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ORIGINAL PAPER

Pyrolysis of Dry Toilet Substrate as a Means of Nutrient Recycling in Agricultural Systems: Potential Risks and Benefits

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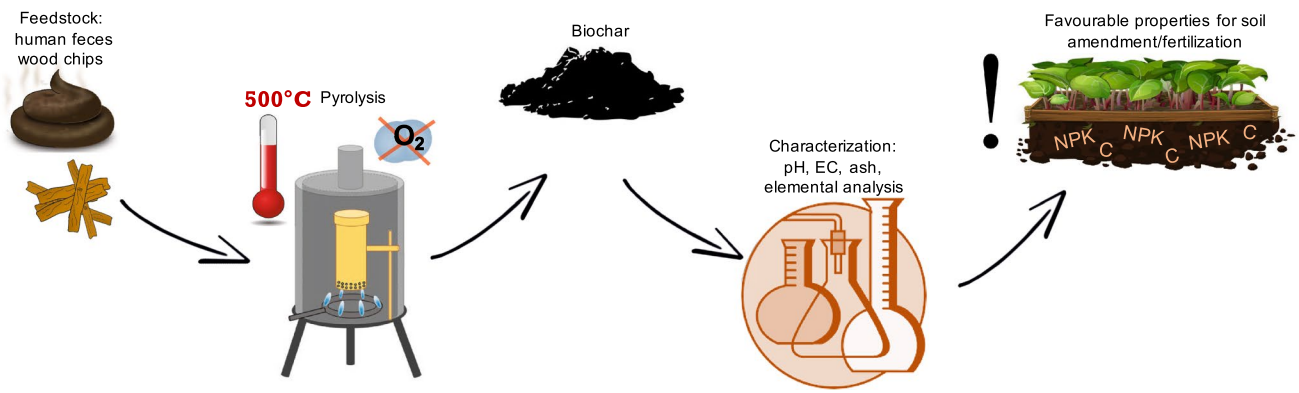
Abstract

Biochar is increasingly being applied as a soil amendment in agriculture. Biochar is typically produced from plant biomass and contains relatively low amounts of plant nutrients (e.g., N, P, and K), thus providing limited fertilizer value. Human excreta contains plant nutrients that could be recycled to create sustainable agricultural nutrient cycles. This study investigated the potential of biochar derived from a dry toilet substrate as soil amendment. The substrate consisted of urine, faeces, and wood chips, and was pyrolyzed at 500–650 °C for 10 min. The biochar was analyzed for plant available P, water leachable P and K, carbon stability, pH, electrical conductivity, polycyclic aromatic hydrocarbons (PAHs), polychlorinated biphenyls (PCBs), dioxins, and germination tests with barley and lettuce were conducted to estimate the biochar fertilizer value and potential bio-toxicity. The biochar contained 25.0 ± 1.0 g N/kg dry mass (DM), 33.1 ± 2.1 g P/kg DM and 20.7 ± 0.2 g K/kg DM. 65% DM P was extractable by formic acid solution, 31.7% DM P and 60.5% DM K were water leachable in a ten-day column water-leaching experiment. The biochar complied with European regulations for PAHs, PCBs, dioxins and heavy metal concentrations, except for Zn and Ni. Germination of salt-resistant barley was not affected by biochar doses < 50% DM, while salt-sensitive lettuce germination was inhibited at doses ≥ 2% DM, indicating that the dry toilet substrate biochar induced salt stress. Based on these results, it is recommended that urine separation should be considered for biochar of excreta, which could reduce salt stress while maintaining concentrations of "fixed" or bioavailable nitrogen.

Graphic Abstract



Keywords Biochar · Dry toilet · Pyrolysis · Sanitation · Germination test · Nutrient recycling

Statement of Novelty

Excreta is a valuable resource and well-managed dry toilets can provide adequate sanitation. In this research, we converted a dry toilet substrate containing urine, faeces, and

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sawdust to biochar, completed a comprehensive characterization, and assessed its impact on plants with two different germination tests. Prior to resource recovery from excreta, pathogens need to be eliminated, which is achieved with high temperatures during pyrolysis. Other than nitrogen, nutrients are largely conserved in the biochar. The results contribute to knowledge of safe management of dry toilet substrate, resource recovery for plant production, and the development of human waste based value chains.

Introduction

A growing world population, fast urbanization and a changing climate pose new challenges when it comes to providing enough drinking water and food for the world's inhabitants, while fresh water resources continue to diminish [1, 2]. The prevailing use of flush toilets, accounting for 20–40% per capita water use in industrialized countries [3], puts stress on the increasingly scarce fresh water resources. With 2.4 billion people remaining without basic sanitation services, sanitation approaches using less water, such as dry toilets or vacuum toilets, should be considered in light of limited water resources [4]. However, waterless sanitation systems result in the production of fecal sludge, which is defined as the "raw or partially digested, semisolid or slurry resulting from collection, storage or treatment of combinations of excreta and blackwater, with or without greywater" [5]. Fecal sludge collected in dry toilets represents a valuable resource of nutrients and organic matter for use in agricultural [6]. However, due to the risk of environmental contamination and protection of public health, faecal sludge should not be used in agriculture without adequate treatment for hygienisation and stabilization [6, 7].

Slow-pyrolysis could be a promising technology to efficiently treat and sanitize fecal sludge from dry toilets. Pyrolysis is defined as a "process of thermal decomposition of carbonaceous organic materials in the complete or near absence of oxygen" [8] and results in a carbon rich biochar. Pyrolysis operates at temperatures of 300 °C or higher, thus, leading to the inactivation of pathogens contained in the feedstock [9]. Nutrients such as phosphorous, potassium, magnesium and calcium in their ionic forms, are not volatilized during pyrolysis, and are maintained in the biochar [10]. Furthermore biochar has the potential to increase the water holding capacity of soils [11]. Another benefit of using biochar as a soil conditioner is as a carbon sink, as the carbon remains stable, thereby reducing the net formation of greenhouse gases [12]. Compared to biochar derived from wastewater sludge, dry toilet substrates can also have lower concentrations of heavy metals [13]. However, there is a lack of research on the use of excreta from dry toilets as pyrolysis substrate. Previous studies have included human faeces [14], and faecal sludge from pit latrines [15] and septic tanks [16], which have different characteristics from dry toilets with excreta and wood chips [7]. This also concerns the environmental safety and the effect of soil biochar addition on the growth of plants and consumption of resulting produce by animals and humans. For example, biochar can contain salts, chemicals (e.g., polycyclic aromatic hydrocarbons (PAHs), polychlorinated biphenyls (PCBs) and dioxins), and heavy metals (e.g., Cd and Pb) that following soil application in high concentrations can be toxic to plants, humans (e.g., following consumption of produce) and the environment (e.g., soil, water) [17–19].

