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Development and prototype testing of a novel small-scale pyrolysis system for the treatment of sanitary sludge

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ABSTRACT

An innovative twin auger pyrolysis reactor for the intermediate conversion of partially wet sanitary faecal sludge generated on train toilets (mobile applications) is designed and successfully commissioned. The effects of operating temperature, feedstock feeding rates and moisture content and their interaction on the distribution and properties of product biofuels with the use of no carrier gas were assessed. Approximate yields of 50 %, 40 % and 10 % were observed for the biooil, syngas and biochar respectively at 500 °C. Meanwhile, the highest calorific value (HHV) of the syngas did not exceed 10 MJ/Nm^3. Acidic compounds (10 % RPA) and nitrogen-containing hydrocarbons (18 % RPA) substituted the abundant species in biooil fraction at 500 °C with the lowest feed volumes of feedstock. Biochar presented the highest fixed carbon (58 %) and ash content (37 %) upon higher operating temperatures for the minimum and maximum volumes of feedstock. Interestingly, the retention of inorganics within the structure of char were observed. Meanwhile, the calorific value of the biochar reduced due to intensive reduction reactions at higher temperatures ranging to a minimum of 19 MJ/kg. Effects of reactor design and configuration on the pyrolytic products are discussed accordingly and further implications are provided. Overall, extensive further research for the end-use of pyrolytic products and the investigation of emissions, upon energy recovery or land-application of by-products are suggested.

1. Introduction

Implementation of thermal treatment technologies for the decentralised sanitation of human wastes has been recently identified as a cornerstone for proper faecal sludge (FS) management [1]. Researchers have extensively investigated the applicability of combustion and gasification processes to provide rapid, energy-efficient and environmental friendly solution to the improper disposal of this hazardous waste [2 3]. However, major challenges for the automation and optimisation of these processes due to high heat fluxes and the fuels physiochemical characteristics within the reactor were observed [1 4]. Researchers have claimed the disruptive role of fuel's moisture content and particle size on consistent conversion of various feedstock, delaying ignition and subsequent conversion reactions [4 3 2]. Meanwhile, the application of small-scale pyrolysis for sanitation of FS is barely explored with a few investigating the principles in lab scale [5 6 7]. These technologies are commonly known to be suitable for low-income countries or situations where sewage infrastructure is inaccessible or difficult to access. This work follows the findings from our earlier study [1], on the selection of pyrolysis as the suitable technology for these applications. A review of the available research on sludge management methods has revealed a major gap in the utilisation of thermal treatment methods for on-site sanitation. In addition, small-scale implementation of such technologies still lack research on operational challenges, process efficiencies and emissions.

Pyrolysis is the thermal degradation of the organic matter taking place in a non-reactive atmosphere at temperatures ranging from 300 to 700 °C depending on the type of biomass and process conditions [8]. Through the pyrolysis stage of a fuel, various fractions such as non-condensable (syngas), condensable (biooil) and a solid fuel (biochar) are formed [9]. The composition and yield of the products are heavily dependent on the process conditions whilst very high heat fluxes (~600 °C) and short vapour/solid residence times (fast pyrolysis) favour the production of liquid bio-oil products. Moreover, lower heating temperatures and longer residence times (slow pyrolysis) contributes to the production of solid char [10].

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However, the main application of this process in commercial scale is known to be the maximization of the liquid yield [11]. In addition to high heating temperature, heating rate and residence times, the pyrolysis efficiency and product specifications are affected by the raw fuel's characteristics such as moisture content and particle size [1]. Hence, various stages of pre-treatment might be implemented to further reduce the size and moisture content (<10 % wt.) of the waste [11].

Conventionally, different reactor designs and process principles are used for the pyrolysis of various types of biomass and waste materials such as Rotary kilns, rotating cone and ablative processes to facilitate the application constraints and render a desired product for energy applications [10]. High heat and mass transfers are achievable from reactors such as bubbling and circulating fluidised beds (BFB and CFB respectively) associated with high levels of operational complexity whilst requiring large quantities of carrier gas. More information on the existing pyrolysis technologies and reactor designs can be found elsewhere [12]. In addition to the aforementioned reactor designs, auger reactors are considered to deliver a high technology strength and market attractiveness [13]. This technology provides mechanical fluidisation of the waste through the length of the reactor in the presence of heat carrier materials (i.e. hot sand) for mainly fast pyrolysis such as the twin auger reactor designed in the IOWA state university [14] or convective heating of the tube for slow and intermediate modes of pyrolysis [15]. Utilisation of auger reactors have been extensively researched for various types of biomass and waste solid materials [10 8 16]. Morgano et al. (2015) carried out the pyrolysis of beech wood chips achieving 13.2 wt % bio-oil at 450 °C and observed a reduction of 13 wt% in the yield of char through temperature change from 350 °C to 500 °C [17]. Yu et al. (2016), observed a declination in the yield of char through temperature escalation while the change in temperature from 350 °C to 600 °C contributed to the higher yield of gas by 13 % and 11 % for rice husk and corn stalk respectively [18]. Of the advantages of screw pyrolysis reactors are their suitability for mobile applications with limited infrastructure and lower energy demand due to the smaller specific size of the reactor [8]. Furthermore, the design is capable of handling various raw materials with variable particle sizes. Typically, the gas residence times are higher in augers in comparison to BFB and CFB which should be of no disadvantage when the goal is a higher yield of non-condensable fraction [18]. In addition, the prolonged gas residence times in auger reactors favour the production of more char at the expense of liquid oil which could be mitigated by introducing exit ports along the reactor if the condensable fraction is desired [19].

This study aims for the development and commissioning of a micro-scale twin auger pyrolysis reactor for the stabilisation of human faecal sludge generated in on-board train toilets considering the application constraints. In addition, it investigates the chemical properties of various faecal sludge feedstock generated in rolling stock for the first time to the best of our knowledge. Following these, valuable insights into the yield and properties of the achievable pyrolytic products from such technology are provided. Utilisation of twin screw pyrolysis reactors enable an enhanced mixing and dispersion of feedstock via the intermeshing augers for an effective heat transfer through particles [10]. Additionally, these reactor configurations results in cleaning of sticky materials plugging on the screw flights which potentially form in the case of single screw reactors [20].

2. Methods

2.1. Fuel preparation and thermochemical characterisation

The wastewater samples were collected from various passenger railway depots. Proximate properties of the faecal waste such as moisture, volatile and ash contents were determined after sieving (dewatering) the faecal sludge from wastewater feedstock with a 1 mm mesh in line with BS EN ISO 18134–2, BS EN 15148, and BS EN 14,775 respectively, while the fixed carbon content was obtained through subtraction from 100 %. The calorific value (HHV) and the concentration of sulphur and chlorine were measured utilising a Parr 6400 calorimeter and a Dionex ICS-1600 ion chromatographer, respectively. The ultimate properties of feedstock were analysed via an elemental analyser from ELEMENTAR while the approximate content of oxygen was calculated by deduction of the wt.% of carbon, hydrogen and nitrogen from 100 %, showcased in Table 1.

Thermal gravimetric analyses on the waste samples were performed with a TGA8000 from Perkin Elmer to understand their maximum degradation points. These sets of tests allowed the further selection of operating temperatures for the pyrolysis unit. The thermal analyses were conducted under 60 mL of nitrogen purge at varying heating rates between 25 and 100 °C/min.

