Research Paper

Operating parameters for three resource recovery options from slow-pyrolysis of faecal sludge

M. Gold, M. Cunningham, M. Bleuler, R. Arnheiter, A. Schönborn, C. Niwagaba and L. Strande

ABSTRACT

Slow-pyrolysis is a treatment technology that is being explored for treatment of faecal sludge (FS) from onsite sanitation technologies. Next to pathogen inactivation, the technology produces treatment products. Revenues from these products could offset treatment costs and contribute to financially viable sanitation. In comparison to lignocellulosic biomass and other biowastes, little information is available on operating parameters for FS pyrolysis to produce char for different resource recovery options. In Kampala, Uganda, this bench-scale study investigated the influence of two major operating parameters, hold time (10, 20 and 40 minutes) and pyrolysis temperature (350, 450 and 600 °C) for pyrolysis of FS into char for solid fuel production, soil enhancement and carbon sequestration. Hold time: 10 min was the most suitable hold time for all resource recovery options as char characteristics had only minor variations between hold times. Temperature: Char characteristics identified 350 °C as the most suitable for fuel production and 450 or 600 °C for carbon sequestration. FS char had characteristics for soil enhancement comparable to biowaste and lignocellulosic biomass chars, with heavy metal concentration exceeding guideline concentrations. The most suitable temperature needs to be selected based on plant and soil type, and legal regulations.

Key words | char, pyrolysis, sanitation, treatment, Uganda, wastewater

NOMENCLATURE

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INTRODUCTION

Approximately 2.8 billion (10⁸) people rely on onsite technologies for access to sanitation (World Health Organization/United Nations Children’s Fund (WHO/UNICEF) 2017). In low-income countries, faecal sludge (FS) collected in onsite sanitation technologies is inadequately managed, resulting in negative implications for public and environmental health, and economic development (Hutton & Haller 2004; Mara et al. 2010). A current challenge in FS management is the lack of long-term operating treatment plants. If treatment plants do exist, they often fail due to a lack of financial resources for operation and maintenance (Bassan et al. 2014). Designing treatment...
processes to allow resource recovery from treatment products could create revenues and contribute to the sustainability of treatment plants (Diener et al. 2014). Resource recovery options include soil conditioners and fertilisers, fuels, feedstock for anaerobic digestion or animal protein production, and building material components (Diener et al. 2014).

The market value of FS treatment products is dynamic and varies between regions and countries. For example, market research indicates that fuels generate the largest revenue in Sub-Saharan Africa (Diener et al. 2014; Gold et al. 2014). Treatment technologies that can produce several products when changing operating parameters might best harness their local market value. Pyrolysis, the thermo-chemical conversion of biomass in the absence of oxygen into solids (e.g., char), liquids (e.g., tars, water), and gases (e.g., carbon dioxide) is an example of such a technology (Basu 2013). During slow-pyrolysis (hereafter referred to as pyrolysis), the biomass is exposed to a slow heating rate considering the biomass residence time (Basu 2013). Pyrolysis temperatures are in the range of 300–700 °C, which is enough to inactivate pathogens (United States Environmental Protection Agency (USEPA) 1994; Manyà 2012; Basu 2013). According to Jeffery et al. (2013), additional benefits include the possible use of the char produced during pyrolysis as a solid fuel or soil conditioner, or for pollutant immobilisation and carbon sequestration.

The most important operating parameters in pyrolysis that influence char characteristics are pyrolysis temperature (i.e., the maximum temperature during pyrolysis), hold time, and heating rate (Manyà 2012; Basu 2013; Crombie et al. 2014; Lehmann & Joseph 2015). However, these data are mostly lacking for FS. In China, Liu et al. (2014) investigated the influence of five pyrolysis temperatures on FS char characteristics for use as a soil conditioner, and concluded that its benefits included a high nutrient content and its structure. However, the study did not evaluate potential contaminants, such as heavy metals, polycyclic aromatic hydrocarbons (PAHs), polychlorinated biphenyls (PCBs), and dioxins. In South Africa, Koetlisi & Muchaoneyrwa (2017) concluded that pyrolysed pit latrine FS has a potential for immobilisation of cadmium. In Uganda, Kiwana & Nalwagga (2016) observed that char briquettes produced from FS and charcoal dust produced similar emissions and performed similarly in household stoves, but did not consider different operating parameters. The influence of operating parameters on char characteristics varies for different feedstocks. However, information on the influence of these three operating parameters for other feedstocks could potentially be transferred from experience with lignocellulosic biomass (i.e., wood), animal manures, wastewater sludge, and faeces (Rajkovich et al. 2012; Crombie et al. 2014).

