Research article

Optimising pyrolysis conditions for high-quality biochar production using black soldier fly larvae faecal-derived residue as feedstock

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ABSTRACT

The disposal of feacal matter from Urine Diversion Dry Toilets is a significant challenge due to limited land availability, possible underground water contamination, and the risk of spreading diseases. The collected faecal matter can be fed to Black Soldier Fly Larvae to produce protein-rich larvae used as animal feed. The disposal of the leftover waste (BSFL residue) is still a problem due to the risk of residual pathogen contamination. The BSFL residue contains residual plant nutrients and can be further processed into biochar. Faecal matter biochar offers an exciting value proposition where the pyrolysis process guarantees a 100% pathogen elimination. It also results in significant waste reduction in transport, storage weight, and volume. A preliminary study was conducted to (i) optimise pyrolysis conditions (optimal temperature treatment and residence time) for biochar production using residue obtained after faecal matter from urine diversion dry toilets was fed to black soldier fly larvae as feedstock; and (ii) determine the physicochemical and morphological characteristics of biochar produced. The residue was pyrolysed at 300, 400, and 500 °C and characterised for chemical, biological and physical characteristics. Surface area (6.61 m^2 g^{-1}), pore size, and C: N (9.28) ratio increased at 500 °C for 30 min. Exchangeable bases, (Calcium) Ca, (Magnesium) Mg, (Potassium) K, and (Sodium) Na increased with increasing pyrolysis temperature. The increase in basic cations resulted in an increase in pH from 6.7 in the residue to 9.8 in biochar pyrolysed at 500 °C. Biochar pyrolysed at 500 °C can therefore be used to improve acidic soils. Phosphorus increased with increasing pyrolysis temperature to 3 148 mg kg^{-1} at 500 °C. Biochar produced at 500 °C for 30 min had desirable characteristics: surface area, exchangeable bases, and pH. Also, biochar can be used as a phosphorus source with potential for crop production, although an external nitrogen source is needed to meet crop nutrient requirements.

1. Introduction

Biochar is a stable carbon (C) rich material made through pyrolysis — the heating of any organic material (feedstock) at high temperatures ranging from 300 to 700 °C under oxygen-limited conditions (Lehmann and Joseph, 2015). The quality of biochar depends on the type of feedstock, pyrolysis temperature, and residence time (Tomczyk et al., 2020). Biochar can be produced from a wide range of feedstock such as crop residues (wheat straw, maize straw), woody material with a recent focus on invasive plants, algae, fruit peels, animal manure, and wastewater sludges (Hassan et al., 2020; Ippolito et al., 2020; Zhao et al., 2019). Faecal matter from Urine Diverting Dry toilets (UDDTs) can potentially be used as feedstock for pyrolysis. There is increasing interest in faecal matter pyrolysis to address problems of faecal disposal in filled-up pits (Zhao et al., 2019).

Faecal matter from UDDTs can also be fed to black soldier fly larvae (BSFL) to produce protein-rich larvae used as animal feed (Banks et al., 2014; Maleba et al., 2016). The process generates residual waste, comprising of partly digested material, residual larvae, and larvae droppings. The leftover residual waste can be used as a feedstock to produce biochar. Mostly biochars from faecal origin can be used as a nutrient-rich waste-based phosphorus (P) fertiliser product due to the inherent high P content in faecal matter. The high-value biochar can be used as a plant nutrient source to supply P, increase soil organic matter, and lime acidic soils due to its high pH (Sun et al., 2018).

Although there is potential for the BSFL technology to be applied in faecal sludge management, there is little information on BSFL residue's pyrolysis derived from faecal matter collected from UDDTs to produce biochar (Mutsakatira et al., 2018). Research is needed to optimise

pyrolysis conditions for pyrolysis temperature and residence time effect on the physical and chemical properties of biochar derived from faecal-based black soldier fly residue and assess the safety of the product by undertaking a microbial analysis.

Therefore, the objectives of this study were to (i) determine the optimal temperature and residence time for high-quality biochar production using residue from BSFL as feedstock and (ii) characterise the physical/chemical and microbial properties of the biochar produced.

2. Materials and methods

Black soldier fly larvae (BSFL) residue used during the study was collected and stored at 4 °C before the pyrolysis was done.

2.1. Biochar production

Pyrolysis was carried out in metal trays using a temperature-controlled muffle furnace at the Soil Science Laboratory (University of Kwa-Zulu Natal, Pietermaritzburg Campus, South Africa). Three temperatures (300, 400, and 500 °C) were used and timed to determine optimum pyrolysis conditions for complete charring. Table 1 shows the pyrolysis temperatures and the corresponding residence times. Only the biochar that pyrolysed without burning at a particular temperature and residence time was considered for characterisation.

Biochar samples were crushed and passed through a 2 mm sieve and stored in airtight Mason jars. The biochar samples were analysed for moisture content, total solids, volatile matter, organic matter, and fixed carbon following proximate methods described by Aller et al. (2017). Biochar yield was calculated as a percentage of the mass of char generated after pyrolysis (Equation 1).

$$\text{Biochar yield } (\%) = \frac{\text{Initial mass (g)} - \text{Final mass (g)}}{\text{Final Mass (g)}} * 100 \quad \text{Equation 1.1}$$

2.2. Proximate analysis

Approximately 10 g of the biochar sample was added to glass beakers in triplicate. The moisture content and total solids were determined by oven heating at 105 °C for 24 h (Zhang et al., 2017). The ash content was determined by adding 5 g samples of biochar from the moisture-determined samples used for ash content in a muffle furnace at 550 °C for 6 h (Mierzwa-Hersztek et al., 2019; Singh et al., 2017). Volatile matter was determined on the moisture determined samples by combusting 1 g of biochar at 950 °C for 7 min in crucibles with the lid on, according to the American Society of Testing Materials (ASTM D 271-48, 1954). Moisture, ash and fixed C were calculated according to ASTM D1762–84 (2007).

