EC was detected at 420 °C. A similar trend has been reported by Figueiredo et al. (2018) for SS biochars, but the very opposite trend and significantly lower values

Table 1. Main physicochemical properties and ultimate analysis of tested sewage sludge and biochar samples

Treatment	pH	EC (μS/cm)	Ash	C	N	O	H/C	C/N	O/C	BC yield (wt. %)
			(wt. %)				(AR)			
SS-con	5.62 ± 0.04	3267 ± 35.1	35.0 ± 0.59	31.3 ± 0.09	4.49 ± 0.04	22.9 ± 0.05	1.95 ± 0.03	8.13 ± 0.04	0.55 ± 0.00	na
BC-220	5.51 ± 0.02	2387 ± 37.9	37.2 ± 0.31	32.4 ± 0.38	4.47 ± 0.05	20.2 ± 0.54	1.66 ± 0.01	8.44 ± 0.01	0.47 ± 0.02	91.3 ± 0.01
BC-320	5.76 ± 0.10	536 ± 7.09	46.7 ± 0.44	32.6 ± 0.05	4.29 ± 0.02	11.8 ± 0.15	1.31 ± 0.01	8.84 ± 0.03	0.27 ± 0.00	70.5 ± 0.25
BC-420	6.21 ± 0.06	854 ± 6.35	57.0 ± 2.55	26.5 ± 0.21	3.52 ± 0.02	9.9 ± 0.24	0.95 ± 0.00	8.78 ± 0.02	0.28 ± 0.01	60.0 ± 0.81
BC-520	6.72 ± 0.03	548 ± 2.65	62.5 ± 0.18	26.3 ± 0.03	3.24 ± 0.02	5.54 ± 0.15	0.70 ± 0.01	9.44 ± 0.04	0.16 ± 0.00	53.2 ± 0.86
BC-620	8.02 ± 0.02	480 ± 4.93	68.5 ± 0.20	24.8 ± 0.23	2.69 ± 0.02	2.12 ± 0.30	0.41 ± 0.01	10.7 ± 0.04	0.06 ± 0.01	50.3 ± 0.15

Values shown represent the arithmetic mean ($n = 3$) ± standard deviation. Pyrolysed samples are denoted as BC-220, BC-320, BC-420, BC-520, BC-620 – target pyrolytic temperatures of 220, 320, 420, 520 and 620 °C. SS-con – control; na – not analysed; AR – atomic ratio; SS – sewage sludge; BC – biochar; EC – electrical conductivity

of EC were determined by Suliman et al. (2016) for wood BCs. The authors demonstrated increasing EC with increasing temperatures (350–650 °C), reaching a maximum value of 350 $\mu\text{S}/\text{cm}$.

The total content of carbon, nitrogen, and oxygen. The content of C in pyrolysed samples increased up to 320 °C and remained relatively stable at a temperature range between 420 °C and 620 °C. Within this range, a slightly decreasing trend was observable, and the total C content reached ~25% at 620 °C. The same effect of pyrolysis temperature has been reported (Figueiredo et al. 2018, Xu et al. 2018). Moreover, Xu et al. (2018) found no significant differences in C contents in SS biochars prepared at the temperatures ranging between 600 °C and 900 °C. In the present study, the total content of N in samples started to decrease from a temperature of 320 °C. Between 320 °C and 620 °C, the decline in N content followed a linear trend (Table 1). Oxygen content in samples was decreasing from temperature 220 °C. A sharp decline in O content was observed at temperatures between 220 °C and 320 °C, and around 2% of O was detectable at 620 °C. Atomic H/C ratio indicating the level of saturation of organic compounds in the materials, was ~2 in SS-con. This indicates the majority of organic compounds being saturated. H/C decreased with increasing temperature and reached 0.4 at 620 °C. According to Xiao et al. (2016), the H/C ratio < 1 may indicate a presence of benzene rings and/or aromatic clusters. Our results, therefore, indicate that the aromatisation of SS biochar began around temperature 420 °C. It corresponds well with find-