The objective of this study was to evaluate whether slow-pyrolysis is a suitable process for transforming faecal sludge from dry toilets into biochar to be used as a soil conditioner. A dry toilet substrate (DTS) consisting of human feces, urine, toilet paper and wood chips was used as model substrate, and the biochar was analyzed for nutrients, carbon sequestration, and plant bio-toxicity.

Material and Methods

Pyrolysis Feedstock

DTS (mixture of human feces, urine, toilet paper and wood chips) was collected at the storage facilities of Kompotoi (a rental service for dry toilets) in Zurich, Switzerland in March 2016.

The feedstock was collected from a single residential dry toilet without urine separation, which was used by > 10 inhabitants of a shared house. The collected waste had been stored in an airtight vessel for several weeks before sampling. Several random samples were collected from the whole vessel volume (400L). From visual observation, about 25 vol% of the sample consisted of wood chips.

Following sample collection, the feedstock was dried for two days at 60 °C in a laboratory oven, and several small samples were taken from the dried feedstock to form a composite sample for the feedstock analyses.

Slow-Pyrolysis Experiments

Nine batches of the dried DTS were pyrolysed with an externally heated pyrolysis reactor (Fig. 1). The temperature was measured with a temperature sensor (Mantelthermoelement Typ K Otom group, Germany) in the center of the pyrolysis steel cylinder. Temperature was recorded manually each minute.

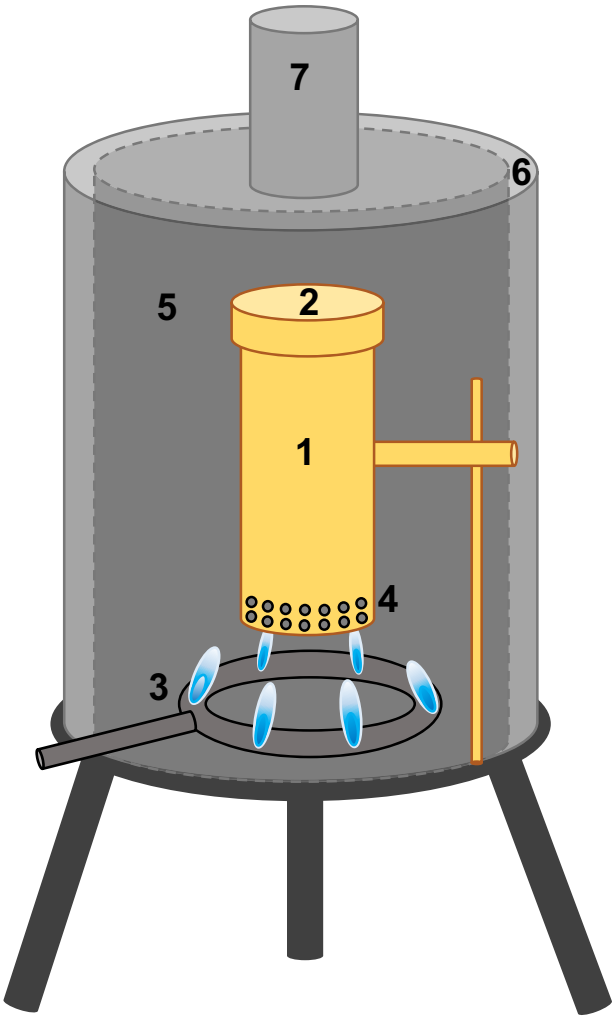

Fig. 1 Schematic diagram of pyrolysis batch reactor. (1) Steel cylinder (Ø:76.1 mm, h:300 mm) containing feedstock (2) screw cap (3) ring burner run with propane gas initiating pyrolysis process (4) perforations for the escape of pyrolysis gases (5) aluminium cover (Ø:353 mm, h:500 mm) (6) insulation wool 50 mm (7) outlet for exhaust gases (8) temperature sensor

The reactor was heated with a ring burner powered by propane gas. After 10 to 15 min, the temperature had reached 500 °C. Regulating the ring burner by hand, the temperature was held constant between 500 and 650 °C for ten minutes, with the highest pyrolysis temperature (between 600 and 650 °C) in all nine pyrolysis runs. After these 10 min, the hot samples were removed from the steel cylinder and air-cooled until they reached ambient temperature. A composite sample for further analysis was formed from the biochar resulting from all nine experiments. The biochar yield was calculated as the quotient of output and input dry weight.

Feedstock and Biochar Analyses

Analyses were conducted at the Swiss Federal Institute of Aquatic Science and Technology (Eawag), Zurich University of Applied Sciences (ZHAW) and the Paul Scherrer Institute (PSI) in Switzerland.

Analysis methods for the biochar in this study were selected based on International Biochar Initiative guidelines [20], the European Biochar certificate [17] and recommendations by Bachmann et al. [21]. For quality control, all samples were analyzed in triplicate. Additionally, two standard biochars (received from the UK Biochar Research Center (UKBRC)) were analyzed along with the biochar to assure the accuracy of the analysis methods (for details see Supplementary Material). Where applicable, the feedstock was analyzed with the same methods as the biochar. For an overview of all conducted analyses, see Table 1.

Before analysis all samples (feedstock and biochar) were 100% dried in a laboratory oven (Binder FDL 115, Germany) at 105 °C according to Standard Methods [22] and ground with a kitchen mixer (Nutribullet Migros, Switzerland). pH and electrical conductivity (EC) were analyzed in solution with a pH-meter (Hach-Lange PHC301, Switzerland) and an EC-meter (Hach-Lange CDC401, Switzerland) according to DIN ISO 10,390 [23]. pH was analyzed in 2.5 g of sample mixed with 25 mL 0.01 M $CaCl_2$ for one hour, and EC was analyzed in the filtrate of 2 g sample mixed with 20 mL deionized water for one hour. Volatile solids in the samples were analyzed gravimetrically according to ASTM D1762-84 [24] as the mass loss after heating under oxygen restriction to 950 °C for 7 min in a muffle furnace (Nabertherm L3, Germany). Ash was determined gravimetrically as the residue following combustion in the same muffle furnace according to EBC (2012). Fixed carbon content was calculated according to ASTM D3172 [25] as 100 (% DM) − Volatile solids (% DM) − Ash content (% DM) = Fixed carbon content (% DM).