2.2. Design and fast prototyping

A schematic of the faecal sludge treatment system is provided in Fig. 1, through which the sanitary solid wastes undergo a dewatering stage where the faecal sludge from each toilet flush is separated from the effluent and fed into a drying unit. Following this, the feedstock moisture content is partially removed in the dryer and the nutrient-rich condensate generated through the evaporation of moisture is extracted away. The dried faecal matter is accumulated in a storage chamber which feeds the partially dried FS into the pyrolysis reactor via mechanical means. The rationale behind the implementation of the chamber is to allow the batch processing of the waste per day to reduce energy consumption in comparison to a constant operation of the reactor. Finally, upon applying thermal loads, FS undergoes pyrolysis degradation with an aim to maximise the reduction of char, the essential rule for hazardous waste treatment [1], and boost the production of syngas with the highest yield and calorific value. Production of high-quality syngas enables the chance of convenient energy recovery through its combustion and limits the energy consumption of this system via recycling of the heat which are suggested for future studies.

Described in Table 2, to obtain an insight on the possible volumes of waste stream entering the system, the reported values of the median daily adult human faeces were referred. Commonly reported, the average values range from 35 g to 796 g [21]. Hence, an assumption was made for the average value as 160 g/cap/day for both adult men and women to pursue with the calculation of mass flow rates for the primary design of the prototype. In addition, an arbitrary amount of tissue (16 g) per defecation was considered in the calculations. Depending on the application, a maximum of 16/defecation/day was also considered in the calculations. Finally, a safety factor of 2 was considered for all the parameters affecting the mass balance of faecal sludge in the system. It is

Table 1
Chemical characteristics of faecal sludge and toilet paper.

	Faecal Sludge	1	2	3	4	Toilet paper
Proximate % (Dry basis)	M_C[1] (arb%)	91	90	93	92	6.5
	Ash	5	4	7	6	2
	V_M[2]	83	82	83	81	89.5
	FC[3]	12	13	10	13	8.5
Ultimate % (Dry basis)	C	44	42	46	43	42
	H	7	7	8	7	5
	N	1	1	2	1	0
	O	43	46	37	43	51
ppm	S	960	–	0	0	–
	Cl	3450	–	3511	5226	–
Calorific value	Dry basis (MJ/kg)	18	19	17	17	17

[1] M_C arb: Moisture content – as received basis.
[2] V_M: Volatile matter.
[3] F_C: Fixed carbon.

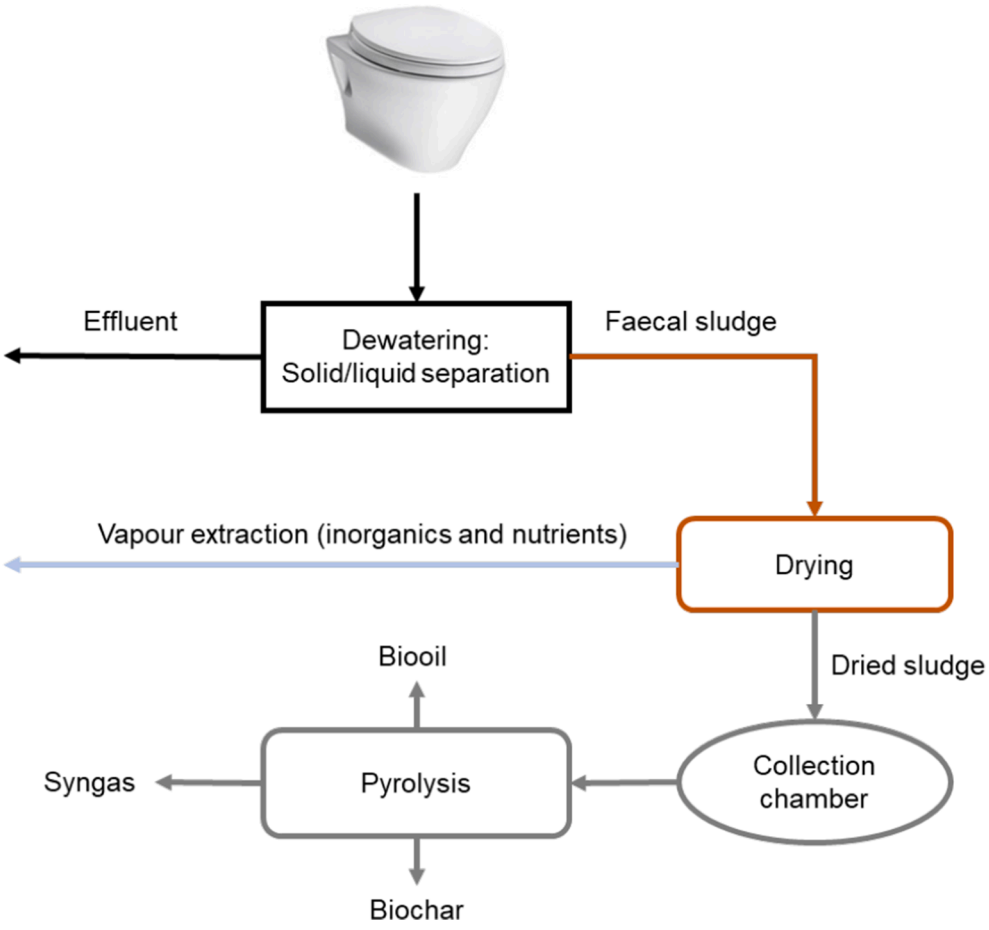

Fig. 1. Faecal sludge stabilisation system schematic.

Table 2
Calculation of average mass flowrate of faecal sludge on daily basis.

Ref.	Literature	Assumption	Safety factor (*2)
[23]	Median faecal wet mass 128 g/cap/day	Average weight/stool for both men and women were assumed as 160 g.	Total solid weight of 352 g/defecation (Including tissue paper)
[24]	Wet wt.% 110–170 g/cap/day		
[25]	250 and 111.3 g/ stool for men and women respectively		

notable that the assumption of safety factor for the number of defecations per day increases the total mass of faecal sludge entering the system. However, this is different to that of the mass of sludge per defecation that affects the drying efficiency of the dryer unit. Multiplication of all the parameters by the safety factor leads to an overall total wet solid generation of ~ 11 kg/day. The described calculation method considers the worst-case scenario for the waste streams that enters the solid waste thermal treatment system.

The fast-prototyping process of the pyrolysis reactor was primarily based on the fundamental mass transfer in screw reactors, followed by the thermodynamic calculations to predict the fuel and product residence times for the pyrolysis reactor. Literature on the design and fabrication of screw conveyors for thermal processing were referred to [14 22].

Typically, the efficiency of the thermal drying process is reported between 20 and 70 % [26] with the screw dryer being approximately 50 % [22]. Hence, a diagram for the overall functions was generated to clarify the potential mass flow of the dry matter and moisture in each phase, Fig. 2.The mass balance for the pyrolysis reactor upon conduction of heat to the sludge feedstock could be noted as:

$$\frac{dm}{dt} = \dot{M}_{FS} - \dot{M}_{GP} - \dot{M}_{CH} = 0$$

Where M_{FS}, M_{GP} and M_{CH} are the mass flow rates of the FS, gaseous products, and the solid char in kg/h respectively. This steady state analysis assumes all biochar exits within the length and the gaseous products include the condensable, non-condensable and aerosol fractions. The fast-prototyping process was continued by considering the pyrolysis reactor as a screw conveyor whilst various constraints due to the application were accounted for.

Twin auger reactors are known best to mitigate the chance of clogging whilst improving the conversion efficiency of the biomass with their higher mixing rates [27]. In this design, the effective means of thermal load for heat supply were conductive and so the counter rotation (up-pumping) of the intermeshing twin augers were provisioned to maximise the heat transfer through particles upon contact with the whole heated circumference of the reactor. However, down pumping with counter rotation of screws is observed to better enhance the mixing behaviour of the materials inside the reactor and so a more efficient heat transfer when heat carriers are fed with the feedstock for fast pyrolysis [14 28].

