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Potential Bio-oil Production from Smouldering Combustion of Faeces

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Abstract This study examined the potential bio-oil production from the smouldering combustion of faeces mixed with sand, through a series of experiments. Surrogate faeces, with demonstrated equivalence to human faeces, were used in order to minimize variability in the composition. The yield of bio-oil was studied as a function of the following experimental parameters: original moisture content of the faeces, airflow rate and sand-to-faeces mass ratio. The amount of bio-oil collected was shown to be dependent on the airflow rate and the relative amount of sand used but independent on the moisture content. The bio-oil obtained was characterized by ultimate analysis, water content and calorific value. Under the experimental conditions studied, up to 70 g of bio-oil per kg of dry faeces (HHV = 27.6 kJ/g) can be produced. Finally, experiments demonstrated that sand can be reused after each experiment. The effect of the ash accumulation in the sand matrix on smouldering was investigated over five successive tests with the same batch of sand. The implementation of this technology to other type of organic waste would contribute to the development of an integrated waste treatment technology, in combination with bio-oil production.

Introduction

The alternative treatment of faecal matter and human waste has been growing attention during the last years due to the substantial amount of water required in the conventional flush toilet [1] and the necessity of affordable and effective methods for faeces treatment in developing countries [2]. Furthermore, as human faeces are produced in large quantities [3], the potential for energy recovery and its valorization should not be disregarded. Valorization of faeces, providing a solution to sanitation, would make the alternative technologies more feasible from an economic perspective.

Recently, smouldering as a treatment for human faeces mixed with sand has been proposed and studied for the first time [4]. This was in response to the Bill and Melinda Gates Foundation project *Reinvent the Toilet Challenge*, launched in 2011 [5] to develop an integrated, low cost, off-grid toilet that rapidly disinfects human waste [6]. The combination of temperature–time achieved during the smouldering process ensures pathogen destruction.

Smouldering combustion is a slow, relatively low-temperature and non-flaming form of combustion [7, 8]. A common example of smouldering is glowing red charcoal in barbecues. Smouldering combustion can be self-sustaining meaning that energy input is not required after ignition [9]. While flaming combustion involves the gasification of the fuel and subsequent oxidation of the gas phase, smouldering is the oxidation of a condensed phase on the fuel surface. During smouldering the heat loses are minimal compared to incineration [10] allowing

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smouldering of fuels with heat of combustion too low or with moisture content (MC) too high for incineration. Due to its high energy-efficiency, self-sustaining smouldering has been applied as a thermal treatment for different types of waste [4, 11, 12], especially those with high moisture content such as faeces.

Smouldering needs a porous medium for the propagation of the combustion reaction, enhancing oxygen diffusion, and thermal insulation [13]. Hence, smouldering can occur in solid fuel when they are porous (polyurethane foam [14], wood [15]), or in liquids materials when they are embedded in a porous matrix.

In the case of faeces, sand is used as a porous medium; it is an appealing matrix for smouldering treatments because it is widely available and has been proved successful in smouldering for soil remediation [16]. The sand reutilization after the smouldering treatments could save important costs, mainly associated with transportation. Importantly, smouldering of faeces was demonstrated to be self-sustaining over a wide range of conditions by Yermán et al. [4], meaning that the utilization of supplementary fuel is not required. Faeces can be treated with moisture content up to 75 %, depending on the smouldering conditions. This would allow the elimination of the energy-intensive drying step. That paper also presented the range of operational conditions where self-sustaining is possible, showing an interdependent relationship between the experimental parameters. For example, the moisture content can be increased if the pack height mixture is reduced, and the airflow can be reduced if the relative amount of sand is increased.

The sensitivity of the smouldering performance as a function of these operational parameters was later studied in [17], while the influence of the reactor scale in [18–20]. Smouldering has also been employed for the treatment of wastewater biosolids up to 80 % moisture content [21]. Smouldering performance was assessed by means of peak temperatures and velocity of propagation of the smouldering front.