Carbon (C), hydrogen (H), nitrogen (N), sulfur (S) and oxygen (O) were analyzed with a CHNSO analyzer (Leco, Tru Spec Micro, USA) according to DIN 51,732 (2014). Carbon stability was estimated as the carbon remaining from 0.1 g of carbon following digestion with 0.01 M hydrogen peroxide (H_2O_2) and 7 mL of deionized water at 80 °C for 144 h according to Cross and Sohi [26]. Nutrients analyzed in this research included nitrogen (N), potassium (K), phosphorus (P), sulfur (S), Calcium (Ca), Iron (Fe) and plant available phosphorus (Plant-P). Phosphorus and potassium were analyzed with Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES) following microwave digestion (ultraCLAVE 4, MLS GmbH, Germany) at 250 °C and 120 bar for ten minutes of 0.2 g sample with 5 mL nitric acid (HNO_3), 1 mL H_2O_2 and 0.3 ml hydrofluoric acid (HF). Plant-P was estimated as the phosphorus extractable by

Table 1 Characteristics of 100% dry toilet substrate (DTS) and DTS-biochar

	Feedstock (DTS)	DTS-biochar	Element conservation after pyrolysis
Biochar yield [% DM]	–	29.7 (±0.8)	
Volatile solids [% DM]	76.5 (±0.4)	12.3 (±0.3)	
Ash content [% DM]	8.2 (±0.1)	42.0 (±2.7)	
Fixed carbon [% DM]	15.4 (±1.4)	45.7 (±6.4)	
C [% DM	44.3 (±0.5)	61.9 (±1.0)	41%
H [% DM]	6.7 (±0.1)	1.6 (±0.1)	7%
N [% DM]	2.2 (±0.0)	2.5 (±0.1)	33%
P [g/kg DM]	8.9 (±0.7)	33.1 (±2.1)	109%
K [g/kg DM]	5.5 (±0.1)	20.7 (±0.2)	110%
Ca [g/kg DM]	10 (±0.7)	4.6 (±0.1)	14%
Na [g/kg DM]	5.1 (±0.1)	18.7 (±0.0)	107%
Fe [g/kg DM]	0.2 (±0.0)	1.0 (±0.1)	160%
Cl [g/kg DM]	9.1 (±0.6)	24.9 (±0.6)	80%
S [g/kg DM]	3.2 (±0.3)	3.7 (±0.2)	35%
As [mg/kg DM]	< 12.5	< 12.5	–
Cd [mg/kg DM]	< 12.5	< 12.5	–
Cu [mg/kg DM]	21.7 (±0.7)	82.0 (±1.6)	111%
Cr [mg/kg DM]	< 12.5	73.4 (±28.4)	–
Pb [mg/kg DM]	< 12.5	< 12.5	–
Ni [mg/kg DM]	< 12.5	81.3 (±18.2)	–
Zn [mg/kg DM]	188.8 (±3.1)	606.8 (±3.6)	94%
C-stability[a] [%]	NA	97.6 (±1.6)	
pH [−]	NA	10.6 (±0.1)	
EC[b] [mS/cm]	NA	9.7 (±0.5)	
Plant-P[c] [g/kg DM]	NA	20.5 (±0.1)	
PAHs[d] [mg/kg DM]	NA	3.2	
Dioxins [mg/kg DM]	NA	1.98	
PCBs[e] [mg/kg DM]	NA	< 0.06	

Standard deviations are included in parentheses for analyses conducted in triplicate

[a]Carbon stability

[b]Electrical conductivity

[c]Plant available phosphors (formic acid extractable phosphorous)

[d]Polycyclic aromatic hydrocarbons

[e]Polychlorinated biphenyls

[N]*NA* not analyzed

formic acid digestion according to Wang (2013) and IBI (2014). Plant-P was analyzed in the filtrate of 0.35 g sample and 35 mL CH_2O_2 following 30 min of mixing with the vanadomolybdate colorimetric method [27]. Chlorine analysis was conducted by first digesting the sample according to Schöninger [28], thereby transferring covalent chlorine to ionic chlorine. Ionic chlorine was then analyzed in the adsorption solution with ion chromatography according to DIN EN ISO 16,994:2015–7 [29].

Heavy metals analyzed in this study included chromium (Cr), copper (Cu), nickel (Ni), lead (Pb) and zinc (Zn). They were analyzed with ICP-OES after digestion in the same way as total phosphorus and potassium. PAHs, PCBs and dioxins were determined by external laboratories (Eurofins Umwelt, and LUFA Nord-West, Germany) according to standard methods [17].

The element conservation after pyrolysis was calculated as

$$Element\ conservation\ [\%DM] = \frac{Element\ content\ in\ biochar\ [\%DM] \times yield\ [\%DM]}{Element\ content\ in\ feedstock\ [\%DM]} \times 100$$