2.3. pH, cation exchange capacity, mineral composition, and trace elements

A mixture of 1 g biochar and 20 ml water was prepared and used for pH determination. Samples were stirred for 30 min using a magnetic stirrer, and pH was read using a calibrated pH meter (HANNA Instruments, Romania). The same sample solution used for pH was used for measuring electrical conductivity (EC) using a conductivity meter (HANNA Instruments, Romania). The pH in KCl was determined using 20

ml of 1M KCl. The ammonium acetate method, buffered at pH 7, was used to assess cation exchange capacity (Munera-Echeverri et al., 2018; Song and Guo, 2012). Briefly, the biochar samples were passed through a 2 mm sieve before analysis. A mass of 0.5 g biochar sample was added into a centrifuge tube. A volume of 40 ml 1M ammonium acetate solution was added to the centrifuge tube and shaken thoroughly overnight. The sample settled undisturbed for 30 min, and the leachate was filtered with Whatman no. 1 filter paper using a Buchner vacuum pump connected to a 100 ml volumetric flask. Three consecutive volumes of 30 ml of fresh ammonium acetate were added to the funnel and filtered until the leachate volume reached 90 ml. The leachate was collected, and the leachate's exchangeable cations were determined by an atomic absorption spectrophotometer (AAS) (Sequential Atomic Absorption Spectrometer, Varian, AA280FS, California, USA). Calcium and magnesium were determined using an atomic absorption spectrophotometer. Sodium and potassium were determined using a flame photometer. The remaining biochar sample was washed with four separate additions of 30 ml of 99% isopropanol to remove excess ammonium. The leachate from this process was discarded. To extract the adsorbed ammonium in the biochar sample was washed with three portions of 30 ml of 1 M KCl, and the leachate was collected. The leachate was diluted using 1 M KCl to reach a volume of 100 ml. The ammonium concentration in sodium chloride extract was analysed using the Thermo-Scientific Discreet Gallery (Scientific Therm Fisher, Waltham, Massachusetts, USA, 2014).

The gallery's principle of action is that ammonia reacts with hypochlorite ions generated by alkaline hydrolysis of sodium dichlorocyanurate to form monochloramine. Monochloramine reacts with salicylate ions and sodium nitroprusside at pH 12.6 to form a blue compound. The absorbance of the blue compound is measured spectrophotometrically at a wavelength of 660 nm. The absorbance is related to the ammonia concentration on the calibrated curve. Ammonium ions were analysed using the Thermo-Scientific Discreet Gallery (Scientific Therm Fisher, Waltham, Massachusetts, USA, 2014). The leachate's exchangeable cations were determined by an atomic absorption spectrophotometer (AAS) (Sequential Atomic Absorption Spectrometer, Varian, AA280FS, California, USA). Cation exchange capacity was calculated following Eq. (2) below (Munera-Echeverri et al., 2018).

$$\text{CEC (cmol}_c \text{/ kg)} = \frac{\text{NH}_4 - \text{N (mg/L) (0.25L)} * 100}{\text{Mass of biochar (g) (1meqNH}_4 - \text{N per 14mgNH}_4 - \text{N)}}$$
$$\text{Equation 1.2}$$

Mineral elements from biochar were analysed from a 0.5 g sample of biochar. Ashing was done for 6 h at 550 °C in a muffle furnace. The ash was solubilised on porcelain crucibles using 10 ml aqua regia (HNO_3 and HCl mixed in a ratio of 1:3) and digested using microwave-assisted digestion (von Gunten et al., 2017). Calcium, Mg, K, Zn, Cu, Fe, and Mn concentrations were determined using Flame Atomic Absorption Spectroscopy (FAAS) (Varian AA280FS). Potassium and Na were analysed using Flame Atomic Emission Spectroscopy (FAES) with a fast sequential absorption spectrometer (Varian AA280FS). A mass of 0.2 g of the air-dried biochar sample in crucibles was heated at 1 450 °C for 6 min under furnace conditions to analyse carbon, nitrogen, and sulfur (CNS) using the Leco-TruMac CNS Autoanalyser (Leco-cooperation, LECO CNS-2000, St Joseph MI, USA, 2012).

Mineral nitrogen (NH_4^+-N and NO_3–N) was extracted with 2M KCl solution using 1:10 biochar: extracting solution volume ratio. From each sample, 2 g of sample was added to 50 ml plastic tubes with 20 ml of the extracting solution (Maynard et al., 1993). An overhead shaker was used to shake the mixture for 30 min. The mixture was filtered using Whatman Filter paper, and the filtrate was transferred to storage bottles. The NH_4^+-N and NO_3–N were analysed using the Thermo-Scientific Discreet Gallery (Scientific Therm Fisher, Waltham, Massachusetts, USA, 2014).

Phosphorus was extracted following the AMBIC-2 method (Van der Merwe et al., 1984). The AMBIC extractant was made using 0.25 M ammonium bicarbonate (NH_4HCO_3) + 0.01 M (NH_4)$_2$ EDTA +0.01 M

Table 1. Pyrolysis temperatures and residence time for pyrolysis of residue from the decomposition of faecal matter from UDDTs by BSFL.