The objective of this study was to identify the pyrolysis temperatures and hold times required to produce char from FS for three different resource recovery options: solid fuel production, soil enhancement, and carbon sequestration, to optimise operation, meet local market demands, and generate revenue to support long-term operation of FS treatment plants.

MATERIAL AND METHODS

This research was conducted over nine months (January to September 2016) in Uganda and Switzerland.

Pyrolysis feedstock preparation

The FS for the pyrolysis experiments was collected from vacuum trucks at the National Water & Sewerage Corporation (NWSC) Lubigi Wastewater and Faecal Sludge Treatment Plant in Kampala, Uganda. During vacuum truck discharge, four 1.5-L grab samples were collected: one at the beginning, two during the middle, and one at the end. In Kampala, septic tanks and pit latrines are the commonest sanitation technologies. Therefore, to take a representative sample of FS in Kampala, samples were collected from nine vacuum trucks that collected FS from pit latrines, and ten vacuum trucks that collected FS from septic tanks.

All grab samples were combined for dewatering. The composite sample had a water content of 98.5%, and was dewatered with a 0.3-mm mesh polyester fabric to reduce the water content to 91.2%, and then dried in a laboratory oven at 105 °C. To minimise changes in the pyrolysis feedstock characteristics, the 100% dried FS was stored in a fridge and protected from light with aluminium foil for...
the two months of pyrolysis experiments. FS was dried again at 105 °C immediately before the pyrolysis experiments were conducted to maintain a 100% dry pyrolysis feedstock.

**Slow-pyrolysis experiments**

FS pyrolysis was conducted at Makerere University in a laboratory tunnel furnace (Carbolite MTF 12/38/250/E301). Dried FS was pyrolysed following a 3 x 3 full-factorial design at three pyrolysis temperatures (350, 450, and 600 °C) and hold times (10, 20, and 40 minutes), at a constant heating rate of 25 ± 3 °C min⁻¹. According to Basu (2015), pyrolysis temperature is the maximum temperature during pyrolysis. Operating conditions were selected based on previous research (Liu et al. 2014; Ward et al. 2014). During the experiments, the tube was flushed with nitrogen gas at 50 L h⁻¹ to maintain an oxygen-free environment (Liu et al. 2014). To minimise the feedstock and furnace variability, 8 ± 0.5 g of the dried FS sample was carbonised for each combination of pyrolysis temperature and hold time between four and nine times. Following pyrolysis at the studied pyrolysis temperature and hold time combination, the glass tube was manually removed from the furnace and cooled to room temperature (approximately 25 °C) for 15 (pyrolysis temperature: 350 °C) or 20 minutes (pyrolysis temperature: 450 and 600 °C). Following each experiment, the char was weighed and the char yield was calculated according to the equation reported by Lohri et al. (2015).

**Analyses parameters**

FS feedstock and char were characterised at the Swiss Federal Institute of Aquatic Science and Technology (Eawag), Zurich University of Applied Sciences (ZHAW), and the Paul Scherrer Institute (PSI). The feedstock and char were analysed for parameters that are relevant metrics for solid fuel production, soil enhancement, and carbon sequestration (Crombie et al. 2014; Jeffery et al. 2015; Gold et al. 2017a). These include char yield, pH, proximate analysis, calorific value, carbon, carbon stability, phosphorus, plant-available phosphorus (Plant-P), cation exchange capacity (CEC), heavy metals, dioxins, PAHs, and PCBs.

**Analytical methods**

The analytical methods in this study were selected based on those reported by the International Biochar Initiative (IBI) (2014), European Biochar Certificate (EBC) (2012), and Bachmann et al. (2016). Analytical methods were established by comparing results for FS with char with results for standard biochars (UK Biochar Research Centre, Edinburgh, UK). In addition, for quality control, all samples produced at hold times of 20 min, and all elemental analyses were conducted in triplicate. The standard deviation of these replicate measurements can be considered as an estimate for the variability in analysis results of the different parameters.