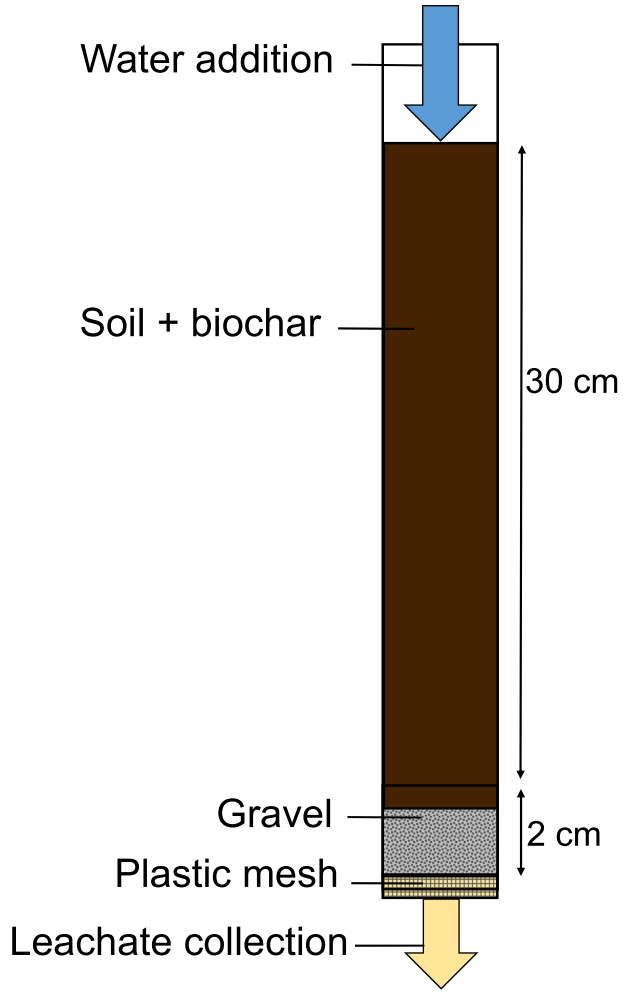

Fig. 2 Cross section of soil column used for leaching experiment

Water-Leaching Experiments

Soil column leaching experiments were conducted to study the water-leaching of nutrients from the biochar when incorporated at a commonly used dosage into a moist soil matrix. The experiment was setup in a university greenhouse of ZHAW. Following the model of Laird et al. [30], columns were made out of 40 cm long plastic pipes with a diameter of 2 cm (Fig. 2). A gravel layer of 2 cm was added at the bottom of each column to facilitate drainage and a fine plastic mesh (2 mm) was placed at the bottom of the columns.

The soil chosen as a reference substrate was a black peat substrate with low nutrient content (Proter + Profi Torf, Fenaco, Switzerland). The initial peat substrate had a pH of 2.3 which was adjusted to 7.7 by liming with 4.5 kg/m^3 calcium carbonate ($CaCO_3$) and 2 kg/m^3 calcium hydroxide ($Ca(OH)_2$). pH values both before and after liming were measured according to the method described above. Following liming, the peat was sieved through a 1 cm mesh, resulting in a substrate with a dry bulk density of approximately 0.16 kg/L.

Three of the columns were filled with the control substrate described above. For each column, 35 g of dry reference substrate was wetted with 50 mL of deionized water and thoroughly mixed. Three additional columns were filled with a biochar-containing substrate. 1.75 g of biochar was manually mixed with 35 g of dry reference substrate. The substrate was then wetted with 50 mL of deionized water. This biochar dose corresponds to 5% DM or 56 t biochar/ha with an application depth of 30 cm.

Substrates were added to the column in five portions. After the addition of each portion, the substrate was compacted with a wooden rod. This resulted in a depth of 30 cm of the pipe length being filled with substrate (corresponding to a volume 94.3 mL).

Two days after the column preparation, 60 ml of deionized water was added to each column and the leachate was collected in analytical tubes. After one hour, pH and EC in the water-leachates was analyzed with a multi-probe (HQ40D, Hach-Lange, Switzerland). Afterwards, the water-leachate was stored in sealed tubes at 5 °C for further analysis. On the following nine days, 30 mL of deionized water was added and collected daily. pH and EC were analyzed in all water-leachates one hour after the addition of water.

Anions (Cl^-, NO_2^-, NO_3^-, PO_4^{3-}, SO_4^{2-}) were analyzed in the water-leachates with ion chromatography with an 881 Compact IC pro (Metrohm, Switzerland) including an 858 Professional Sample Processor (Metrohm, Switzerland) according to DIN EN ISO 10,304–1 [31]. Cations (Na^+, NH_4^+, K^+, Ca_2^+, $Mg2^+$) were analyzed with a 761 Compact IC (Metrohm, Switzerland) including a 766 IC Sample Processor (Metrohm, Switzerland) according to DIN EN ISO 14,911 [32]. The limit of detection was < 1 mg/L for anions and < 5 mg/L for cations.

Germination Tests

Germination tests were performed to assess plant-toxicity of DTS-biochar. Previous research concluded that germination inhibition induced by carbonaceous soil additives (e.g. biochars) could have two reasons: firstly, salinity stress caused by high concentrations of soluble salts in the biochar, and secondly, toxicity of certain biochar components (e.g. heavy metals, PAHs) [33]. To consider both reasons for bio-toxicity, the use of two germinations tests has been suggested to assess biochars [33]. Therefore, the authors analyzed the germination of a salt sensitive plant (lettuce) to test salt toxicity and the germination of a salt resistant plant (barley) to test other potential toxins.

Lettuce and barley germination tests are used to assess bio-toxicity potentially originating from carbonaceous soil additives such as biochars [33]. The lettuce and barley

germination tests conducted in this study, including biochar doses, substrates and climate chamber settings, were chosen according to methods proposed by Busch et al. [33].

For the lettuce germination test, doses of 2 g, 4 g, 8 g and 16 g of biochar were thoroughly mixed with washed river sand to achieve a total mass of 100 g (2, 4, 8 and 16% DM). Three replicates were produced for each dose and compared to a control with no biochar addition. Each mixture was placed into a large petri dish (Ø 15 cm). Then, 40 lettuce seeds (*Lactuca sativa analena*, Rijk Zwaan Welver GmbH, Germany), pelleted and dressed with thiaram, were placed in the petri dish. The petri dishes were wetted with tap water to the maximal water holding capacity of the sand and covered with a lid.

For the barley test doses of 2.5 g, 5 g and 12.5 g (5 vol%, 10 vol% and 25 vol%) of biochar were thoroughly mixed with the peat reference substrate (see above) to produce a total volume of 100 mL. Three replicates were produced for each dose and compared to a control with no biochar addition. Each mixture was placed into a transparent plastic vessel. 15 barley seeds (*Hordeum vulgare,* Probstdorfer Saatzucht GesmbH & CoKG, Austria) were planted approximately 0.5 cm deep into the substrate. The plastic vessels were wetted with tap water to the maximal water holding capacity of the substrate and covered with a lid.

All petri dishes and plastic vessels were placed in a climate chamber (KE 601, Klimavent Ag, Switzerland) at 20 °C with a 12 h/12 h day/night cycle at 3000 lx. The samples were wetted with tap water daily. Lids were removed after four days. After seven days (lettuce) and ten days (barley), the germinated seeds were counted and the biomass above the substrate surface was collected using scissors. The wet weight of the biomass was directly determined using a laboratory scale.