Pyrolysis temperature (°C)	Residence times (minutes)
300	120; 90; 60
400	60; 45
500	60; 45; 30

ammonium fluoride (NH_4F) + 0.05 g L^{-1} super floc. The pH was adjusted to 8.0 using ammonia solution. A volume of 25 ml AMBIC solution was added to a pre-weighed mass of 2.5 g biochar. The mixture was shaken on an overhead shaker for 30 min at a speed of 180 rpm. The solution was filtered using Whatman no.1 filter paper. The extracted solution was mixed with a colour reagent and changed to a blue colour after 45–50 min (Van der Merwe et al., 1984). The absorbance of the solution was read using an ultra-violet (UV) spectrophotometer (Varian Alpha, UV-VIS, Spectronic, Unicam, Berlin, Germany) at a wavelength of 670 nm. A standard curve was drawn using different concentrations to determine the P concentration of the samples.

2.4. Biochar physical properties analyses

The surface morphology of biochars was examined using a scanning electron microscopy (SEM) system (EVO LS15, Carl Zeiss Microscopy, New York, USA). Biochar samples were sputtered with gold coating before viewing using a gold sputtering machine (Quorum Q150R ES, Quorum Technologies, East Sussex, UK). Pore diameter measurements were taken using Image J software (Soft Imaging System, Munster, Germany). The surface area was analysed in liquid nitrogen at 25 °C using the Brunauer–Emmett–Teller (BET) surface area analyser (NOVA 2200e analyser Quantachrome Instruments, Boynton Beach, FL, USA) (Brunauer et al., 1938). Physisorption isotherms were plotted based on nitrogen adsorption at increasing pressure using the Barret, Joyner and Balenda (BJH) method. This method is used to determine mesopore size distribution (Barrett et al., 1951; Sing, 2001; Sinha et al., 2019).

2.5. Microbiological analyses

Both BSFL residue and biochar samples were analysed for total co-liforms, faecal coliforms, *Escherichia coli*, *Ascaris*, and helminths at Talbot, Pietermaritzburg ($29°37'55.6''S$ $30°22'41.4''E$).

The pour plate membrane filtration method was used to analyse total coliforms and faecal coliforms, while the membrane filtration coupled with 4-methylumbelliferyl-beta-D-glucuronide (MUG) fluorescence was used for *Escherichia coli* (E. coli). A water sample was filtered through a membrane filter upon which the bacteria were entrapped. The filter was placed on a selective growth medium and incubated at 35 °C for 24 h, after which all characteristic colonies were counted. A sample dilution of 1:10 was made using sterile deionised water and filtered after dilution.

The E. coli was determined after a positive count was made on colonies that developed a blue fluorescence around the colony's margin when grown in an NA-MUG (Nutrient Agar + MUG) medium at 35 °C ±0.5 °C for 2–4 h. Total coliforms were determined from counts with red bacterial colonies with a (golden-green) metallic sheen within 18–24 h when incubated at 35 °C on m-Endo type medium containing lactose. To determine faecal coliforms, counts were made on bacteria that produced typical blue colonies on m-FC medium without Rosolic acid within 18–24 h when incubated at 45 °C.

Ascaris and *helminths* were analysed using "tween 80" as a surfactant. A 10 g dried sample was weighed and soaked in "tween 80 solution". This solution was filtered using 150 μm sieve. The filtrate that passed through the sieve was collected and transferred to a centrifuge tube and centrifuged. The pellets were collected and resuspended using zinc sulphate, allowing *helminth* eggs to float in a solution. The supernatant was filtered using a 20 μm sieve. The residue that remained on the sieve was then collected and transferred to a slide for observation under a microscope. Observed *helminth* eggs were counted and calculated per g of dry biochar or residue.

2.6. Data analysis

All data sets were subjected to a general Analysis of Variance (ANOVA) using GenStat 18th Edition Statistical Package VSN

International, Hemel Hempstead, UK. Means were separated using Fisher's Least Significant Difference at a 5% level of significance.

3. Results and discussion

Appendix 1 shows images of biochar pyrolyzed at different pyrolysis temperatures and residence times. The optimum pyrolysis conditions were found to be 500 °C and 30 min. The biochar was not burnt, and the chemical and physical properties were comparatively better than the other two pyrolysis temperatures. These results are comparable with those from studies at Makerere University, where faeces were pyrolysed using a laboratory muffle furnace (Carbolite MTF 12/38/250/E301), at 350, 450, and 600 °C for 10, 20, and 40 min, and showed that pyrolysis was achieved without burning in all cases (Gold et al., 2018). Figure 1 shows biochar yields from residue subjected to different pyrolysis temperatures and residence times. Residue subjected to the highest pyrolysis temperature (500 °C) and the shortest residence time (30 min) produced the lowest yield. Biochar yields decreased significantly with increasing pyrolysis temperature ($p < 0.001$) from 51.5 to 56.9% at 300 and 500 °C, respectively (Figure 1). The observed decrease in yield could probably be attributed to thermal dehydration of hydroxyl groups as moisture is lost through evaporation and the volatilisation of organic fractions (Koetlisi and Muchaonyerwa, 2017; Liu et al., 2014). During pyrolysis, organic C is lost due to the thermal degradation of lignocellulose material in the human excreta (Liu et al., 2014). A rapid mass loss was observed at 300 °C. At this temperature, torrefaction (the slow pyrolysis process where organic material starts to pyrolyse at temperatures between 200 and 320 °C) results in a more significant mass loss. These results are similar to those from Sun et al. (2018), who observed an initial rapid mass loss at 300 °C during the pyrolysis of sewage sludge; and reported low biochar yields with increasing residence time at a low temperature of 300 °C, using different agricultural wastes (from low ash to high ash).