All char samples were completely dried following Standard Methods (APHA/AWWA/WEF 2005) prior to analysis. The pH was analysed in solution using a pH meter (Hach-Lange PHC301) according to ISO 10390 (2005) and the Federal Compost Quality Assurance Organisation (2003). To determine the pH, 2.5 g of the sample was mixed with 25 mL 0.01 M CaCl₂ for one hour. Volatile solids were analysed gravimetrically following ASTM D762 (2015) as mass loss through combustion at 950 °C for 7 min with a lid (Nabertherm L3). Ash was determined gravimetrically from the residue of combustion without a lid (Nabertherm L3) (EBC 2012). The fixed carbon content was calculated according to Equation (1) according to ASTM D5172: (ASTM D762 2015; ASTM D5172 2013)

\[
\text{Fixed carbon (%) = 100% - Volatile solids (%) - Ash content (%)}
\] (1)

Carbon was analysed using a CHN analyser (Leco Tru Spec Micro) following DIN 51732 (2014). Carbon stability was estimated as the carbon remaining from 0.1 g of carbon following digestion with 0.01 M H₂O₂ and 7 mL of deionised water at 80 °C for 144 hours, according to Cross & Sohi (2013). The nutrients analysed in this research included nitrogen, phosphorus, and Plant-P. Nitrogen was analysed using a CHNSO analyser (Leco Tru Spec Micro). Total phosphorus was analysed through inductively coupled plasma optical emission spectrometry (ICP-OES) following the microwave digestion (ultraCLAVE 4) of a 0.2-g sample with 5 mL of HNO₃, 1 mL of H₂O₂, and 0.5 mL of HF at
250 °C and 120 bar for 10 min. Plant-P was estimated as the amount of phosphorus extractable by 2% formic acid extraction according to Wang (2013) and IBI (2014). The extracted phosphorus in the filtrate of a 0.35-g sample and 35 mL of CH₂O₂ was analysed following 30 minutes of mixing using the vanadomolybdate colorimetric method (Gericke & Kurmies 1952). CEC was determined following the cobalt-hexammine method according to Ciesielski et al. (1997). The absorbance was determined using a Hach Lange DR3800 photometer at wavelengths of 380 nm and 475 nm following filtration of a 1.25-g sample and 50 mL of a 0.0166-mol L⁻¹ H₁₈N₆Cl₃Co solution that was mixed for one hour. Heavy metals (chromium, copper, nickel, lead, and zinc) were analysed in the same manner as total phosphorus. PAHs, PCBs, and dioxins were determined using 5 g of a composite sample of all chars of one pyrolysis temperature according to EBC (2012) at Eurofins Umwelt and LUFA Nord-West Germany.

RESULTS AND DISCUSSION

Results of replicate analyses of dried FS and char characteristics of this study are available on the Open Science Framework (Gold 2018).

Feedstock characteristics

Table 1 presents the characteristics of the dried FS analysed in this study with a range of mean values reported in the literature. FS is highly variable (Niwagaba et al. 2014), however, the results in this study were within the range reported in the literature. Overall, the FS in this study had poor characteristics for resource recovery from pyrolysis in comparison to the FS (Liu et al. 2014) and faeces (Ward et al. 2014) used in previous pyrolysis studies. For example, Liu et al. (2014) used septic tank FS for pyrolysis with a higher calorific value (18.1 MJ kg⁻¹) and phosphorus content (3.1%), and a lower fraction of ash (17.0%). This is most likely due to differences in the onsite sanitation design and operation. In this research, the FS used for pyrolysis was obtained from septic tanks and pit latrines.