In related work, self-sustaining smouldering has been shown to be effective in decomposing used tires and pyrolysis oil recovery [11]. To the authors' knowledge, that study is the only publication examining fuel production through smouldering combustion. That study showed maximum oil recovery at 35 %, expressed as the mass of oil relative to the initial mass of the tires. The yield of oil recovery was observed to be dependent on the airflow rate used. Those results suggest that the experimental parameters should have an impact on the extent of pyrolysis reactions and the yield of oil produced.

Having demonstrated the potential implementation of this technology for faeces treatment, this paper aims to demonstrate the feasibility of producing pyrolysis products, such as oil, during the treatment of such waste. The nature and moisture content of faeces makes this waste very challenging, and the oil yields are not expected to be as high as for used tires. This is mainly because water and sand act as an energy sink, leaving less energy available to feed the pyrolysis reactions.

In this work we performed a series of smouldering combustion experiments of surrogate faeces mixed with sand to determine the amount of bio-oil produced as a function of the following experimental parameters: airflow, moisture content of the faeces, and sand-to-faeces mass ratio (S/F, dry basis). In order to perform a systematic study, a surrogate faeces formula was used due to the high variability of the real faeces. The bio-oil collected was characterized by means of ultimate analysis, water content and higher heating value (HHV). Additionally, as it would be ideal to recycle the sand for minimizing material costs, the reutilization of the sand after the experiments and the effect of the ash accumulation in the sand matrix on smouldering were investigated over five successive tests.

Materials and Methods

Materials

Experiments were carried out using a surrogate formula based on a report by NASA [22]. The list of the ingredients of the formula, the function of each ingredient and the equivalence to real faeces are presented in [4]. The surrogate faeces formula and the bio-oil collected from the experiments were characterized by its calorific content (HHV) using a bomb calorimeter and by ultimate analysis in a Carlo Erba—CHNS Elemental Microanalyser, model NA 1500. The values from both analyses are the average of three repetitions.

A new batch of fresh surrogate formula was prepared immediately prior to conducting the experiments. The ingredients were mixed in a food blender, into which sand was subsequently introduced. These experiments employed commercial sand with a sand grain size between 0.6 and 1.2 mm (0.9 mm on average).

Experiments

Smouldering combustion experiments were carried out in a 316 stainless steel tube as shown in Fig. 1. The reactor tube is 16 cm in internal diameter and 100 cm in height, placed over a stainless steel base. The bottom of the base contains an air diffuser and at the top of the base an electric heater is placed, connected to a solid state relay to control the power. The column is insulated with mineral wool insulation (Rockwool®, 50 mm thickness, R-4.0) that is firmly secured to the column to minimise heat losses.
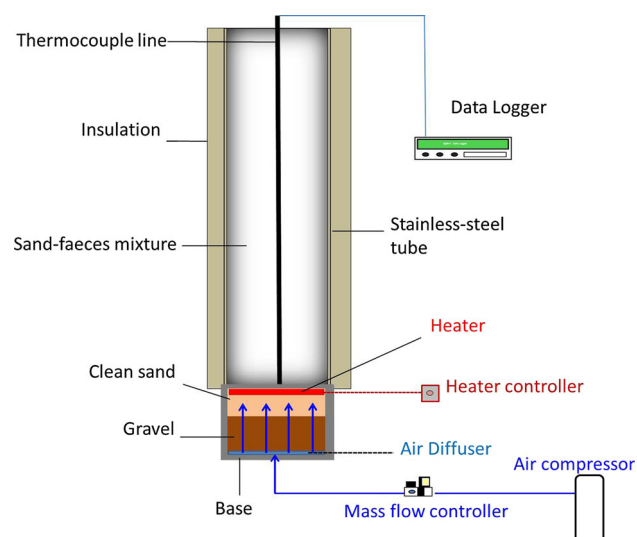
Fig. 1 Schematic representation of the reactor used in the experiments

The base is filled with gravel to bury the air diffusor. Atop this layer, sand is placed to cover the heater coil. An air compressor connected to a mass flow controller supplies air to the reactor through the air diffuser. The diffuser consists of a ring-shaped tube with six pairs of diametrically-opposite holes. Once the base is filled with gravel and sand, the reactor tube is put into position and filled with the sand-faeces mixture.