Differences between mean values in germination tests were statistically analyzed with one-way analysis of variance (Anova) and Tukey's honest significant difference (HSD) test on a confidence interval of 95%.

Results and Discussion

Feedstock Characteristics

Characteristics of the feedstock (DTS) are presented in Table 1. Dried DTS had 76.5 (±0.4) % DM volatile solids, 8.2 (±0.1) % DM ash and 15.4 (±1.4) % DM fixed carbon. The ash content of the DTS was noticeably lower than in fecal sludge (47–58% DM [13]) and wastewater sludge (40–57% DM [13]). Fecal and wastewater sludge often contain inorganics from sources other than feces like grit, sand or municipal solid waste [34, 35]. Reported ash content of feces are in the range of 8–16% DM [36], which corresponds to the results of this study. The ash content of DTS-biochar being in the lower range of the values reported for feces by Rose et al.[36], can further be attributed to wood chips contained in the DTS, as wood has typically an ash content < 4% DM [18].

The carbon content of DTS was 44.3 (±0.5) % DM, which is higher than values reported for fecal sludge (28–29% DM) and wastewater sludge (17–32% DM [13]). DTS carbon content was in the same range as in human feces (44 and 55% DM [36]).

Nutrient concentrations of the DTS were 22.0 (±0.0) g N/kg DM, 8.9 (±0.7) g P/kg DM and 5.5 (±0.1) g K/kg DM. These nutrient concentrations are somewhat lower than the N, P and K concentrations reported for feces and fecal sludge (46 g N/kg DM, 27 g P/kg DM, 10 g K/kg DM [37] and 59 g N/kg DM, 31 g P/kg DM, 12 g K/kg DM [16]). However, the N and P concentrations of DTS were in the range of values for compost that qualifies as fertilizer (20–28 g N/kg DM, 7–10 g P/kg DM [38]).

Heavy metal concentrations of 188.8 (±3.1) mg Zn/kg DM, < 5 mg/kg DM Cd, and < 12.5 mg/kg DM for Cu, Cr, Pb and Ni in the DTS substrate were lower than values reported for fecal sludge or wastewater sludge [13]. In summary, in comparison to reported values for fecal sludge and wastewater sludge, DTS in this study had higher carbon concentrations, and lower heavy metal and ash concentrations. Together with nutrient concentrations, DTS was determined to be a suitable feedstock for the production of biochar for use as fertilizer or soil amendment in agriculture and horticulture.

Pyrolysis Performance

Characteristics of the DTS-biochar are presented in Table 1. The biochar yield in this study was 29.3% DM. Yield generally depends on heating rate, pyrolysis temperature and residence time of the feedstock in the reactor [39]. Higher temperatures and higher heating rates usually cause more compounds to volatilize, leading to a lower yield [39]. The yield in this study was comparable to biochar yields with human feces produced at 450 °C and 750 °C (29 and 30% DM [14]). Biochar production from fecal sludge at 500 °C, 600 °C and 700 °C also had comparable yields of 35, 31 and 31% DM [16]. Char yield is a useful parameter for the dimensioning of pyrolysis reactors destined for a specific purpose such as DTS pyrolysis.

Potential Risks of DTS-Biochar Soil Application

The potential risks of using DTS-biochar as a soil amendment were assessed by measuring heavy metals, toxins (i.e., PAHs, PCBs, dioxins), and inorganic salt contents in the DTS biochar and comparing these results to guideline

values. These parameters were selected for their potential effect on human and environmental health. In addition to the biochar characterization, germination tests were performed to assess the toxicity of biochar addition to soil substrates for plants.

Heavy Metals

The selection of heavy metals for analysis was based on the European Biochar Certificate (EBC), and included As, Cd, Cr, Cu, Ni, Pb and Zn [17]. The EBC provides guideline limits for parameters for certification of biochar for soil applications. Heavy metal concentrations and bioavailability in soils are key metrics to assess potential negative effects. Total heavy metal concentrations in the DTS-biochar were all lower than the EBC recommendations, except for Ni and Zn (limit values of < 50 mg Ni/kg, and < 400 mg Zn/kg respectively), and the EBC guideline for Cd (< 1.5 mg Cd/kg) was below the detection limit in this study (< 12.5 mg Cd/kg). Heavy metal concentrations in the DTS-biochar were < 12.5 mg As/kg DM, 73.4 (± 28.4) mg Cr/kg DM, 82.0 (± 1.6) mg Cu/kg DM, < 12.5 mg Pb/kg DM, 81.3 (± 18.2) mg Ni/kg DM and 606.8 (± 3.6) mg Zn/kg DM. Based on previous results with biochar from wastewater sludge [40–42], Cd, Ni, and Zn are most likely less bioavailable in the char than in the feedstocks, reducing the risk of heavy metal toxicity to plants and transmission to food products.

Overall, heavy metals in DTS-biochar were much lower than reported for biochars derived from wastewater sludge. Previously reported Cu concentrations exceeded 390 mg Cu/kg DM and were as high as 1500 mg Cu/kg DM, while Zn concentrations were above 1430 mg Zn/kg DM up to a maximum of 3922 mg Zn/kg DM [41, 43, 44]. Khanmohammadi et al. [43] further reported high Cr and Pb concentrations (96–178 mg Cr/kg DM and 132–152 mg Pb/kg DM). Especially, Cr and Pb concentrations are a concern, as they are toxic for human and environmental health [45].

In conclusion, although the DTS-biochar did not fully comply with EBC heavy metal guidelines, the heavy metal concentrations were lower than in biochars produced with wastewater sludge. This highlights that feedstocks from well-managed household on-site sanitation systems such as dry toilets could be more suitable feedstocks for nutrient recovery by pyrolysis over wastewater sludge from sewer-based systems. Heavy metals can contaminate fecal sludge during containment, if toilets are also used for disposal of materials other than fecal sludge (e.g. batteries, solvents), or fecal sludge collection and transport if vacuum trucks are also being used for transport of other materials than fecal sludge [13, 46, 47]. Hence, education on the appropriate use of toilets is important. Heavy metals can also originate from human feces or wood chips [35]. Whereas in wastewater sludge, heavy metals can also come from sources such as storm water run-off or industrial sources, which are difficult to mediate.