Influence of hold time on char characteristics and resource recovery

Table 1 also summarises the characteristics of the FS char produced in this study at different hold times and pyrolysis temperatures. Hold time is an important operating parameter with a large influence on the treatment capacity of a pyrolysis reactor. Shorter hold times allow for a higher treatment capacity, and therefore may be more economical. In this study, when comparing a hold time of 10 min to 20 min and 40 min at the same pyrolysis temperature, the differences between all parameters were smaller than 20%, excluding volatile solids, nitrogen, CEC, chromium and nickel (see Supplementary material, available with the online version of this paper). For these parameters, the differences between hold times were 21.5%, 20.4%, 32.6%, 21.8% and 20.8%, respectively. Based on the maximum treatment capacity and hold times used in this research, 10 min is an optimal hold time for all resource recovery options, as char characteristics only vary slightly between hold times of 10, 20, and 40 min. The 10 min observed in this study is much shorter than the 40 and 120 min reported for pyrolysis of FS by Liu et al. (2014) and faeces by Ward et al. (2014), however, they did not evaluate the influence of hold time. All reported research has been conducted at the bench-scale, therefore, future studies should investigate whether results are transferable to operation at pilot- and full-scale.

Influence of pyrolysis temperature on char characteristics and resource recovery

Pyrolysis temperature is also an important operating parameter with a strong influence on char production, characteristics, and the energy balance of pyrolysis reactors. In this study, differences in char characteristics were observed between different pyrolysis temperatures, indicating that pyrolysis temperatures could be selected to optimise the characteristics for the intended resource recovery options. As the differences in characteristics with different hold times were small, in the following presentation of results, mean values for the three hold times are used.
Operating parameters for slow-pyrolysis of faecal sludge

### Table 1

Characteristics of dried FS used for pyrolysis in this study in comparison to the range of mean literature values and characteristics of FS char produced at different hold times (10, 20, 40 min) and pyrolysis temperatures (350, 450, 600°C). All results are for 100% dry feedstock and char. Standard deviations are included in parentheses for analyses conducted in triplicate.

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<th>Parameter</th>
<th>Unit</th>
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<th>Char</th>
<th>350°C 10 min</th>
<th>350°C 20 min</th>
<th>350°C 40 min</th>
<th>450°C 10 min</th>
<th>450°C 20 min</th>
<th>450°C 40 min</th>
<th>600°C 10 min</th>
<th>600°C 20 min</th>
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<td>%</td>
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<td>69.3</td>
<td>62.6</td>
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<td>58.3</td>
<td>57.8</td>
<td>57.7</td>
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<tr>
<td>Ash</td>
<td>%</td>
<td></td>
<td></td>
<td>40.1(±1.8)</td>
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<td>35.0</td>
<td>34.7</td>
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<td>Fixed carbon</td>
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<tr>
<td>CV(^c)</td>
<td>MJ kg(^{-1})</td>
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<td></td>
<td>13.4(±1.8)</td>
<td>13.1</td>
<td>13.0</td>
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<td>CV(^b)</td>
<td>MJ kg(^{-1}) ash-free</td>
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<td>22.3(±2.9)</td>
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<td>Carbon</td>
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<td>Plant P(^e)</td>
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<tr>
<td>Chromium</td>
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<td>Zinc</td>
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<td>669(±25)</td>
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\(^a\)Bassan et al. (2013); Liu et al. (2014); Muspratt et al. (2014); Appiah Effah et al. (2015); Seck et al. (2015); Byrne et al. (2016); Gold et al. (2017a, 2017b).

\(^b\)Calorific value.

\(^c\)Plant available phosphorus.

\(^d\)Cation exchange capacity.

\(^e\)Carbon stability values >100% are likely due to inaccuracies in the determination of carbon before and after H\(_2\)O\(_2\) digestion.
Solid fuel

Char yield, calorific value, and ash content are important metrics for the use of pyrolysis to produce solid fuels. Calorific value is an estimate of the char's energy content, and ash is an indicator of the quantity of char that will not combust and accumulate in a stove or kiln, interfering with efficient combustion. High char yields and caloric values, and low ash content are desirable characteristics for solid fuels.