The propagation of the reaction is recorded by 24 Inconel-sheathed thermocouples (TC) placed along the axis of the tube. The temperatures from each TC are recorded every 5 s using a data logger. The first five TCs (TC1 to TC5) are placed in 1 cm intervals to have more resolution in the ignition zone, starting with TC1 at 2 cm from the heater. Beyond TC5, they are placed at 5 cm intervals. The temperature histories obtained from a typical self-sustaining smouldering experiment can be seen in Fig. 2. The experiments begin by heating the bottom layer of mixture until the temperature at TC1 (2 cm from the heater) reaches a certain temperature (250 °C in the figure). At this point, the airflow is initiated using a differential pressure mass flow controller (Cole Parmer 32907-75). The igniter is turned off when the temperature at TC1 peaks and the smouldering reaction then propagates upward. This procedure and configuration yields a robust, repeatable ignition across a wide range of conditions [9, 16]. The successive peaks (in the absence of external energy being applied) indicate the advancement of the smouldering front in a self-sustaining manner.

A series of nine experiments was performed using a constant mass of faeces (500 g, dry basis) under different conditions of MC, S/F and airflow rate. Experiments repeatability was already demonstrated in [4, 17]. Here,

every experiment was repeated at least twice. The reactor exhaust was bubbled through 1000 g of ice water in an aluminum vessel, which was submerged in an insulated ice water bath to maintain low temperatures to condense water and other compounds present in the exhaust. The non-condensable emissions were sent to an extraction system, while the captured condensate separated at rest into a lower aqueous layer and an upper non-aqueous layer. The non-aqueous-layer (bio-oil) was then quantified using a separatory funnel.

The oily phase was characterized by HHV and elemental analysis. The amount of CO, CO_2 and O_2 in the effluent gas was analyzed in a ADC MGA3000 multi-gas analyzer. Volatile fatty acids (VFA) were quantified using GC/MS for an aqueous phase sample that was collected, in this single case, using a vertical series of conical metal screens in line with the reactor's exhaust.

Finally, a series of five consecutive experiments, under the same experimental conditions (50 % MC, 7.5 S/F and airflow 80 L/min), and using the same batch of sand was performed to study the feasibility of sand reutilization.

Results and Discussion

Characterization of Faeces

The consistency and appearance of the surrogate formula is similar to real faeces. Table 1 summarizes the HHV and proximate analysis results of the surrogate formula. Both are compared to those reported for human faeces. The heat of combustion for the surrogate is 19.8 kJ/g (dry basis), which is within the range 17.6–25.1 kJ/g (dry basis) reported for human faeces [23–25].

The carbon and hydrogen content of the surrogate formula are comparable to that reported for human faeces [26], while nitrogen and sulphur contents are lower in the surrogate formula. The equivalence of this formula to real faeces was previously demonstrated [4]. The present study demonstrates the feasibility of treating and disinfecting faeces in combination with pyrolysis oil production. The quality and characteristics of the pyrolysis oil produced in a real scenario must be assessed for every case and the operational conditions (e.g. porous medium, airflow, mixing waste from different streams) may change. Hence, we can assume the difference observed in N and S composition will not affect the study presented here.

Pyrolysis During Smouldering

Results from thermo-gravimetric analysis (TGA) under nitrogen atmosphere (presented in [4]) showed that pyrolysis of surrogate faeces occurs at temperatures between