Among various types of screw flights, some are well known for handling sticky and viscous materials (i.e., ribbon and interrupted flights) [10 29], but here a ‘standard’ flight commonly in various applications was selected to mitigate the risk of further challenges in fabrication stage. The pitch to diameter ratios of the screw conveyors

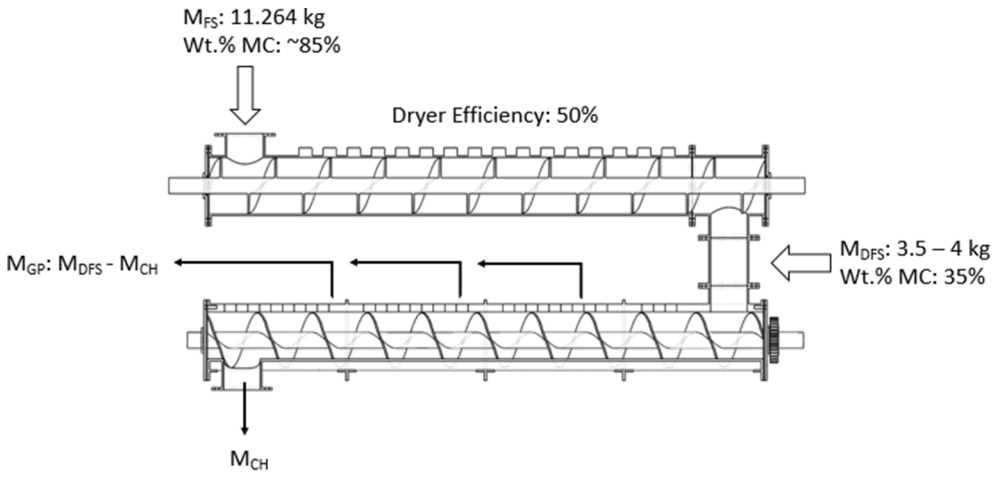

Fig. 2. Mass flow schematic.

were specified as 1 since it has been commonly found optimum in industrial practices [30].

Following multiple configurations and the selection of components, manufacturer benchmark on the standard small auger sizes were consulted [31] from which the relative volumetric capacities to the rotational speed and auger's outer diameter sizes could be predicted. Simply, a single screw diameter (d_o) of 5.08 (cm) allows the conveyance of ~ 9 kg/hr of FS with a density of ρ: 1600 kg/m^3 [21], at a screw speed of 1 RPM. Considering the fact that a twin auger configuration increases the throughput capacity by a factor of 1.5, a screw diameter of 2.54 cm could have been suitable. However, consultation with an auger manufacturer directed the design towards an auger diameter of 7.2 cm for the twin auger pyrolysis reactor. This could potentially reduce the processing timeframe of stored FS when the throughput is higher. In addition, this auger size provides a chance for heat recycling through the system from the combustion of energy-rich product gases via the hollow shaft to limit the energy intensity of the system in future investigations. Such configuration has been practiced stabilising the heat gradient in the reactor via introduction of a heat transfer fluid [27 32]. Additionally, the larger auger size would allow the sticky FS to flow freely within the unit, mitigating the chance of cloggage and is simpler to fabricate. It is notable that the trade-off between the size and screw rotation speed could make the volumetric capacity of the reactor adjustable.

Stainless steel AISI 316 was used for the manufacturing of all components in the system. The nominal feedstock and product residence times as well as velocities were calculated as a function of the occupied cross-sectional area within the reactor to form an iterative pattern and predict the length of the reactor. Considering the application constraints, fast pyrolysis was considered impractical for the calculations since vapour residence time of < 2 s is needed for this pyrolysis mode. Henceforth, aiming for a slow/intermediate pyrolysis of human FS, the screw speed was considered as 1 RPM and the cross section of the reactor was defined as a rectangle to simplify the calculations.

The twin auger forms a (ω) cross section with the flights intermeshing with each other, Fig. 3. The gas exit ports drilled as a part of modular design which remained unused were sealed with nuts throughout the operation of the unit.

In order to predict the heat transfer mechanisms within the reactor, some assumptions were made on the required heat for the pyrolysis (enthalpy) which includes the sensible heat and the heat of pyrolysis. The heat for pyrolysis of various biomass and waste materials has been investigated in the past, ranging from 0.7 to 8 MJ/kg depending on the fuel type and the moisture contents [33]. In the case of sludge materials, an enthalpy of 0.7 MJ/kg was reported [34]. In this study, an initial 3 MJ/kg was selected as the enthalpy value since it was assumed that a fuel with ~ 15 % moisture content could be suitable for the pyrolysis

Fig. 3. Cross-section of the twin auger reactor.

[3], whilst the specific heat capacity of the sludge was noted as 4 kJ/kg. K [35]. A review of the literature revealed the use of various types of heaters and different heating powers depending on the required mode of pyrolysis for the application [36 17].

2.3. Setup and commissioning

Flexible heating tapes of 940 Wh were wrapped around the reactor to gradually increase the sludge temperature throughout the tube length. To measure and control the temperature within the reactor, K type thermocouples were fixed on gas/vapour exit ports and connected to separate PID controllers to maintain a constant temperature at different zones of the unit, illustrated in Fig. 4. The whole reactor tube and the piping were insulated with 25 mm (thickness) ceramic fibre blankets to minimise heat dissipation or condensation of products within the reactor.

Through a novel design for the lab testing of the unit, a vertical hopper (cylinder) having two sliders on the top and bottom was bolted to the feedstock inlet. The sliders reduced the chance of air ingress within the reactor during operation while the feedstock entered the reactor via gravitational forces.

Prior to the commissioning, in order to obtain an insight on the achievable solid residence times respective to the screw speed (RPM), multiple cold flow tests with partially dried FS were conducted, Fig. 5. Typically, solid residence times for slow/intermediate pyrolysis are known to improvise around 8 min [1]. Thus, to narrow down the tests

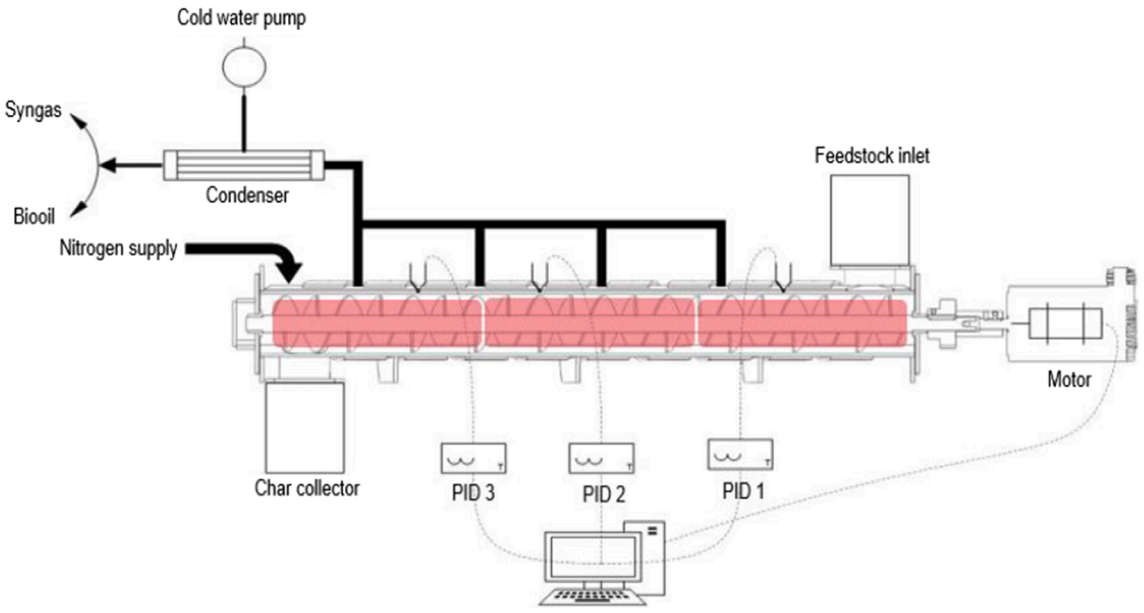

Fig. 4. Side view of the twin auger pyrolysis unit and the instrumentation.