Table 2 shows the proximate analysis results of biochar pyrolyzed at different pyrolysis temperatures and different residence times. The residue had the lowest ash content of 12.40%, which increased with increasing pyrolysis temperature. Ash content increased to 15.42% at 300 °C, 18.57% at 400 °C, and 24.18% at 500 °C (Table 2). The higher amounts of inorganic compounds such as K, P, Mg, and Ca, compared to the residue which remained in the biomass after degradation of organic material and volatilisation of C, H, O and volatile solids could also explain the high ash content (Domingues et al., 2017). Volatile matter (VM) of the residue was 76%. The VM decreased to 63, 55, and 33% at 300, 400, and 500 °C, respectively, which corroborated the observation that increasing temperatures resulted in a proportional rise in VM driven off from the biochar (Titiladunayo et al., 2012). Liu et al. (2014) established a positive relationship between VM and organic matter, showing that high VM indicates high organic matter. Organic matter decomposition by loss on ignition was highest in biochar pyrolysed at 500 °C because, at higher temperatures, more organic origin material is burnt.
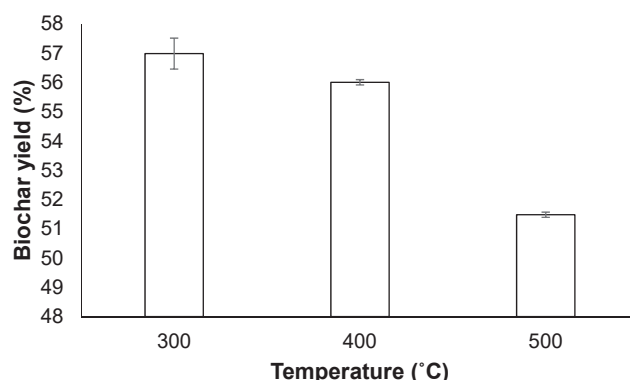

Figure 1. Biochar yield at different pyrolysis temperatures and residence times.

Table 2. Proximate values (percentage of dry matter) of biochar produced under different pyrolysis conditions.

Treatment	Ash (%)	Volatile (%)	Fixed C (%)	Moisture (%)	TS (%)	OM (%)
Residue	12.40[a]	75.83[a]	11.78[a]	51.07[b]	48.93[a]	87.60[a]
B300	15.42[b]	62.80[b]	21.78[b]	0.54[a]	99.46[b]	84.58[b]
B400	18.57[c]	55.01[c]	26.42[c]	0.24[a]	99.76[b]	81.43[c]
B500	24.18[d]	32.77[d]	43.06[d]	0.18[a]	99.82[b]	75.82[d]
s.e.d	0.1002	0.415	0.459	0.4080	0.4080	0.1002
l.s.d	0.2452	1.016	1.122	0.9990	0.9990	0.2452
p-value	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001
c.v (%)	1.1	0.9	2.2	1.9	0.3	0.2

Superscripts represent mean differences (P < 0.05) within each column according to Fisher's test.
B300, B400 and B500 indicate biochar pyrolysed at 300 °C, 400 °C, and 500 °C for 60, 45, and 30 min respectively.
TS is the total solids; OM is the organic matter; Fixed C is the fixed carbon.
s.e.d. is the standard error of deviation.
l.s.d. is the least significant difference at the 5% level.
c.v. is the coefficient of variation.

The residue had the lowest fixed C of 12%, which increased with increasing pyrolysis temperature. The highest fixed C was 43% at 500 °C. Fixed C refers to the non-volatile solid remains, other than ash after the volatile matter has been driven off a biochar particle in the absence of air (Leng et al., 2019). As pyrolysis temperature increased, more volatiles were driven off the carbon matrix resulting in biochar with low VM, increasing the fixed C.

Pyrolysis resulted in a significant decrease in moisture from the residue (51.7%) to 0.54% (300 °C), 0.24% (400 °C) and 0.18% (500 °C) (Table 2). However, further increasing the pyrolysis temperature did not result in a significant decrease in moisture. Total solid (TS) behaved inversely to moisture, with the residue having the lowest TS (48.3%) and increasing with pyrolysis temperature up to 99.8% at 500 °C.

Table 3 shows the chemical characteristics of biochar pyrolysed at different pyrolysis temperatures and residence time. The C: N ratio increased with increasing pyrolysis temperature with the highest C: N ratio of 9.28% at 500 °C and the lowest ratio of 8.33% at 300 °C due to the total N decreasing at a faster rate than C with the increase in temperature. A similar trend of decreasing C with increasing pyrolysis temperature was observed when poultry litter was pyrolysed at 300, 400, and 500 °C (Song and Guo, 2012). Ammonium decrease with increasing pyrolysis temperature resulting in the reduction of total N is attributed to the loss of NH_4^+-N through volatilisation when volatile matter containing N groups are also lost at 200 °C (Bagreev et al., 2001).