Fig. 2 Example of temperature histories obtained from a self-sustained smouldering of surrogate faeces experiment
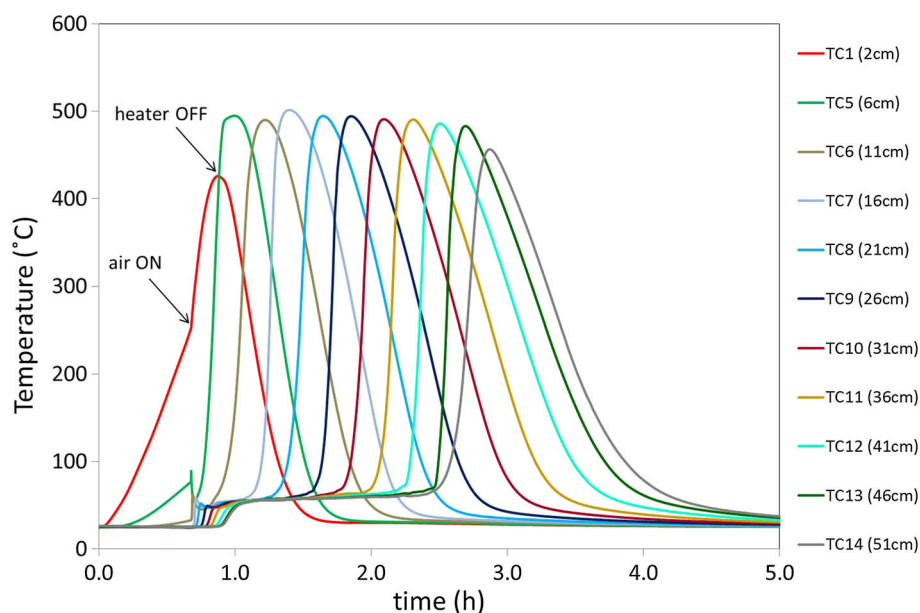

Table 1 High heating value (HHV) and elemental composition of surrogate and human faeces (dry basis)

Faeces	HHV (kJ/g)	% C	% N	% H	% S
Surrogate	19.8 ± 0.3	41.9 ± 0.5	1.5 ± 0.2	7.8 ± 0.2	0.00 ± 0.01
Human	17.6–25.1[a]	42.5 ± 2.5[b]	4.1 ± 0.4[b]	7.2 ± 0.7[c]	0.60 ± 0.10[c]

[a] [23–25], [b] [26], [c] [22]

200 and 500 °C, with maximum decomposition close to 350 °C. The information obtained from these TGA is crucial to understand how the yield of pyrolysis products can be maximized during smouldering.

Figure 3 shows the vertical spatial distribution of temperature and oxygen concentrations for a smouldering combustion reaction depicted at a particular moment in time. As the air enters from the bottom (x = 0), the oxygen concentration is initially at ambient air conditions ($Y_{O2,i}$). The first region (Zone I) is defined as the region where all fuel has been consumed by the passing smouldering front. In this zone no fuel is present; no reactions are occurring and therefore the oxygen remains constant at ambient concentrations. The temperature increases toward the combustion zone (Zone II).

In the Zone II, oxygen concentration decreases gradually as it is consumed in oxidative reactions. Temperature increases at higher rates due to the exothermic nature of these reactions. The point where the temperature drops marks the end of the smouldering front and the beginning of pyrolysis (Zone III) of the unreacted fuel above (T_P).

Ahead of the smouldering front heat is transferred via conduction, convection, and radiation to the unreacted sand and fuel. As the available heat decreases, the temperature eventually reaches a critical point (T_H) where pyrolysis cannot be sustained. The extent of the pyrolysis zone (ε)
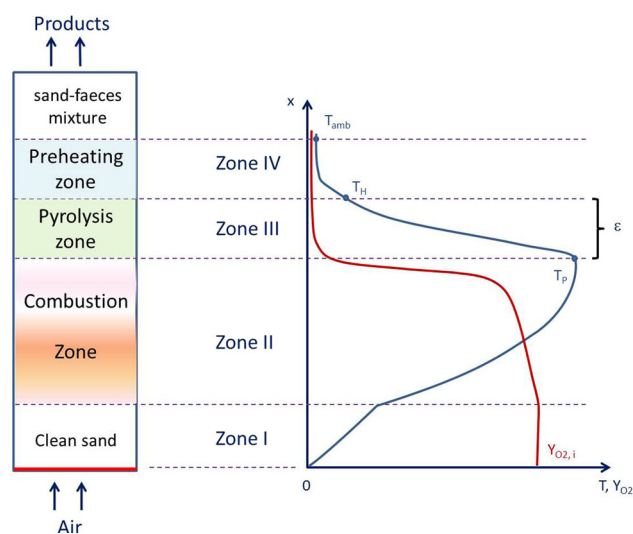

Fig. 3 Relationship of temperature and oxygen concentrations through the propagation of a smouldering front

would determine the amount of pyrolysis products obtained. Therefore, the yield of pyrolysis products can be increased if ε is increased.