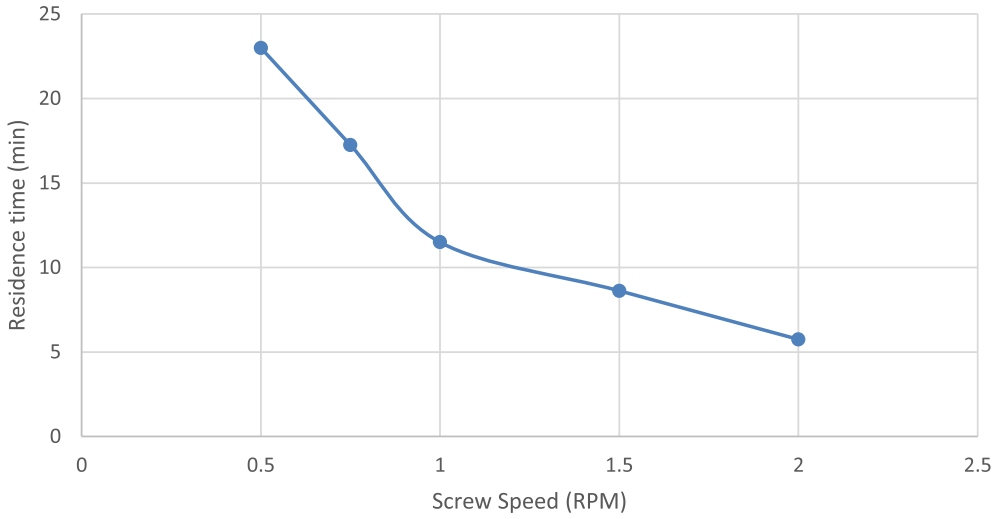

Fig. 5. Faecal sludge residence times at different screw speeds of twin auger pyrolizer.

plan due to feedstock shortage, pyrolysis efficiency at varying temperatures with a residence time of ~ 12 min was solely conducted in this study.

For the commissioning of the unit, the reactor was inclined 5° upwards toward the outlet to prevent the water from flowing into the char chamber. Furthermore, it was purged with 3 L of nitrogen for 1 h while the lower slider of the hopper was shut to evacuate the present air/oxygen through unit dismantling. Meanwhile the unit was heated up to a desired temperature and allowed for a steady heat profile. The partially dried feedstock was poured into the hopper and the top slider of the hopper was shut. Prior to opening of the bottom slider, the nitrogen purge was terminated, and the motors turned on. Each test was continued for 20 min to ensure the complete processing of FS from the hopper and the emptying of char from the unit via mechanical conveyance.

2.4. Pyrolytic product's analytical methods

Collection chamber was fixed on the outlet to collect the char at the end of each experiment. The gaseous products from the pyrolytic degradation of FS exit the reaction area using four pipes adjusted along the tube and pass through a stainless-steel condenser. At this point, the non-condensable fraction was collected in 3 L Tedlar gas bags while the condensable hydrocarbons enter a large vessel fitted at the bottom of the condenser. The condensation tower was dismantled after each experiment and rinsed with plenty of acetone and de-ionised water and further dried in an oven overnight at 105 °C.

Following the successful completion of each test, the syngas collected in gas bags was analysed for the content of combustible fractions and their yield was determined by the subtraction of biooil and biochar from 100 %. 9 mL of gases was dispensed in a syringe and three repetitions, each 3 mL, were injected into an Agilent 990 micro gas GC operated by SOPRANE II. The device contained two modules, MS5A SS 10 mm*0.25 mm*30um and a PoraPLOT Q UM 10 m*0.25 mm*8um heated at 80 °C and 60 °C respectively. Quantification of the chromatogram peaks were completed using standard calibration gas mixtures of 0.02 %, 0.2 % and 1 % containing CH_4, CO, C_2H_6, C_3H_8 and H_2 as well as CO_2. Energy content of syngas mixture was then calculated (assuming the selected

species contribute to the 100 % of the syngas fraction), using the theoretical data on the calorific value of various gas species available in literature [37 38].

Biooil samples were weighed and the qualitative analysis of the light and heavy fraction of the biooils were performed using a coupled Clarus 680 GC – Clarus SQ8 MS from Perkin Elmer. The chromatographic column used was a ZB-WAX (L = 30 m, ID = 0.25 mm) from Phenomenex suitable for polar materials with high purity grade helium as the carrier gas (1 mL/min). Similar to the method outlined by Gaojin et al. (2015), about 200 mg of biooil was dissolved in 5 mL of acetone and almost 3 g of anhydrous sodium sulphate was added to the mixture for dehydration due to the high moisture content of the feedstock [39]. The mixture was then filtered and 1 μL was automatically sampled and injected in the inlet, heated at 245 °C. The oven temperature was programmed as following: hold for 2 min at 50 °C followed by ramp up to 90 °C at 10 °C/min without holding and then temperature increase to 120 °C/min at 4 °C without holding followed by ramp up to 230 °C at 8 °C/min and hold for 10 min. The mass spectrometer's (MS) source was kept at 230 °C with the mass selective detector operating in electron impact (EI) ionisation mode at 70 eV.

The reactor was allowed to cool down and the solid char was weighed and analysed for their calorific value (HHV) after drying at 105 °C for two hours. The washings from the bomb calorimeter were analysed for the content of inorganic anions using a Dionex ICS-1600. To understand the proximate properties of the char, thermal gravimetric analyses were performed using a TGA8000 from Perkin Elmer. Similar to the method outlined by Saldarriaga et al (2015) for proximate analysis with TGA [40], the temperature program used for this analysis was as following: switch the purge gas to nitrogen at 60 mL/min and Hold for 1 min at 50 °C following which the furnace was heated to 105 °C at a rate of 30 °C/min and hold for 30 min for complete moisture evaporation, then the temperature was raised to 700 °C at a rate of 30 °C/min and held for 30 min for the volatiles to be driven off, then the purge gas was switched to air at 60 mL/min and held for another 15 min for the complete combustion of the samples and determination of fixed carbon and ash content. Lastly, the content of C, N, H were determined using an elemental analyser from ELEMENTAR. The content of oxygen was calculated by subtraction of C, H and N contents from 100 %.