Further increase in temperature could have resulted in a different nitrogen group (pyridine), resulting in a further decrease in total N (Khanmohammadi et al., 2015). Gezahegn et al. (2019) explained that higher N in biochar pyrolysed at 300 °C compared to a temperature above 300 °C is due to the presence of N compounds that cannot be decomposed at 300 °C.

The residue's pH value was 6.70 and increased with increasing temperatures (8.8, 9.3, and 9.82 at 300, 400, and 500 °C, respectively) (Table 3). This suggests that increasing pyrolysis temperature produced more alkaline biochar. The increase in pH could be explained by the increase in basic cations such as Ca, K, Mg and Na, which had the following concentrations: 0.99, 0.19, 0.59, and 0.30 cmol$_c$/kg, respectively at 300 °C. The concentration increased to 1.12, 0.20, 0.65 and 0.33 0.30 cmol$_c$/kg for the same elements at 500 °C (Table 3). Evidently, the alkali metals became separated from the organic matrix at increasing temperature during pyrolysis, resulting in biochar enriched with alkali metal salts (Cao and Harris, 2010; Gasco et al., 2005; Gaskin et al., 2008; Singh et al., 2010; Yuan et al., 2011). Thus, suggesting that the relative increase in pH with increasing pyrolysis temperature could be attributed to an increase in ash content. During pyrolysis, carboxyl groups in the biochar are reduced and acidic groups are deprotonated to conjugate bases resulting in alkalinity. Furthermore, pyrolysis has been associated with an increase in basic functional groups and decreased carboxylic functional groups, increasing pH, as observed by Ronsse et al. (2013).

Table 3. Chemical (C: N, pH, EC, CEC, exchangeable cations, and metal concentrations) properties of biochar produced under different pyrolysis conditions.

	C: N	pH	EC (mScm^1)	CEC (cmolc kg^{-1})	Exchangeable bases (cmolc kg^{-1})			Trace metals (mg kg^{-1})							
					Ca	K	Mg	Na	Cr	Cd	Cu	Fe	Mn	Ni	Zn
Residue	8.07[ab]	6.70[a]	2.11[a]	15.11[a]	0.39[a]	0.07[a]	0.20[a]	0.17[a]	17.8[a]	1.0[a]	89.7[a]	3 407[a]	336.6[a]	22.1[a]	347.1[a]
B300	7.94[a]	8.80[b]	2.22[b]	29.07[b]	0.99[b]	0.19[b]	0.59[b]	0.30[b]	153.7[b]	3.0[c]	135.9[b]	6 224[b]	458.5[b]	34.7[a]	649.8[b]
B400	8.33[b]	9.32[c]	2.47[c]	70.01[c]	1.07[c]	0.18[b]	0.63[c]	0.32[c]	176.8[c]	2.7[b]	148.0[c]	8 929[c]	448.3[b]	54.8[b]	683.5[b]
B500	9.28[c]	9.82[d]	2.51[c]	70.06[c]	1.12[c]	0.20[b]	0.65[c]	0.33[c]	210.6[c]	4.0[d]	221.6[d]	14 737[d]	504.0[c]	64.7[b]	805.2[c]
Sewage sludge[+]	-	-	-	-	-	-	-	-	350	3	120	-	<260	150	200
s.e.d	0.094	0.018	0.013	0.352	0.022	0.004	0.008	0.005	8.370	0.104	1.790	30.0	11.54	4.15	11.51
l.s.d	0.230	0.044	0.033	0.861	0.054	0.012	0.019	0.011	20.48	0.256	4.380	73.3	28.24	10.15	28.16
p value	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001
c.v (%)	1.4	0.2	0.7	0.9	3.0	3.7	1.8	2.0	7.3	4.8	1.5	0.4	3.2	11.5	2.3

*Superscripts represents mean differences (P < 0.05) within each row according to Fisher's test (Williams and Aldi, 2010; Aller et al., 2017).
CEC is the cation exchange capacity.
s.e.d. is the standard error of deviation.
l.s.d. is the least significant difference at a 5% level of significance.
c.v. is the coefficient of variation.
B300, B400 and B500 indicate biochar pyrolysed at 300 °C, 400 °C, and 500 °C for 60, 45, and 30 min respectively.
Sewage sludge + represents the maximum allowed limit for land application for crop production.

The high pH characteristic of biochar is beneficial when applied to acidic soils (Berek et al., 2011).

The residue had the highest EC, which decreased at 300 °C and started increasing at 400 and 500 °C (Table 3). Similar trends have been observed where biochars produced from sewage sludge at low temperatures up to 400 °C have electrical conductivities lower than the residue (Figueiredo et al., 2017; Singh et al., 2010). However, as pyrolysis temperature increases, EC increased. The EC is an indicator of total salts, and it can be used to estimate the quantity of total dissolved salts in a sample. Electrical conductivity is important when considering biochar use for crop production because materials with high salinity can have toxic effects on plants (Song and Guo, 2012).

Table 3 shows that cation exchange capacity increased with increasing pyrolysis temperature. At 300, the CEC was 29.07 cmol$_c$/kg and increased to 70.01–70.06 cmol$_c$/kg, at 400 and 500 °C, respectively. Cation exchange is the ability of a material to adsorb cations. Cation exchange capacity has been found to increase in biochar because of an increase in surface area. A similar study showed no significant difference in biochar pyrolysed at 550 °C and 650 °C, indicating that increasing pyrolysis temperature did not affect CEC (Koetlisi and Muchaonyerwa, 2017). However, on the contrary, Méndez et al. (2013) reported that the increase in CEC might be caused by the formation of phenolic functional groups when sewage sludge was used as feedstock.