The end of the pyrolysis zone marks the beginning of the preheating region (Zone IV) where heat is consumed through preheating of the unreacted zone. As the available

heat decreases, the temperature eventually reaches ambient temperature (T_{amb}), which delineates the end of the pre-heating zone.

The length of the pyrolysis zone is estimated as the length in the temperature spatial distribution (Fig. 4). The start of the pyrolysis zone corresponds to the peak temperature (T_P), while the end of the pyrolysis zone (T_H) is chosen to be 200 °C according to the temperatures observed in the TGA under pyrolysis conditions. The selection of these temperatures is an approximation and the ε values estimated here should be considered only for comparison. Figure 4 shows the plot of the temperature evolution as a function of the reactor's vertical axis for different times through the experiment where T_P, T_H and ε are indicated, as an example, for 180 min. For each experiment, ε was calculated as the average over ten different times and at a minimum distance of 10 cm from the heater to avoid its influence. The estimated values for ε as a function of different experimental parameters are presented in the next section.

Bio-oil Production

Table 2 presents the average product distribution obtained in the smouldering experiments of surrogate faeces. The smouldering conditions chosen allow for complete combustion of the organic matter, leaving 5 % ash (i.e., inorganics). This value is in agreement with the inorganics present in the surrogate formula. 65 g of liquid are obtained in the condenser per 100 g of dried faeces, in which 7 g corresponds to the oily phase and 58 g to an aqueous phase. The remaining 37.3 % of faeces mass is converted into gases. The mean dry gas concentrations by

Table 2 Average mass balance of the products obtained during smouldering combustion of faeces

|	wt%	vol%
Solid/ash	5.0	
Liquid	57.7	
Oily phase	5.9	
Aqueous phase	51.8	
Gas[a]	37.3	
O_2		10.5
CO		2.9
CO_2		7.7
Total	100	

Values do not consider the original water present in the faeces

[a] By difference

volume (±1 SD) across all nine experiments used for the oil mass recovery data are: CO_2: 7.7 % (±1.1 %); CO: 2.9 % (±0.6 %); O_2: 10.5 % (±1.9 %). These results showed that the volume of these gases add up to the original injected O_2 (21.1 %), meaning that while other gas components were not analyzed, its composition is not significant from a fuel perspective. These values represent a CO/CO_2 ratio of 0.377, which is typical for smouldering processes [27].

The effect of the original moisture content of the faeces on the bio-oil production is shown in Fig. 5. In these experiments, the mass of oil ranges between 54 and 62 g per kg of dry faeces. With a variation of only 12 %, the mass of bio-oil produced appears to be roughly independent of the original moisture content of the faeces, at least in the range studied (0–40 %). Figure 5 also shows the extent of the pyrolysis zone determined for these