As summarised in Table 1, the char yields and calorific value decreased while the ash content increased with increasing pyrolysis temperature. This suggests that the lower pyrolysis temperature of 350 °C is the most suitable for solid fuel production. These results agree with those of previous researchers who have used faeces (pyrolysis temperature range: 350–700 °C), FS (300–700 °C), wastewater sludge (300–700 °C), and cow manure (300–600 °C). However, the gradients for these parameters differed between these different feedstocks (Hossain et al. 2011; Rajkovich et al. 2012; Liu et al. 2014; Ward et al. 2014). In contrast, lignocellulosic biomass has a small increase in ash and large increase in calorific value with increasing pyrolysis temperature (Rajkovich et al. 2012). The variation in the results between these feedstocks could be due to ash and a difference in the thermal stability of the organic compounds in the feedstocks. As shown in Table 1, pyrolysis increased the calorific value of FS on a dry ash-free basis. However, as pyrolysis volatilises organic compounds, ash increases, char yield decreases and calorific value decreases (Basu 2013). Organics in FS could have a lower thermal stability than lignocellulosic biomass (Ward et al. 2014). As the calorific value and quantity of char decreases by pyrolysis, the co-combustion of dried FS may be more economical, especially in the absence of appropriate technologies for efficient heat recovery in countries in development. Pyrolysis reactors with heat recovery can combust the energy-rich organic compounds that volatilise to provide heat for feedstock drying or the start-up of the reactor.

Overall, the char produced from FS in this study had poor fuel properties. Char produced from wood and bio-waste had calorific values of 20–30 MJ kg⁻¹ and <5–20% ash, while those for the char produced from FS were <11.3 MJ kg⁻¹ and >56% ash in this study, respectively (Lohri et al. 2016). This is likely due to digestion during storage in onsite sanitation technologies, and also the high ash content of the FS in Kampala. The results reported by Gold et al. (2017b) suggest that the high ash content is also due to the presence of sand, which can constitute 20–32% (dry basis) of the content of FS from septic tanks and lined pit latrines due to poor onsite sanitation technology design and operation. In comparison, the char produced from undigested faeces (without sand) at 300 °C had a calorific value of 25 MJ kg⁻¹ (Ward et al. 2014). Mixing FS with inorganic material, such as sand, which contributes to high ash contents needs to be minimised to optimise the fuel production from FS. In addition, onsite storage times should be short to reduce the digestion that occurs during storage (Seck et al. 2015; Gold et al. 2017a).

Soil enhancement

Phosphorus, Plant-P, pH, CEC, heavy metals, dioxins, PAHs, and PCBs are important metrics for the use of pyrolysis to produce char for soil enhancement. Phosphorus and Plant-P are estimates of the total and plant-available phosphorus (Wang 2013). Char pH can positively or negatively influence the soil pH, and the bioavailability of minerals and nutrients (Ketterings et al. 2005). CEC is an estimate for the potential of char to retain positively charged ions, for example, in fertilisers. Therefore, char with a high CEC can increase the efficiency of fertiliser application (Sohi et al. 2010; Crombie et al. 2014). In contrast to the benefits, heavy metals, dioxins, PAHs, and PCBs in char also need to be monitored to prevent environmental contamination. Desired values for all parameters depend on factors such as soil and plant type, and local regulations. Therefore, no single char characteristic and pyrolysis temperature can be recommended for soil enhancement, as local requirements need to be considered (Jeffery et al. 2015).

As summarised in Table 1, in this study, phosphorus and Plant-P increased with pyrolysis temperature. As illustrated in Figure 1, considering the standard deviation of the results at the same temperature, the proportion of phosphorus that could be plant available (e.g., plant-P/P) did not vary between temperatures. Comparing these results to those in literature in Figure 1 indicates that the influence of pyrolysis temperature on phosphorus and Plant-P for FS is different to that for lignocellulosic biomass. For example, phosphorus...
also increased with pyrolysis temperature in char produced from cow manure (Rajkovich et al. 2012) and wastewater sludge (Hossain et al. 2011), but decreased for char produced from wood (Rajkovich et al. 2012). An increase is to be expected as phosphorus is generally not volatile. Plant-P decreased with pyrolysis temperature in char produced from wood and wheat straw (Crombie et al. 2014), and wastewater sludge (Hossain et al. 2011) above 450–500 °C (not included in Figure 1). These results suggest that, in contrast to lignocellulosic biomass, the phosphorus in FS char can be increased with pyrolysis temperature without decreasing its availability to plants. However, the use of different analytical methods to determine Plant-P in these studies may limit comparison.