Table 3 shows that exchangeable cations (K, Ca, Mg and Na) and trace elements such as Cd, Cu, Cr, Mn, Ni, and Zn increased after pyrolysis (Gondek and Mierzwa-Hersztek, 2017; Sadaka et al., 2014). Gezahegn et al. (2019) reported an increase in the concentration of main elements Ca, Mg, and K with increasing pyrolysis temperature using wood feedstocks. Phosphorus, calcium, and magnesium are essential macronutrients; hence their availability in biochar is beneficial to crop production.

Although trace elements' concentrations increased, they remained within the acceptable thresholds for agricultural (Verster, 2016) (Table 2). Human beings do not normally consume and excrete heavy metals. Trace amounts are only found when the heavy metals originate from dietary sources (Tervahauta et al., 2014). Also, there could be possible contamination from external sources such as foreign objects thrown into pits such as batteries.

Figure 2 shows the ammonium and orthophosphate content of biochar pyrolyzed at different pyrolysis temperatures and residence time. Ammonium was highest at the lowest temperature of 300 °C (2.07 mg kg^{-1}) and decreased with increasing pyrolysis temperature to 1.22 and 0.90 mg kg^{-1} at 400 and 500 °C, respectively. The decrease in inorganic N in the form of ammonium can be attributed to the loss of NH groups. However, these results are contrary to the report from Hossain et al. (2011), who observed that increasing pyrolysis temperature resulted in an increase in nitrates to 0.24 and 0.32 mg kg^{-1} at 500 and 700 °C, respectively. When sewage sludge was used, there was an increase of 43% in ammonium at 700 °C (Hossain et al., 2011). In the same study, ammonium decreased from 1175 mg kg^{-1} to 25 mg/kg at 500 °C. In this study, orthophosphates increased significantly with increasing pyrolysis temperature, 806 mg kg^{-1} at 300 °C, and 3148 mg kg^{-1} at 500 °C (Figure 3). Increasing pyrolysis temperature increased the P content of biochar at 500 °C by 25.6%. Phosphorus is related to the residue's inorganic fraction (Capodaglio et al., 2017; Khanmohammadi et al., 2015). The high P content in the biochar suggests that faecal matter pyrolysis can be a potential alternative P source. Similar results were reported from an experiment in which sewage sludge was pyrolysed at 450 °C, and resulted in a 2-3-fold increase in P (Frišták et al., 2018).

Figure 3 shows isotherms for biochar produced at 300 °C (A), 400 °C (B), and 500 °C (C) with their respective Scanning Electron Microscopy (SEM) images. All biochars had similar type IV isotherms (Figure 4). As the pressure increased in all isotherms, the amount of nitrogen gas adsorbed onto the biochar's surface increased, forming a monolayer indicated by the knee on the graph. Type IV isotherms are indicative of a mesoporous pore structure. At 300 °C, the pores were smaller and increased in size with increasing pyrolysis temperature. As temperature increases, the pores coalesce to form bigger pores. The corresponding SEM images of the biochars are shown on the right (Figure 3). Increasing the pyrolysis temperature changed the surface morphology of biochar. Biochar pyrolysed at 300 °C had a smooth surface structure. At low pyrolysis temperatures, smaller pores were observed. Pores were clogged with volatiles, which could not escape at 300 °C. Increasing pyrolysis temperature increased the amount of volatiles driven off from the feedstock resulting in more pores and more voids within the biochar matrix (Ma et al., 2016; Zhang et al., 2015). Pore quantity and size increased with increasing pyrolysis temperatures, as evident in the electron microscope images. As pyrolysis temperature increases, the surface of biochar became more rugged.

Figure 4 shows the Brunauer–Emmett–Teller (BET) surface area and pore volume of biochar produced at different pyrolysis temperatures and different residence times.