3. Results and discussion

Raw faecal sludge samples were dried at 105 °C overnight and almost 9 mg was loaded into the carousel. The thermal gravimetric analyses on the faecal sludge at different heating rates were performed under 60 mL

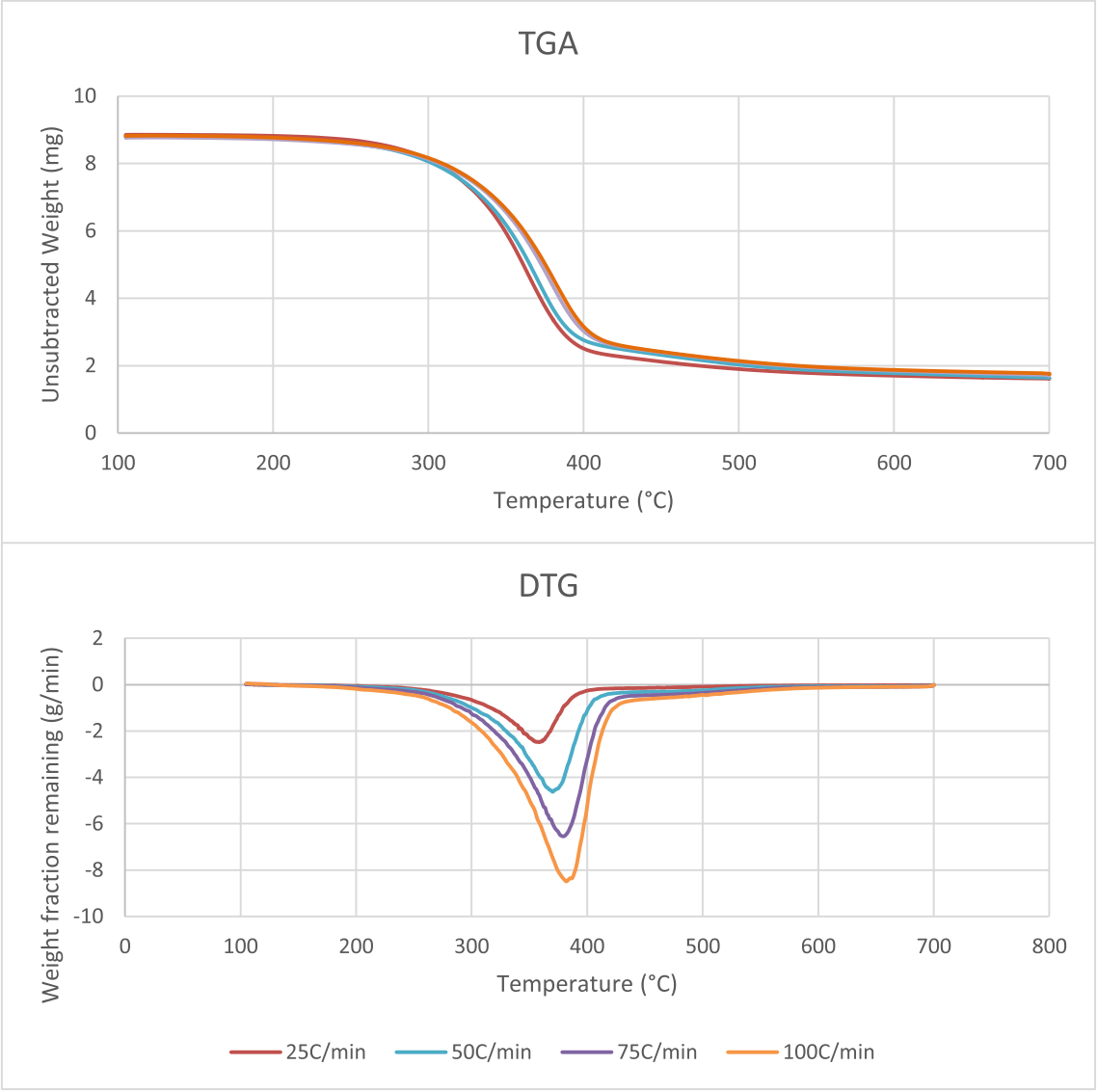

Fig. 6. TGA and DTG analysis of CET waste.

of nitrogen as the purge gas.

The evolution of sample weight respective to temperature and the differential thermal gravimetric (DTG) graphs are illustrated in Fig. 6. Faecal sludge samples as well as the tissue papers showed mainly a cellulosic degradation behaviour potentially due to the high tissue paper content in the waste [41]. Meanwhile, escalation of heating rate within the furnace resulted in a shift to higher temperatures in the thermal degradation curves implying enhanced heat transfer through sludge particles at lower heating rates. The maximum degradation points for the faecal sludge samples improvised around 375 °C during the slow/intermediate pyrolysis modes explored. Hence, together with the findings in our previous study on the suitable temperature ranges favouring the production of high-quality syngas through the pyrolysis of different biomass and wastes [1], the newly built unit was tested at three temperatures of 375 °C, 450 °C and 500 °C.

The moisture reduction efficiency of the dryer integrated within the system prior to the pyrolysis unit for different feed volumes entering the system through each toilet flush is provided in Table 3. The moisture content of all waste samples collected were measured as 91 % ± 2, Table 1.

Analysing the data on the reduction in moisture efficiency of the drying unit, high sensitivity of the dryer to the feedstock masses below 250 g was concluded. Moreover, collection of faecal waste from different sources showed lesser volumes of faecal waste than the figures considered for the design and manufacturing of the pyrolysis reactor. In addition, limitations on the sample collection due to COVID-19 situation further restricted the test plan. Hence, to facilitate the testing of the pyrolysis unit with limited feedstock volumes, batches of 100 g/cap/day and 250 g/cap/day of raw faecal waste for 3 persons were passed through the drying unit and the partially dried products were further fed into the pyrolysis unit. The selected mass figures for pyrolysis feedstock preparation potentially represent the lowest (A) and highest (B) limits of faecal sludge masses entering the system respectively, Table 4. It is notable that the results on the efficiency of the dryer in moisture reduction were not utilised during the fast-prototyping stage of the pyrolysis unit.

The chamber provisioned for the storage of partially dried feedstock prior to pyrolysis, enables an alternative operational frequency for the pyrolizers installed for toilets in different applications processing alternative volumes of sludge feedstocks.

3.1. Product distribution

The yield and distribution of products from the pyrolysis of feedstock prepared in test set A are provided in Fig. 8. The syngas yield curves were normalized by the subtraction of the wt.% of char and biooil from 100 wt% of feedstock. Completing the first test at 375 °C (test 1), due to shortage of raw faecal sludge and the incomplete conversion of the char shown in Fig. 7, no further test at this temperature (test 4) was conducted. The weight percentages for the biochar, biooil and syngas fractions obtained from test set B changed from 12.67 % to 11.50 %, 54.89 % to 52.71 % and 32.43 % to 35.79 % respectively, with an increase in the processing temperature from 450 °C to 500 °C.

Increasing the process temperature favoured the production of more biooil up to 450 °C in both sets of tests after which it declined due to occurrence of secondary reactions during which organic bonds are cracked [42 43 44]. Maximum yield of condensable fraction for the lowest (A) and highest (B) moisture content and masses of waste was 50.13 % and 54.89 % at 450 °C operating temperature of the reactor, respectively. It is notable that the residual biooil in the condensation tower (not obtainable through decanting) were not accounted for in these analyses.

Table 3
Dryer product's moisture content based on feed masses.

Feed mass (g)	Temperature (°C)	Residence time (min)	Product moisture content (%)
100	150	~60	53
160	150	~60	66
250	150	~60	70
400	150	~60	72

Table 4
Pyrolysis tests at different temperatures using the partially dried feedstock from dryer.