As summarised in Table 1, as has been the case for animal manures and wastewater sludge (Hossain et al. 2011; Cantrell et al. 2012; Méndez et al. 2013; Cely et al. 2015; Khanmohammadi et al. 2015; Zornoza et al. 2016), in this study, heavy metals did not volatilise, therefore, their concentration in the char increased with pyrolysis temperature. Heavy metals in FS can represent a potential limitation for the use of FS char to enhance soils. Guidelines for acceptable concentrations of heavy metals are quite variable (EBC 2011; IBI 2014; Shackley et al. 2014; Meyer et al. 2017), however, the char produced in this study did not meet the guideline concentrations. Chromium was the greatest concern, as its concentrations were above all recommended guideline concentrations at all pyrolysis temperatures. Whether the other evaluated heavy metals met or exceeded...
guidelines varied based on the pyrolysis temperature. PAH, PCB, and dioxin concentrations were all below the recommended concentrations (EBC 2012), as they were below 1.9 mg kg$^{-1}$, 0.06 mg kg$^{-1}$, and 2 ng kg$^{-1}$, respectively.

If heavy metal concentrations can be limited, then the results of this study support the findings of Liu et al. (2014), and highlight the potential of FS char for soil enhancement over fuel production. The results for phosphorus, Plant-P, and CEC were comparable to values for chars produced from wastewater sludge, cow manures, and lignocellulosic biomass (see Figures 1 and 2), and the results for pH indicate the potential of applying FS char when an increase in soil pH is desired (Jeffery et al. 2015). Given the variability in plant yields observed with the addition of char from other feedstocks, plant trials are suggested to evaluate the effect of FS char on a specific soil and plant at different application rates (Sohi et al. 2010; Lehmann & Joseph 2015).

**Carbon sequestration**

High total carbon and carbon stability are important parameters for the use of pyrolysis to produce char for carbon sequestration, which can mitigate climate change by incorporating stable carbon into the soil (Bruun et al. 2016). Total carbon is an estimate of the amount of carbon available for sequestration, and carbon stability is an estimate of its susceptibility to degradation in soils (Crombie et al. 2013).

As shown in Table 1, in this study, total carbon decreased and carbon stability increased with increasing pyrolysis temperature. This suggests that a high pyrolysis temperature, 450 or 600 $^\circ$C in this study, is most suitable for carbon sequestration. However, as more carbon would be released to the atmosphere at high pyrolysis temperatures, a suitable temperature for carbon sequestration needs to be determined through creating a carbon balance considering the reactor-specific recovery of carbon from liquid and gaseous pyrolysis products (Crombie & Mašek 2015).

Overall, at 450–600 $^\circ$C, the carbon stability of FS char was similar to that of lignocellulosic biomass char, but its carbon content was three times lower (Rajkovitch et al. 2012; Crombie et al. 2014). This means that three times more FS-char needs to be applied for the same amount of carbon sequestration in soil. This could still be an interesting resource recovery option when combined with the benefits of soil enhancement.

**CONCLUSIONS**

This bench-scale study conducted in Kampala, Uganda, identified conditions for two important operating
parameters, hold time and pyrolysis temperature, for the slow-pyrolysis of FS into char for solid fuel production, soil enhancement, and carbon sequestration. These products can generate higher revenues than conventional products, such as compost, and thereby contribute to financially sustainable FS treatment. However, limited research and operational experience are available for pyrolysis, air emissions are produced, and safe operation requires reliable supply chains and skilled labour. Key findings include the following:

- Short hold times (such as 10 min) can be sufficient for the complete pyrolysis of FS.
- Recommended pyrolysis temperatures differ based on resource recovery options.
- The char produced in this study had poor properties as a solid fuel, but most parameters were comparable to chars of biowaste and lignocellulosic biomass for use to enhance soil and carbon sequestration.
- Based on the temperatures used in this study, the most suitable temperature for solid fuel production is 350 °C, and that for carbon sequestration is 450 or 600 °C.
- No specific temperature could be recommended for soil enhancement.

Future research could focus on testing FS chars with site-specific soils and plants to identify the optimal pyrolysis temperature for soil enhancement. Furthermore, pilot-scale studies are required to assess the transferability of this bench-scale study to wet/liquid FS with variable characteristics.

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