Biochar Toxins

Toxin concentration in the DTS biochar was below the EBC limits (Table 1, [17]). Dioxins were 1.98 ng/kg DM and PCBs below the detection limit of 0.060 mg/kg DM. These concentrations are below the EBC limits of 20 ng/kg DM and 0.2 mg/kg DM respectively.

Naphthalene, phenanthrene, and pyrene were PAHs detected at 2.7 mg/kg DM, 0.4 mg/kg DM and 0.1 mg/kg DM respectively, below the EBC limit of 4 mg/kg DM. Naphthalene and phenanthrene are PAHs typically found in biochars in the highest concentrations [21]. Naphthalene in the DTS-biochar could originate from the wood content in the feedstock, as it is known to be formed during the uncontrolled burning of wood [48]. PAH formation could potentially be reduced by a more controlled pyrolysis process, as PAHs commonly are formed from organic matter during incomplete pyrolysis conditions [49]. All dioxin, PCB and PAH results are included in the Supplementary Material.

Biochar Plant Toxicity

As depicted in Fig. 3, DTS-biochar did not have a negative effect on barley seed germination, with the count of germinated seedlings being equal to the control treatment. The above ground biomass of seedlings in the treatment containing 2.5 vol% biochar was significantly higher than in the control treatment, and at higher doses the above ground biomass of seedlings was not significantly different from the control. These results are in line with those of previous studies conducted with plant-based substrates with similar methodologies as summarized in Table 2. The results of the barley germination tests indicated that the DTS-biochar produced in this study did not have plant toxicity.

However, in contrast to the barley germination tests, the lettuce seed germination tests indicated that DTS biochar can result in salt stress of plants. Above ground biomass was negatively affected by biochar addition at all doses in comparison to the control, and no germination at all was observed for biochar doses higher than 8% DM. Although there were no detectable differences in germination count between the control treatment and the treatment containing 2% DM and 4% DM of biochar, the above ground biomass was significantly lower at these biochar doses in comparison to the control. The DTS-biochar salt content in this study was EC 9.7 mS/cm, which is higher than reported for biochars that did not observe an effect on lettuce germination (EC 4.8 mS/cm [19]). The application of DTS-biochar could result in salt stress in salt-sensitive plants, and is also

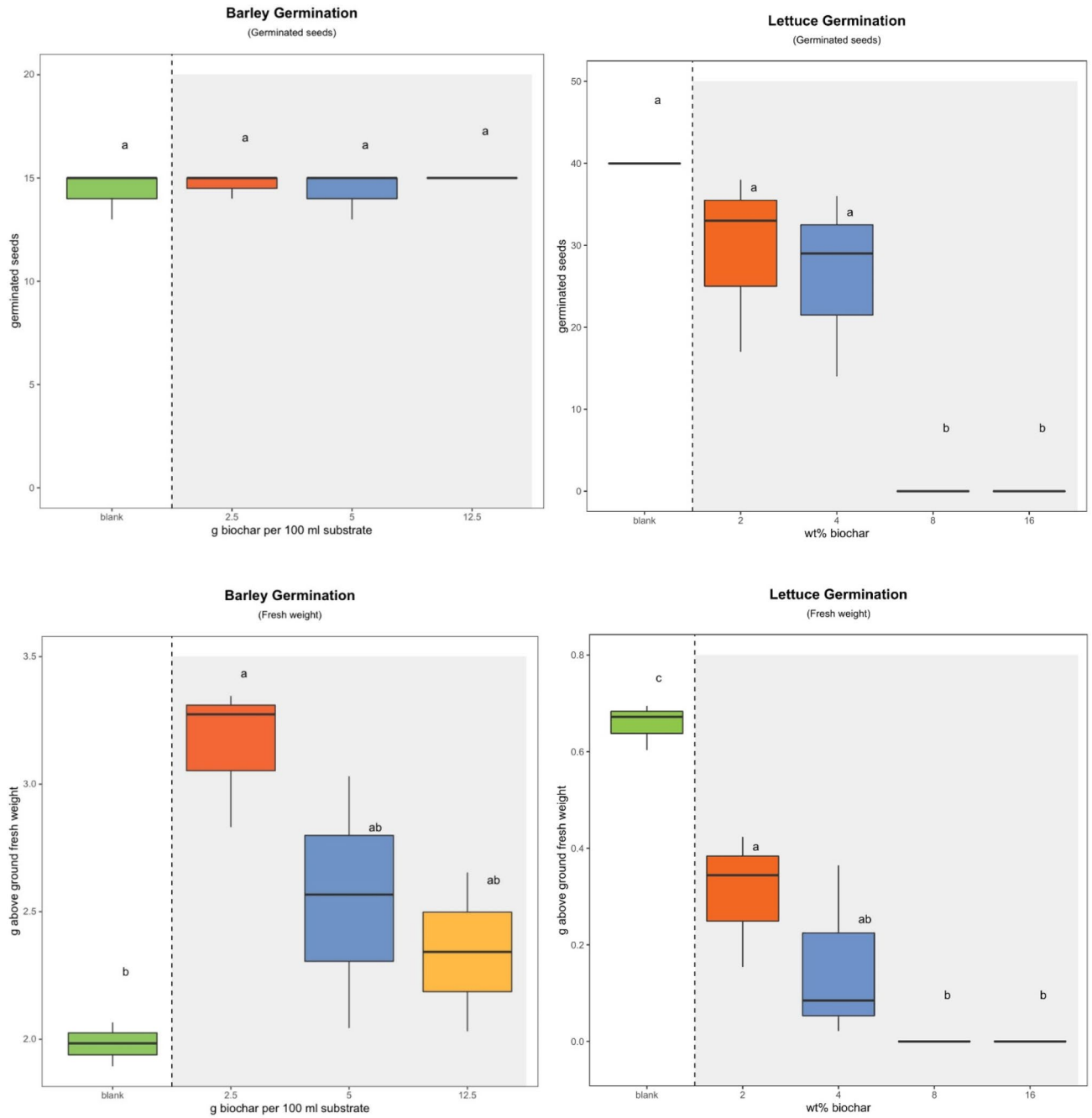

Fig. 3 Boxplot of barley and lettuce germination count and fresh above ground biomass after germination tests as a function of biochar dose. Error bars represent standard deviations of triplicate trials. Letters represent differences as determined by one-way Anova and Tukey's HSD-test (confidence level of 95%)

Table 2 Germination tests results of this study in comparison to previous research performed with plant-based biochars [18, 19]

	Barley germinated seeds	Barley above ground biomass	Lettuce germinated seeds	Lettuce above ground biomass
DTS biochar (this study)	No negative effect	No negative effect	Negative effect starting at 8% DM biochar	Negative effect starting at 2% DM biochar
Peanut hull biochar [18]	No negative effect	No negative effect	No negative effect	–
Argan shell biochar [19]	No negative effect	No negative effect	No negative effect	No negative effect

a concern for soil health. The high salt concentrations in urine [36] were likely the cause of high salts in the DTS-biochar. Urine separation could be a solution to keep salt out of biochar, and also reduce the total amount of nitrogen that is lost due to volatilization during pyrolysis (Table 1) [50]. The urine could then be processed separately for use as a fertilizer, maintaining the overall fixed nitrogen content [51].