ings summarised by Collard and Blin (2014) that at 400 °C, benzene rings become preponderant in the products of cellulose pyrolysis. Similarly, the O/C atomic ratio as indicating the relative content of oxygenated functional groups decreased from 0.55 to 0.06 between temperatures 220 °C and 620 °C. The opposite trend was observed in the case of C/N, and the ratio increased with increasing pyrolysis temperature. From the results above, it can be concluded that with increasing pyrolysis temperature, SS biochar becomes less polar, more hydrophobic, and its aromaticity increases, which is in accordance to similar studies (Wang et al. 2013, Figueiredo et al. 2018, Wei et al. 2019).

The total content of mineral nutrients. In general, the total contents of Ca, K, Mg, and P in samples correlated well with the content of ash; thus, their contents increased with increasing pyrolytic temperature (Table 2). The Pearson correlation coefficients (r) ranged between 0.995 and 0.999 ($P < 0.001$) for these nutrients. The contents of Ca, K, Mg, and P were twice as high in BC-620 as in SS-con. Therefore, these elements are not volatilised at studied temperatures and become concentrated in the resulting biochar. Contrarily, sulphur did not follow the same trend of the remaining nutrients, and the total S content fluctuated around 10 g/kg in all samples analysed demonstrating the volatilisation and consequent release of S from biochar during pyrolysis.

Availability of nutrients as influenced by pyrolysis. Pyrolysis of SS samples at a temperature of 220 °C increased the available contents of all studied

Table 2. Total and available contents of nutrients (g/kg) in sewage sludge and biochar samples

Treatment	Ca		K		Mg		P		S	
	total	available	total	available	total	available	total	available	total	available
SS-con	37.7 \pm 0.27	27.2 \pm 0.23	1.96 \pm 0.04	1.05 \pm 0.01	4.49 \pm 0.01	2.44 \pm 0.00	32.6 \pm 0.30	5.99 \pm 0.00	9.48 \pm 0.39	4.05 \pm 0.01
BC-220	42.3 \pm 0.56	29.8 \pm 0.05	2.28 \pm 0.01	1.08 \pm 0.00	4.95 \pm 0.04	2.56 \pm 0.01	36.4 \pm 0.40	7.31 \pm 0.11	11.2 \pm 0.52	4.71 \pm 0.01
BC-320	51.6 \pm 0.52	24.7 \pm 0.05	2.74 \pm 0.11	0.79 \pm 0.01	5.98 \pm 0.08	2.05 \pm 0.00	44.0 \pm 0.54	7.65 \pm 0.05	11.3 \pm 0.26	3.16 \pm 0.02
BC-420	63.3 \pm 0.51	24.3 \pm 0.06	3.24 \pm 0.17	0.94 \pm 0.00	7.37 \pm 0.08	2.09 \pm 0.01	54.2 \pm 0.67	3.68 \pm 0.01	10.9 \pm 0.13	2.79 \pm 0.01
BC-520	67.7 \pm 0.66	17.0 \pm 0.03	3.41 \pm 0.02	0.62 \pm 0.00	7.87 \pm 0.09	1.37 \pm 0.01	57.8 \pm 0.94	2.34 \pm 0.00	10.7 \pm 0.14	1.12 \pm 0.01
BC-620	72.5 \pm 1.39	13.9 \pm 0.06	3.88 \pm 0.04	0.55 \pm 0.01	8.60 \pm 0.10	1.18 \pm 0.03	60.8 \pm 0.39	1.87 \pm 0.03	11.5 \pm 0.06	0.48 \pm 0.00

Values shown represent the arithmetic mean ($n = 3$) \pm standard deviation. Pyrolysed samples are denoted as BC-220, BC-320, BC-420, BC-520, BC-620 – target pyrolytic temperatures of 220, 320, 420, 520 and 620 °C. SS-con – control; SS – sewage sludge; BC – biochar