Pore diameter, pore area and surface area increased significantly (p < 0.001) with increasing pyrolysis temperature. The surface area was lowest at 300 °C (2.84 m^{2}g^{-1}), followed by 6.14 m^{2}g^{-1} at 400 °C and 6.61 m^{2}g^{-1} at 500 °C. The BET surface area increased with increasing pyrolysis temperature (Figure 4). These results are lower than the findings of Koetlisi et al. (2018), who observed an increase in surface area from 350
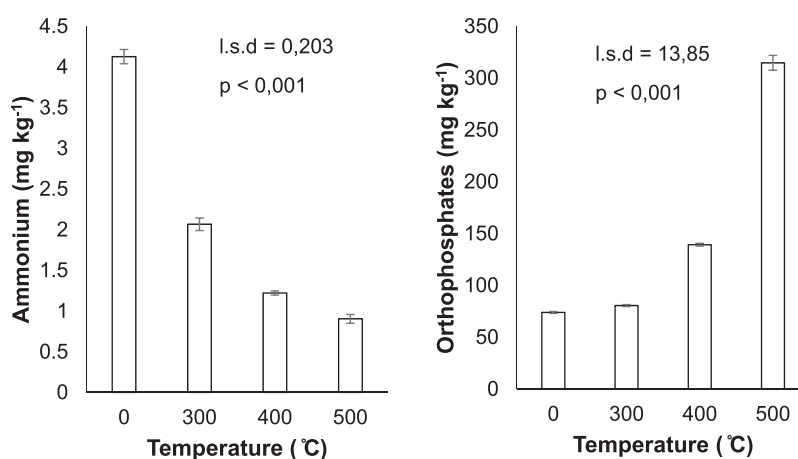
Figure 2. Ammonium and orthophosphate content of residue (0) and biochar pyrolysed at 300, 400, and 500°C.
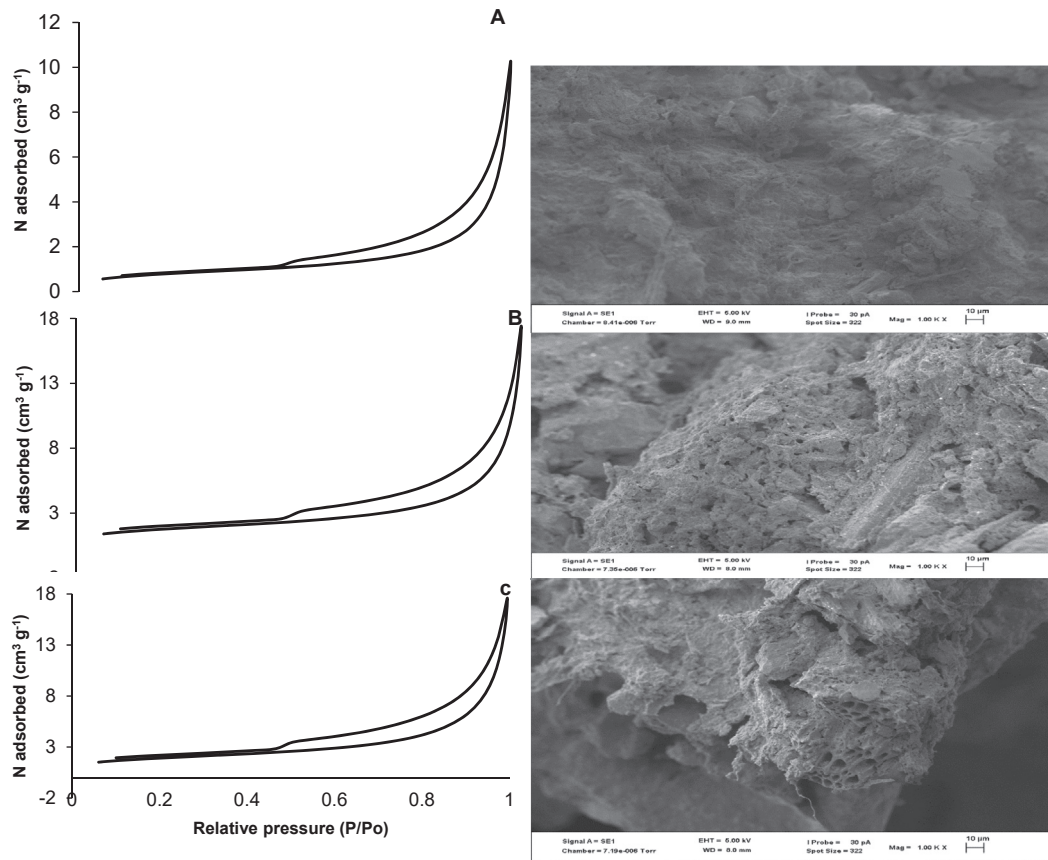
Figure 3. Scanning electron microscopy images presenting the porous structure of biochar pyrolysed at 300 °C (A), 400 °C (B), and 500 °C (C) (yellow vertical line represents the section measured for diameter determination).
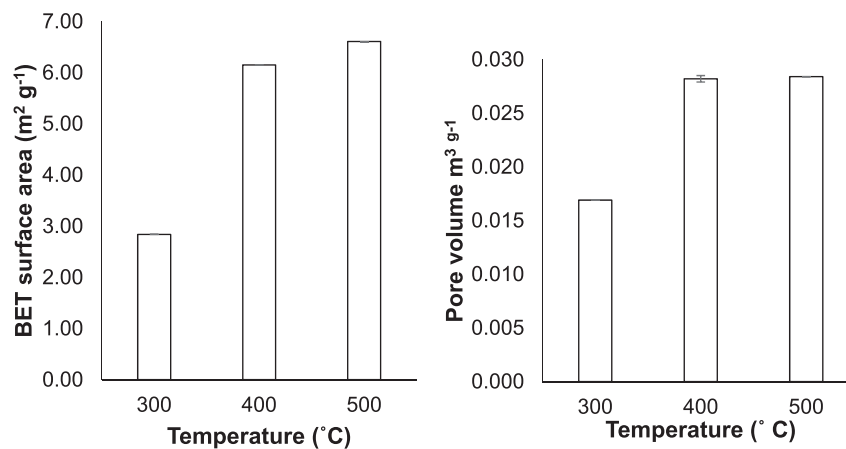

Figure 4. Relationship between BET surface area and pore volumes of biochar subjected to three various temperatures.

to 550 °C using latrine waste and sewage sludge waste. Latrine waste surface area ranged from 7.5 to 25.7 m² g⁻¹.

Table 4 shows the distribution of micropores, mesopores, and macropores in biochar pyrolyzed at 300, 400, and 500 °C. Increasing pyrolysis temperature from 300 to 500 °C increased the area contributed by micropores, mesopores, and macropores.

The residue was characterised by *Ascaris*, viable helminths, *Escherichia coli*, total coliforms, and faecal coliforms (Table 5). Pyrolysis killed all the pathogens, making safe biochar amendments that do not pose a health risk to users.