Test set	Test No.	Cap/day	Mass (~)	Total mass (g)	Temperature (°C)	Mass of partially dried feed (g)
A	1	3	100	300	375	52
	2	3	100	300	450	52
	3	3	100	300	500	54
B	4	–	–	–	375	–
	5	3	250	750	450	119
	6	3	250	750	500	123

It is known that the higher heating temperature leads to the production of more syngas. In addition, water vapour partial pressure in the reaction area contributes to the production of more syngas significantly through steam reforming of the volatiles with the catalytic help of the char [45]. However, variation of temperature in test sets A from 375 °C to 450 °C resulted in declination of syngas yield. Interestingly, this might be due to higher evaporation rates of feedstock moisture content [46], and lesser vapour residence time and subsequent cracking in the reaction area. Above 450 °C in both test sets the syngas yield underwent a negligible rise, which is associated with the overtaking of secondary reactions. Comparing the yield of products in test set A and B, results showed a boost in the production of biooil at 450 °C and above at the cost of syngas potentially as a result of higher moisture content of feedstock and higher feed flowrates. The increase in feedstock flowrate enhanced the production of more biooil in a study on the pyrolysis of sawmill residues in an auger reactor enabling shorter vapor residence times as a result of higher vapour volumetric flowrate within the reactor [43]. In addition to lesser cracking of condensable fraction in the reactor, the biooil deposition in the pipeline is depleted resulting in higher yields of liquid. Another reason for the improved biooil production at higher feed flowrates in test set B can be the inadequate heat transfer through the reactor wall to the sludge particles resulting in almost 7 % lower syngas production than test set A at 450 °C and above by slowing down the reactions. However, this observation with the feedstock flowrates is very much different in every study depending on the size and configuration of reactors [47].

Similar to other research on the pyrolysis of biomass and sludge feedstocks, the char yield decreased when the processing temperature climbed up [48 49]. However, the formation of char remained lowest due to the small content of ash and high volatiles in the faecal sludge feedstock (4 – 7 wt%) in a comparison to other sewage type solid wastes with higher ash contents (17 – 70 wt%) and relatively lower volatile matter pyrolyzed in different reactor configurations and setups [50 51 52 16]. High ash content and nutrients are commonly known to catalyse the char/coke formation [53]. Operating the unit at 500 °C in both test sets A and B, the yield of char remained higher in the former set, potentially due to longer vapor residence times, when feed flowrate is lower, leading to repolymerization of volatiles and further formation of char [54].

Analysis on the product yields demonstrated the key role of reactor configuration in optimal processing of sludge while highlighting the necessity of simultaneous evaluation of the effects of various process parameters. The pyrolysis unit has pipes installed throughout the tube, boosting the evacuation of vapours from reaction area. Hence, the yield of biooil remained dominant in all the tests as a result of higher volume and moisture content of the feedstock.

Fig. 7. Incomplete conversion of char obtained from test 1 at 375 °C.

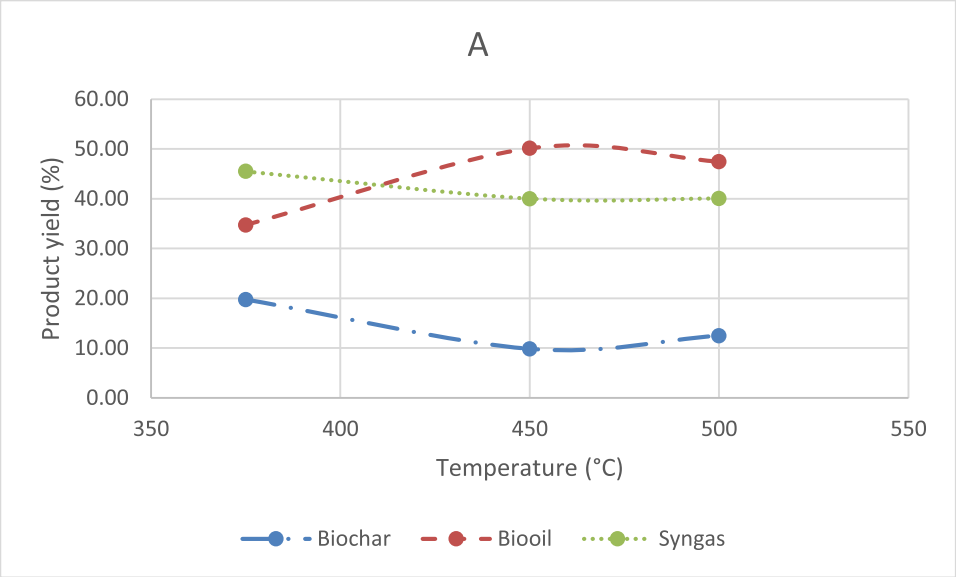

Fig. 8. Pyrolysis products distribution (Test set A outlined in Table 4).

Table 5
Pyrolysis gas composition under intermediate operating mode.

Test set	Test No.	Gas composition (mol%)						CV
		CH_4	CO_2	C_2H_6	C_3H_8	CO	H_2	MJ/Nm^3
A	1	1.0	48.8	0.3	0.6	31.4	2.0	5.4
	2	4.4	43.8	1.2	0.3	25.1	4.9	6.6
	3	6.7	44.4	2.1	0.2	27.7	8.8	8.9
B	5	5.9	44.3	2.0	0.3	25.5	1.5	7.4
	6	10.5	35.7	2.6	0.1	25.2	3.6	9.7

3.2. Syngas

Table 5 showcases the calculated concentration of syngas species for the feedstocks prepared and thermally converted at different temperatures outlined in Table 4. Results confirm the dominant presence of CO_2 followed by CO, CH_4, H_2 and C_2 – C_3 species. Enhanced production of CH_4 was observed with temperature escalation, a result of thermal cracking reactions, ranging up to 6.7 mol% and 10.5 mol% for A and B at 500 °C respectively. Similarly, a study on the pyrolysis of human faeces at low heating rates highlighted the maximum evolution of 35 % methane at 500 °C [6]. CO_2 pursued a downwards trajectory once the processing temperature was increased to 500 °C, reducing almost 5 %

and 8 % in A and B test sets. The peak concentration of CO was at temperatures lower than 400 °C ranging up to 31.4 % in set A decreasing almost 5 % at 500 °C.

Typically, CO and CO_2 are products of cellulose and hemicellulose decomposition while CH_4 and H_2 are attributed to the breakage in lignin fraction [55]. The comparative results observed for the CO_2 and CO are not in line with those confirmed for horse manure where CO is found to be the most abundant [48]. Analysis on the pyrolysis of sewage sludge in a tubular reactor has also shown an enhanced CO production alternating the temperature from 450 °C to 650 °C at the cost of CO_2 with the following reaction [42]:

Reverse Boudouard reaction

$$C + CO_2 \rightarrow 2CO \Delta H_r^0 = 173 kJ/mol \quad (1)$$

It is notable that the combined effects of process parameters also contribute to the evolution of these species complicating the result interpretation. Most researchers explore the effects of process parameters individually with a few ones investigating the interactions between process variables [54]. In this study, the dominance of CO_2 and CO are primarily attributed to the chemical properties of feedstock containing high C and O fractions followed by the oxidation (2) – (3), and water–gas reactions (4) – (5) [56], taking place as a results of air ingress through the hopper and bearing components of the system and high moisture content of feedstock.

Partial/oxidation reactions

$$2C + O_2 \rightarrow 2CO \Delta H_r^0 = -222 kJ/mol \quad (2)$$

$$C + O_2 \rightarrow CO_2 \Delta H_r^0 = -394 kJ/mol \quad (3)$$

Water-gas/shift reactions

$$C + H_2O \rightarrow CO + H_2 \Delta H_r^0 = 131 kJ/mol \quad (4)$$

$$CO + H_2O \rightarrow CO_2 + H_2 \Delta H_r^0 = -41 kJ/mol \quad (5)$$

H_2 underwent a surge with higher temperatures up to 8.8 mol% and 3.6 % for the feedstock used in set A and B respectively. The concentration of H_2 was significantly reduced once the feedstock flowrate and moisture content were increased in set B. Commonly reported, H_2 generation is almost doubled between temperatures of 700 – 1000 °C while the higher moisture content in the feedstock is known to promote the occurrence of secondary reactions boosting H_2 production [45 49 57 58]. The depletion of H_2 in a comparative analysis between set A and B implies the impacts of higher feedstock flowrate overtaking those from higher moisture content and processing temperature. The upsurge in the vapour pressure and flowrate within the reaction area shortens their residence times and consequently reduces the chance of secondary reactions (e.g., steam-methane reforming reactions) for higher hydrogen production [48].