Potential Benefits of DTS-Biochar Soil Application

DTS-biochar contained high enough concentrations of nutrients to be considered a fertilizer. In addition, the DTS-biochar had a pH of 10.6. A high pH is very common in biochars regardless of the feedstock [22, 52, 53] which can contribute to a liming effect if applied to soils [54]. The primary macronutrients were contained in the DTS-biochar with concentrations of 25.0 (±1.0) g N/kg DM, 33.1 (±2.1) g P/kg DM and 20.7 (±0.2) g K/kg DM. Further nutrients S, Ca, Fe were present in concentrations of 3.7 (±0.2) g S/kg DM, 4.6 (±0.1) g Ca/kg DM and 1.0 (±0.1) g Fe/kg DM respectively. In comparison, biochars from woody feedstocks have typically lower concentrations of macronutrients (i.e., N, P, and K), their positive effect on soil fertility is attributed rather to effects like increasing cation exchange capacity, nutrient retention, water holding capacity, and elevation of soil pH [55].

This N concentration in the DTS-biochar was lower than reported for biochar derived from human feces (48 g N/kg DM [14]), but was comparable to fecal sludge derived biochars (24–29 g N/kg DM [16]) and wastewater sludge biochars (9–19 g N/kg DM [56]). These reported biochars were all produced at similar pyrolysis temperatures (450–700 °C) to the one used in this study. The lower concentration of N in DTS-biochar could be due to the addition of wood in the toilets, which is low in N [18, 57, 58].

The average element conservation after pyrolysis was calculated for each element (Table 1). Elements that were not fully conserved during pyrolysis (conservation < 80%) were C, H, N, S and Ca. C, H, N and S elements can be lost due to transfer to the gas phase in the pyrolysis process, forming CO_2, H_2 and N_2 and various sulfuric compounds [59]. Calcium most likely is not transferred to the gas phase but its loss could be explained by incomplete analysis. Incomplete sample digestion was indicated by solid residues after the digestion by 5 mL HNO_3, 1 mL H_2O_2 and 0.3 ml HF (see Supplementary Material). One possible explanation is the formation of calcium silicates during the pyrolysis process. These compounds are usually produced under pressure at temperatures > 170 °C [60]. Some silicates require high dosages of hydrofluoric acid to be decomposed [61] and they might not have been sufficiently digested.

Due to N-volatilization, the NPK-ratio of the unpyrolysed DTS (4:1.6:1) shifted towards P and K in the DTS-biochar (1.2:1.6:1), meaning it would have to be used as a fertilizer accordingly.

Phosphorous and potassium concentrations of 33.1 (±2.1) g P/kg DM and 20.7 (±0.2) g K/kg DM respectively in DTS-biochar were lower than reported in biochar derived from fecal sludge at temperatures of 450–700 °C (78–81 g P/kg DM and 26–28 g K/kg DM [16]). P was present in the same order of magnitude in DTS-biochar as in wastewater sludge biochars produced at 450–700 °C (34–60 g P/kg DM [62]; 44–49 g P/kg DM [56]). K concentrations were significantly higher in the DTS-biochar than in the comparable wastewater sludge derived biochars (2–3 g K/kg DM [62]; 10–16 g K/kg DM [56]). As all alkali salts, most potassium compounds are highly water soluble, which could explain that in water-based wastewater transport and treatment, most K^+ ions end up in the water phase and not in wastewater sludge.

In addition to total concentrations of macronutrients, bioavailability is also important. Water-soluble nutrients are generally considered directly plant available, but since plant roots often produce a slightly acidic microenvironment in the soil [63], more nutrients are extracted from the soil at low pH and become plant available. The formic acid extractable phosphorous method used in this study accounts for this circumstance. It was developed as a correlation between actual plant phosphorous uptake from various high ash biochars (feedstocks including manure and wastewater sludge) and various laboratory phosphorous extractions [64]. The DTS-biochar contained 33.1 (±2.1) g P/kg total P DM of which 20.5 (±0.1) g P/kg DM were formic acid extractable P (plant-P). This suggests that 65% DM of the phosphorous contained in the DTS-biochar is plant available. This was lower than values for biochars derived from undigested cattle manure and wood chips (72–83% DM [64]), but higher than in biochars derived from digested feedstocks such as wastewater sludge (26–37% DM [64]) or digested pig manure (23–32% DM [65]).

The formic acid extraction method is a metric to estimate the total extractable nutrients, without considering their release over time. The ten-day water-leaching experiment conducted as part of this research provides information on nutrient leaching from DTS-biochar. Experiments were performed only with water and no acid was used to extract nutrients. Nevertheless, high amounts of the total phosphorous and potassium contained in the biochar were leached (Fig. 4b). After a leaching period of ten days, 31.7% DM phosphorous contained in the biochar had leached as PO_4-P. In the same period, 60.5% DM of potassium contained in the biochar had leached. Leaching patterns reveal that PO_4-P was released at an almost steady rate over a period of six to seven days, pointing to a relatively slow water-leaching of PO_4-P (Fig. 4a). K on the other hand was leached more quickly with rapidly decreasing concentrations