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nutrients (Table 2). In comparison with SS-con, the available content in BC-220 increased by 10, 3, 5, 22, and 16% for Ca, K, Mg, P, and S, respectively. At the temperature of 320 °C, available contents of Ca, K, Mg, and S decreased compared to both, SS-con and BC-220. The opposite trend was found for P at 320 °C being the only nutrient studied, whose available content in BC-320 was higher compared to SS-con (by 28%). At the temperature of 420 °C, the available contents of all studied nutrients were lower than in initial SS, and the decline in the availability continued with increasing pyrolysis temperature. A significant drop in the available contents was detectable between 420 °C and 520 °C. For all the studied nutrients, the lowest available contents were found in BC-620. Our results indicated an aromatisation of BC at 420 °C, leading to the suggestion that the availability of mineral nutrients is decreasing not exclusively because of mineral-phase transformations but also due to hydrophobic shielding by aromatic organic phase.

The relative availability of nutrients in the materials expressed as a percentage of available portion of the given nutrient from its total content in the material is shown in Figure 2. The relative availability was found to be the highest in the case of Ca and the lowest in the case of P. No significant changes in the relative

availability of Ca and S were found in BC-220 over SS-con. Phosphorus was the only nutrient whose relative availability increased by pyrolytic treatment at 220 °C compared to SS-con. The increment (by 2%) was slight but statistically significant ($P < 0.05$). We assume that this increment in relative P availability is caused by the cleavage of organically bound P. Starting from the temperature 220 °C; the relative availability of all nutrients was systematically decreasing. The only exceptions were K and S, whose relative availability was not statistically different between BC-320 and BC-420. For all studied nutrients, the lowest relative availability was found in samples pyrolysed at 620 °C. Such an effect may be caused by several factors. Firstly, mineral phase transformations such as dehydration and/or (re)crystallisation of amorphous phases occur during thermal treatment (Huang and Tang 2016). Secondly, the mineral phase of the BC may become protected against polar solvents by organic matter as it becomes progressively hydrophobic with increasing pyrolysis temperature.

Our results suggest that the optimal pyrolysis temperature for SS treatment is 320 °C. Such treatment is relatively less energetically demanding compared to higher temperatures, and at this temperature, resulting material showed the highest content of available P whereas available contents of other studied nutrients were reduced only slightly. At this temperature, SS organic matter was already partly transformed and became more hydrophobic, so higher resistance against microbial degradation and longer residence time in the soil can be clearly expected.

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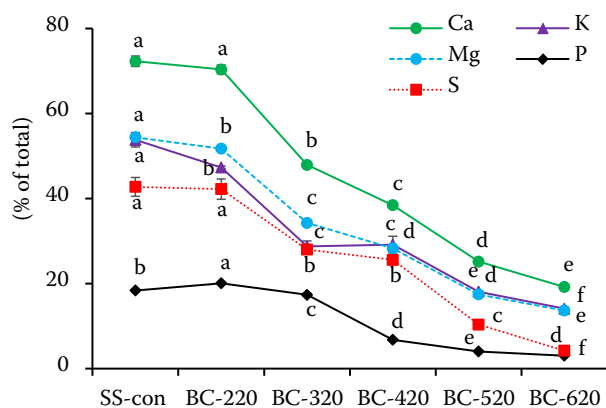


Figure 2. The available proportions of nutrients from their total content in the individual materials. Values shown represent the arithmetic mean ($n = 3$); error bars stand for standard deviation; different letters indicate statistical differences (Tukey's *HSD* (honestly significant difference); $P < 0.05$) between treatments for individual elements separately. Pyrolysed samples are denoted as BC-220, BC-320, BC-420, BC-520, BC-620 – target pyrolytic temperatures of 220, 320, 420, 520 and 620 °C; SS-con – control

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