Analysis on the C_2 – C_3 fractions showed an opposite evolution behaviour with temperature escalation that was negligible and thus not discussed in this study. Meanwhile, the discrepancies with the overall volume of gases detected (~80 %) in all tests is neglected in this study. Quantifying the results, the calorific value (CV) of the syngas mixture increased with higher temperatures mainly due to higher CH_4 and H_2 contents.

3.3. Biooil

Qualitative analysis of the biooil samples obtained from the intermediate pyrolysis of faecal sludge indicated a large number of oxygenated compounds similar to the findings in literature [39 59]. The software (Turbomass) was programmed to generate a report with the 30 of the largest peaks detected in each chromatogram having relative area > 0.5 % listed in supplementary materials. The identified compounds occupied only 50 – 60 % of the total biooil species implying their complex composition. The rest of the compounds could potentially be detected through enhancement of the rudimentary GCMS method utilised except the non-volatiles which are not detectable by GCMS.

Increasing the temperature in both test sets, a larger group of compounds were detectable with the same GCMS method. Meanwhile, comparing the biooil composition from test sets A and B showed a reduction in the total RPA[1] (%) concluding the presence of larger molecules with heavier molecular weights in the biooils from ladder test set which remained undetected. Typically, an increase in the feed flowrate results in shorter residence time for the gas phase in the reactor and lessens the production of light weight hydrocarbons through secondary cracking and repolymerisation of volatiles [60 61].

The most abundant species (RPA > 1 %) in all biooil samples are summarised in Fig. 9, showing the dominant presence of acidic components followed by *N*-containing hydrocarbons, alcohols, ketones and aldehydes. An example of collected biooil is shown in Fig. 10. Results indicated acetic acid as the principal acidic component in biooil from test 1 reducing with the overall acid concentration when temperature was raised to 500 °C. These findings are in line with the observations in a study on the fast pyrolysis of rice husk in a conical spouted bed reactor where temperature escalation increased the phenol concentration at the cost of lesser acids and ketones [62]. In contrast, pyrolysis of sewage sludge with the same reactor, yielded lower oxygenated compounds with higher temperatures as 600 °C except acids which maximised at 500 °C [63]. Increasing the feed flowrates and moisture content (~17 %) of the feedstock in test set B, yielded a totally lower abundance of oxygenated compounds similar to a study by Qureshi et al. (2021) on the pyrolysis of palm shell at 500 °C at different feeding rates [64]. On the contrary to the trends observed with the biooil composition at different temperatures in test set A, the extents of oxygenated compounds at 500 °C were slightly higher than those of 450 °C. This is potentially due to nature of the used feedstock in this test since all the process parameters (e.g., process temperature, moisture content and feed volume) have shown to favour the reduction of such compounds (through enhancement of secondary reactions or better ring-opening from feedstock) once increased [64 65].

The significant concentration of *N*-containing species in biooil from sludge materials is due to the dehydration reaction of peptide bonds in proteins which contribute to NO_x emissions in combustion applications, thus requiring removal [59]. Depicted in Fig. 9, *N*-containing compounds underwent a surge increasing the temperatures to 500 °C in both test sets. In a study by Jaramillo-Arango et al. (2016), the aliphatic and aromatic hydrocarbon concentrations from sewage sludge pyrolysis in fluidized-bed reactor reduced while the nitrogen-containing compounds climbed up at higher temperatures up to 800 °C [44]. It is likely that, the effects of temperature on the evolution of *N*-containing compounds in this study have overtaken the opposite influences of moisture content and feed flowrate in a comparison between biooil composition in set A and B [64]. Mei et al. (2020) reported a drastic reduction of *N*-containing compounds once the fuel's moisture content exceeded 20.57 % [45].

3.4. Biochar

Chemical properties of the biochar derived from the pyrolysis of faecal sludge samples are summarised in Table 6. The volatile matter content of the pyrolytic solid residues reduced 61 % and 40 % in test set A and B respectively, in a positive correlation with their calorific value. This observation was similar to other studies on the production of biochar from human manure [52], while higher temperatures contributed to higher fixed carbon contents. The reduction of calorific value in biochar at higher processing temperatures could also be attributed to the higher ash contents of biochar samples containing 24 % and 32 % more

[1] RPA: Relative peak area.

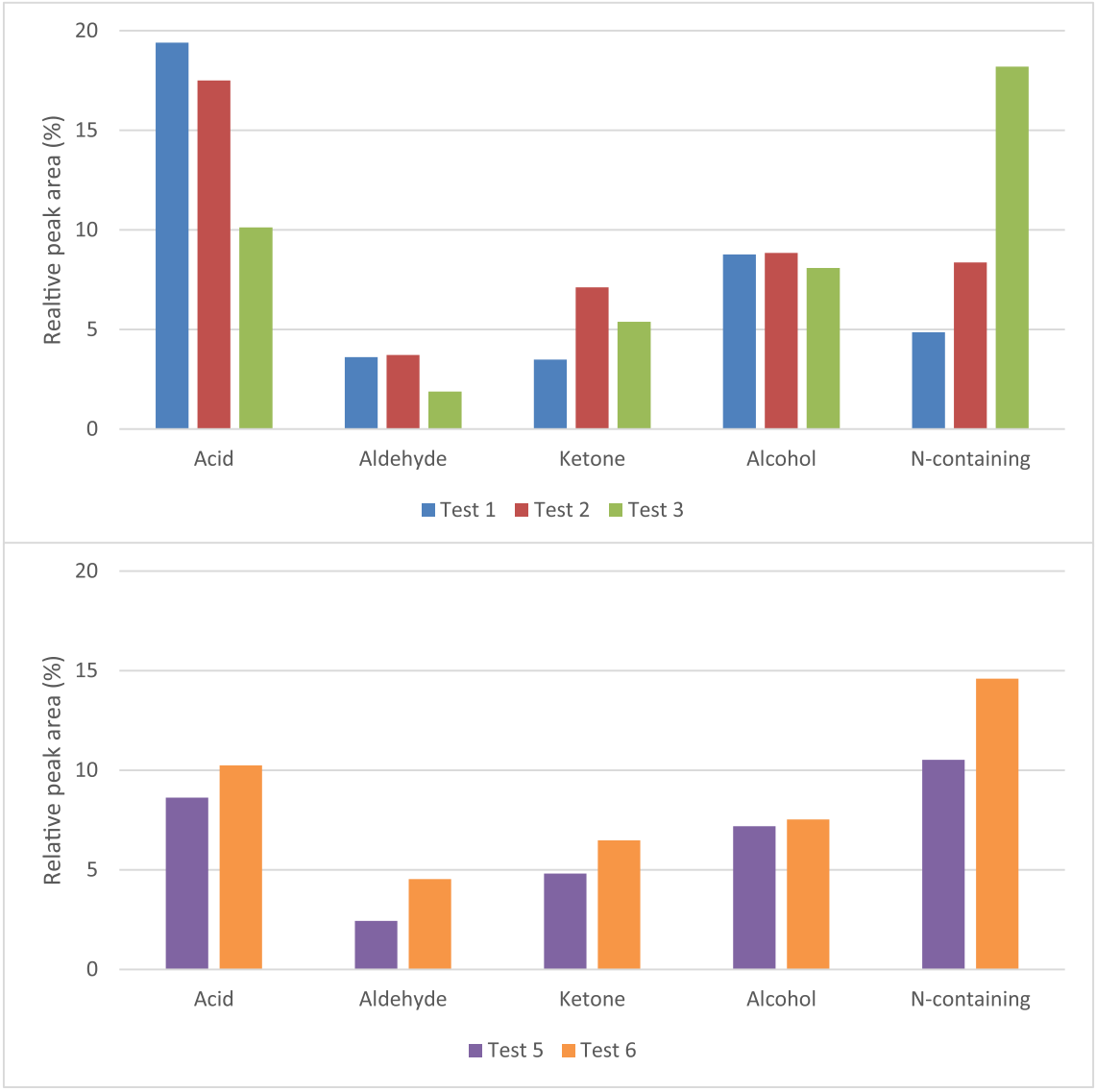

Fig. 9. Major hydrocarbon species in biooil samples from pyrolysis tests in test sets A and B.