4. Conclusions

Pyrolysis temperature and residence time significantly affect the physical and chemical characteristics of biochar produced from the residue after decomposition of feacal matter from UDDTs by BSFL. Physical, chemical, and biological characterisation of biochar is important in determining the pyrolysis temperature and residence time suitable for producing biochar used in agriculture. This study confirmed that biochar yield decreased with increasing pyrolysis temperature. Biochar pyrolysed at 500 °C for 30 min showed excellent characteristics regarding C: N ratio,

Table 4. Pore area distribution, micropores, mesopores, and macropores for biochar pyrolysed at different pyrolysis temperatures.

Temperature (C)	Micropores	Mesopores	Macropores
300	0.12[a]	0.40[a]	1.86[b]
400	0.14[a]	4.41[c]	0.38[a]
500	0.22[b]	1.09[b]	3.21[c]
s.e.d	0.01	0.02	0.06
l.s.d	0.02	0.05	0.02
p value	<0.001	<0.001	<0.001
c.v (%)	5.4	1.2	0.4

Superscripts represent mean differences ($P < 0.05$) within each column according to Fisher's test.

B300, B400 and B500 indicate biochar pyrolysed at 300 °C, 400 °C, and 500 °C for 60, 45, and 30 min respectively.

TS is the total solids; OM is the organic matter; Fixed C is the fixed carbon.

s.e.d. is the standard error of deviation.

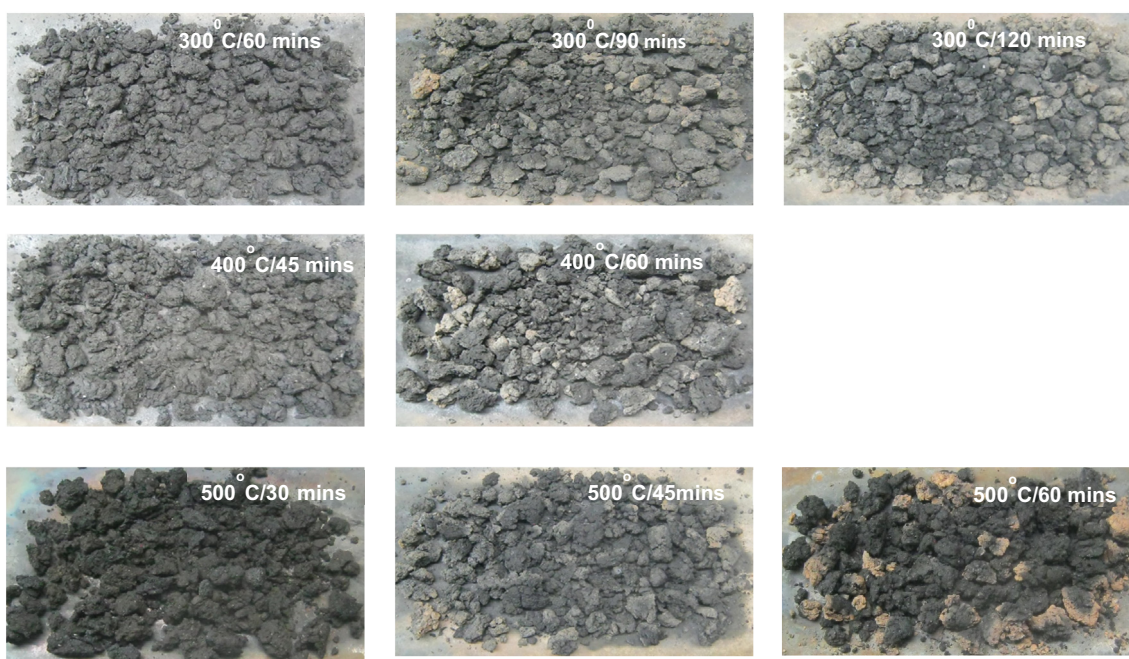l.s.d. is the least significant difference at the 5% level.

c.v. is the coefficient of variation.

Table 5. Biological characteristics of residue used as feedstock for pyrolysis compared to guidelines by (Herselman and Snyman, 2009).

	Ascaris (g^{-1} dry mass)	Viable helminth (g^{-1} dry mass)	*E. coli* (cfu ml^{-1})	Faecal coliforms (cfu ml^{-1})	Total coliforms (cfu ml^{-1})
Residue	1	2	4×10^{-1}	7×10^{-1}	1.1×10^{-1}
Guidelines	-	<0.25	-	<10^3	-

porosity, surface area, major elements, CEC, pH, and fixed C. The highest pyrolysis temperature of 500 °C resulted in the lowest biochar yield when pyrolyzed for 30 min. Phosphorus (orthophosphates) was high at 500 °C, indicating that biochar is a good phosphorus source. The isotherms for biochar produced at 500 °C showed a mesoporous material meaning it has good water and nutrient holding capacity. The high temperatures killed pathogens during pyrolysis making it safe for agricultural use.

Declarations

Author contribution statement

Nqobile Nkomo: Conceived and designed the experiments; Performed the experiments; Analyzed and interpreted the data; Wrote the paper.

Alfred Oduor Odindo and William Musazura: Analyzed and interpreted the data; Wrote the paper.

Roland Missengue: Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data; Wrote the paper.

Data availability statement

Data included in article/supplementary material/referenced in article.

Declaration of interests statement

The authors declare no conflict of interest.

Additional information

No additional information is available for this paper.

Appendix 1. Biochar at different pyrolysis temperatures and different residence times

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