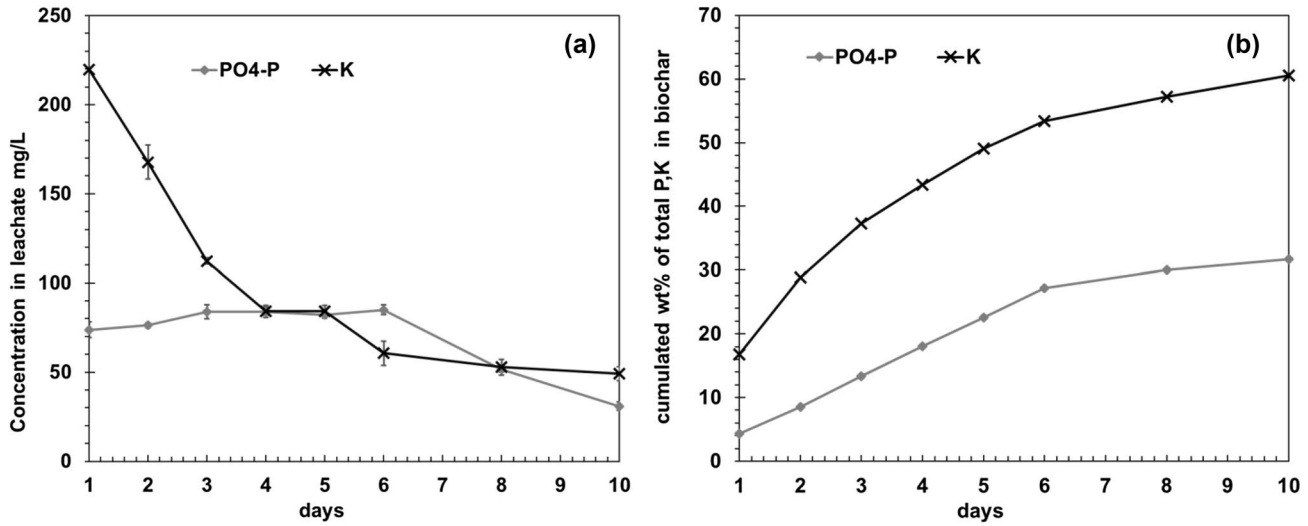

Fig. 4 P and K leaching patterns in a ten-day water-leaching experiment with soil columns containing 5% DM DTS-biochar. Concentration in water-leachate (**a**) and cumulated % DM of total P, K in biochar (**b**). Data points represent the mean of triplicate trials corrected for P, K leached from the reference substrate. Error bars represent standard deviations of triplicate trials in non-cumulated data

in the water-leachates (Fig. 4a). For both PO_4-P and K, water-leachate concentrations decreased over the course of the experiment. Despite the large amounts of water added during the experiment both ions were still detected in the water-leachates at the end of the experiment, indicating that the substrate contained residuals of both ions (Fig. 4a). The total amount of water added to the columns corresponded to 1050 mm of rain.

The gradual release of phosphorous from the biochar could have the advantage of supplying phosphorus to soils at a slow rate and, at the same time, avoiding sudden nutrient leaching to soil or surface waters causing eutrophication [66]. Leaching patterns of all other ions as well as pH and EC values of the water-leachates are included in the Supplementary Material.

Implications

DTS-biochar contained significant macronutrients with 65% DM of plant available phosphorous. However, the P leached relatively slowly indicating it is not as bio-available. This could also prevent high phosphorous run-off when DTS-biochar is used as a soil conditioner. DTS-biochar therefore has the potential to partially replace mineral NPK-fertilizers if applied at suitable doses and considering soil pH. While in acidic soils (pH < 4–6) DTS-biochar additions could be beneficial, excessive application of DTS-biochar could lead to an unfavorably high pH, which is linked to the formation of NH_3 and subsequent N loss from the soils [67].

As pyrolysis emits gaseous carbon and nitrogen, from a nutrient perspective, composting of the DTS substrate may be more valuable over slow-pyrolysis [68]. However, the net of fixed carbon of N as N_2 during pyrolysis could potentially be mediated with the separate collection of urine.

Another benefit of biochar could be carbon sequestration [17, 69]. It is established that a portion of the carbon in biochar is in a stable aromatic form that does not decompose in soils for periods of over 100 years [70]. A high total carbon content and a high carbon stability (aromaticity) lead to the highest carbon sequestration potential [71]. The DTS-biochar had a total carbon content of 61.9 (± 1.0) % DM and a carbon stability of 97.6 (± 1.6) %. Therefore, per kg of biochar introduced into soil, 604 g (60.4% DM) would be expected to remain sequestered in the soil after a period of 100 years. The total carbon content in the DTS-biochar was higher than reported from biochars from human feces (51% DM [14]), fecal sludge (36–42% DM [16]) and wastewater sludge (53% DM [40]). This can be attributed to wood chips contained in the DTS-feedstock, as wood feedstocks usually result in biochars with carbon contents > 60% DM if pyrolysed at > 400 °C [18]. The carbon stability of the DTS-biochar is comparable to values from standard biochars produced and tested by the UK Biochar Research Centre, the developers of the carbon stability analysis method used in this study [72].

Considering a daily excretion of about 29 g feces/capita (dry weight) and a carbon content of roughly 44–55% DM in feces [36], a total amount of about 15 g C/capita/day is excreted. Based on the results of this study, considering a biochar yield of 30% DM and a carbon stability of > 95%, the yearly carbon sequestration potential when pyrolysing human feces is about 1.6 kg C/capita. The yearly CO_2 emission, on the other hand, was 4970 kg CO_2/capita (or 1400 kg

C/capita) [73]. Pyrolysing human feces could therefore neutralize around 0.11% of the human carbon emissions (not taking into account CO_2-emissions from feces collection and biochar distribution and application). The small carbon sequestration potential could be enhanced when pyrolysing DTS that contains a carbon source, such as woodchips from industrial waste or green waste from landscape maintenance [74], as performed in this study. Although the carbon sequestration potential is small, combined with resource recovery beneficial, it could be an additional incentive for the pyrolysis of DTS.

Conclusions

DTS-biochar has potential benefits when applied to soils in agricultural or horticulture, including carbon sequestration and nutrient recovery. Relatively high nutrient contents, low concentration of toxic elements (i.e., heavy metals, PAHs, PCBs, dioxins) and a carbon stability comparable to other biochars indicate that DTS-biochar is suitable for soil application. However, mixing with other biochars or compost, and/or separate collection of urine, is needed to reduce plant salt stress. Further research could confirm the benefits discussed in this research in field applications with different soils and crops under varying climate conditions. Such studies could provide insight as to which conditions the use of DTS-biochar in soils is recommended, and how the biochar could be tailored (e.g. pyrolysis temperature) provide optimal characteristics for specific soils.

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