ash in comparison to their parent faecal sludge (5 ± 2 %, dry basis) shown in Table 1 [59]. This is a unique feature in the biochar made from sewage sludge in contrast to those of biomass materials resulting in higher ash contents with the increasing temperature due to significant retention of inorganics in the biochar matrix [67 68].

The calorific value of the resultant char (20 ± 2 MJ/kg) remained close to that of human faeces and sewage sludge [69 5], but was less than biomass derived chars [64]. The high fixed carbon content of the biochar makes them attractive for contaminant removal such as H_2S in adsorption applications [70 71]. Comparing the carbon content of biochar in between experiments in set A and B, the insignificant effects of secondary reactions (1)(3)(4), in comparison to temperature can be concluded. Mosko et al. (2022) reported a significantly lower carbon content for the biochar obtained from the pyrolysis of sewage sludge in quartz reactor at higher temperatures up to 800 °C conducted under purge gas with 70 vol% water content due to reverse Boudouard reaction [72]. In the pyrolysis unit developed in this study, the gases exit the reaction area immediately reducing the further chances of char-gas interaction.

Finally, significant retention of chlorine in the biochar was observed while the content of sulphur and phosphorus reduced to almost half when the temperature was raised from 375 to 500 °C in test set A.

3.5. *Utilisation of products*

Pyrolysis conversion of solid fuels deliver multiple products that need to be handled properly due to the presence of various inorganics and toxic elements as well as heavy metals in the solid biochar and biooil fractions [73 74]. The main use of the syngas can be for the immediate combustion and energy recovery to limit the energy consumption of the system. In addition, the biochar fraction can be used for energy recovery purposes. However, the exhaust of such a combustion process will need further emission controls to limit the concentration of pollutants below regulative figures for toxic and hazardous elements prior to disposal in the environment. For the use of the biochar as fertilizer, further analyses on the contents of heavy metals and nutrients would be necessary to meet certain standards [74]. The effects of process parameters on bio-oil composition are extensively investigated for different biomass and waste materials individually with a few ones exploring the simultaneous impacts. It is notable that, reactor design plays a key role on biooil composition in parallel to other process conditions [60]. Thus, it is essential to identify the end-use application of biooil before planning any reactor configurations to maximise the quantity of various desirable species. In the case of off-grid utilisation of biooils from this process, phenols can be used as low-cost renewable resins, ketones are suitable

Fig. 10. Biooil from test 6 containing high water content with solubilizing acids, alcohols and ketones from carbohydrate decomposition [66].

Table 6
Chemical characteristics of biochar samples.

		Test Set A			Test Set B	
		Test 1	Test 2	Test 3	Test 5	Test 6
arb%	M_C	2.26	2.66	3.12	1.42	1.46
Dry basis %	V_M	31.43	22.18	12.08	17.25	10.36
	F_C	44.23	49.27	58.45	46.46	52.57
	Ash	24.33	28.53	29.46	36.27	37.05
	C	47.48	47.99	49.84	52.81	52.63
	N	1.88	1.96	2.05	2.37	2.55
	H	4.83	3.47	2.76	2.81	2.29
	O	45.81	46.58	45.35	42.01	42.53
ppm	**Cl**	6272.26	6067.78	11029.72	4966.42	5484.76
	S	895.43	558.12	514.44	558.68	515.63
	P	6336.84	5886.04	3215.63	4610.68	3998.98
mg/L	NO_3^-	467.43	295.21	198.97	312.57	272.36
	NO_2^-	2.9067	6.15	2.04	5.18	5.89
HHV (MJ/kg)		22.17	21.68	21	20.29	19

biofuel compounds forming esters upon oxidation, organic acids upon fractionation could be used for the production calcium salts, an environment friendly road de-icer and aldehydes are a potential renewable fuel for fuel cells [75 66 76 77]. For the recovery of energy contained within biooil, production of furans, phenols and cyclopentenone are of favour ensuring a good combustibility of the biooil [78]. It is notable that the biooils generated in this research are predicted to contain high water contents which makes them unsuitable for combustion purposes.

4. Conclusions

A novel micro-scale twin auger pyrolysis reactor for the conversion/stabilisation of highly wet sanitary faecal sludge was designed and successfully commissioned. The study confirmed the lesser sensitivity of pyrolysis to the fuel's physiochemical properties (e.g., moisture content and particle size) in contrast to gasification and combustion. In the case of pyrolysis, prolonged residence times alongside temperatures from 450 °C and above in such reactor design ensured the conversion of raw solids. However, the extent of conversion and product properties altered. This design pursued the maximum volume reduction (~88 % ± 1) of waste to biochar up to 500 °C with the immediate evacuation of hydrocarbon vapours from reaction area using no carrier gas to limit volatile repolymerisation. The low energy content of the syngas (~8 – 9 MJ/kg) implied the need for a prolonged residence time of solid and gas phases within the reactor to promote the production of higher calorific value syngas for limitation on energy consumption upon its recovery. However, in parallel to the effects of process parameters (e.g., temperature and feed flowrate), the importance of reactor design and configuration on the existing trade-off between the yield and composition of product biofuels was highlighted in this study.

Extending the gas/vapour residence times, more biochar could be yielded while the longer chain oxygenated compounds break into lighter weight species. Acids and *N*-containing species formed the most abundant hydrocarbon species (~10 and 17 % respectively) in the obtained biooils at 500 °C. Typically, direct use of the biooil is impractical due to their high-water content which on the contrary facilitates their transportation due to promotion of lower oil viscosities enhancing their flow properties in burners and atomizers. It is essential to identify the end-use of the pyrolytic products prior to any application for a better design and configuration of the reactors. The biochar contained ~ 58 % fixed carbon confirming suitable adsorption properties. The concentration of nutrients such Phosphorus and contaminants (Cl and S) should be evaluated according to the existing regulatory limitations for thermal processes.

For future work, further tests for a better understanding of feed flowrates in the real-world scenario are favourable. Assessment on the effects of fuel with other moisture contents in necessary whilst the reactor design implications could be planned ahead according to the fate of pyrolytic products.

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CRediT authorship contribution statement

F. Beik: Conceptualization, Methodology, Investigation, Data curation. **L. Williams:** Conceptualization, Writing – review & editing, Supervision, Funding acquisition. **T. Brown:** Conceptualization, Resources, Supervision, Funding acquisition. **S.T. Wagland:** Conceptualization, Methodology, Writing – review & editing, Supervision, Funding acquisition.

Declaration of Competing Interest

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

Data availability

Data will be made available on request.

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