Fig. 4 Temperature distribution showing the beginning (T_P) and the end (T_H) of the pyrolysis zone at 180 min
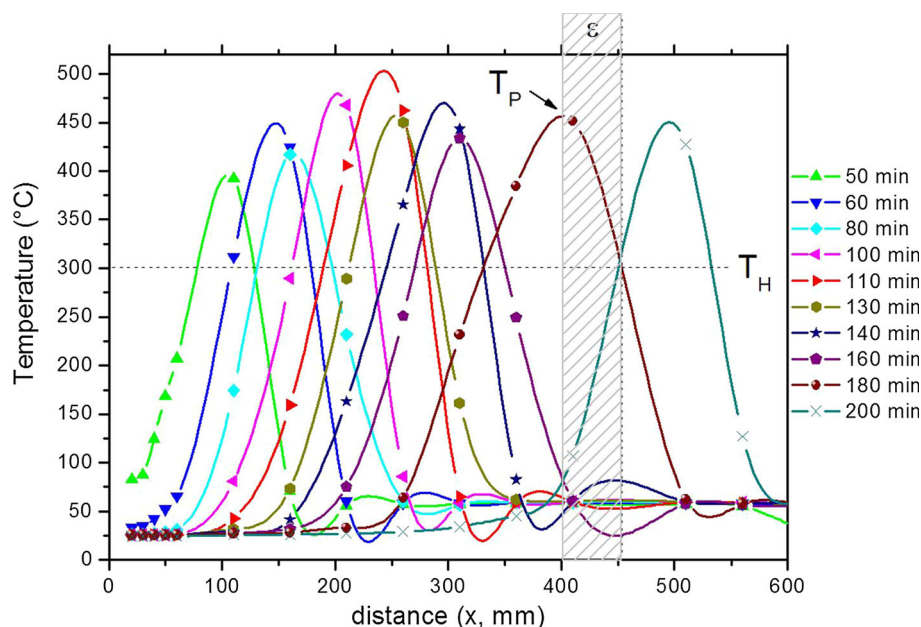
experiments. ε was found to be 52 mm for completely dried faeces (MC = 0 %) and 53 mm for the other 2 cases, suggesting that ε is also independent of moisture content. These results are reasonable since the fuel is completely dried in the pyrolysis zone. The drying of the mixture is occuring at some point in Zone IV (between T_H and T_{amb}).

A different trend is observed for the experiments where the airflow rate was varied. The results for the mass of bio-oil and ε at airflows between 30 and 50 L/min are shown in Fig. 6. In this case, both the mass of oil collected and ε increase with the airflow rate (from 44 to 62 g per kg of dry faeces, and from 49 to 58 mm, respectively). In these experiments it was also observed that higher airflow rates lead to higher smoulder temperatures (averages from 537 to 573 °C). As the peak temperatures (T_P in Fig. 4) are higher, this leads to an expanded pyrolysis zone (ε), as the difference between T_P and T_H is extended.

Figure 7 presents the bio-oil recovered and ε as a function of the sand-to-faeces mass ratio between 15 and 25 g/g. The mass of bio-oil varied from 57 to 71 g per kg of dry faeces, while ε fluctuated between 53 and 58 mm. In both cases, the graph presents a minimum at S/F 20 g/g. As for the case of Fig. 6, the drop in ε from S/F 15–20 g/g can be explained by the drop in the observed temperatures (608 and 555 °C, respectively). However, the smoldering temperature continues decreasing with further increments of S/F (542 °C at S/F 25 g/g), while ε increases. This can be explained by the fact that the thermal conductivity of the sand is lower than that of the faeces, which acts as an insulator [4]. The increased insulation produced by the increased amount of sand at S/F 25 g/g would provoke a slower cooling of the mixture from T_P to T_H, increasing the extent of the pyrolysis zone. The effect of the increased thermal insulation would have greater impact than the effect of increased temperature at higher S/F ratios.
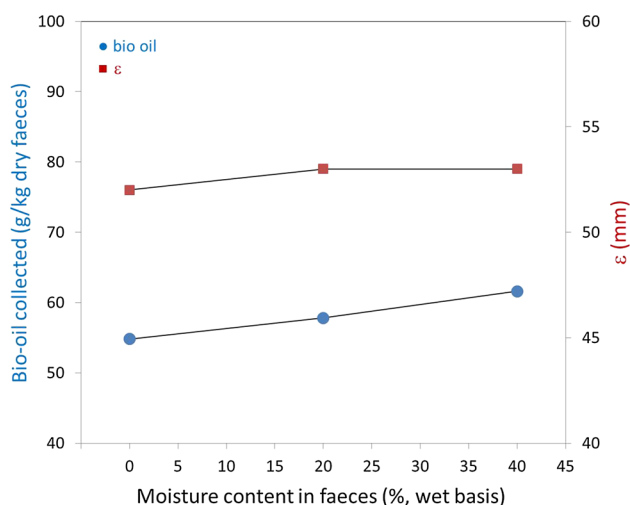

Fig. 5 Mass of bio-oil collected (g/kg dry faeces) as a function of the moisture content, at 20 S/F (dry basis) and airflow 40 L/min
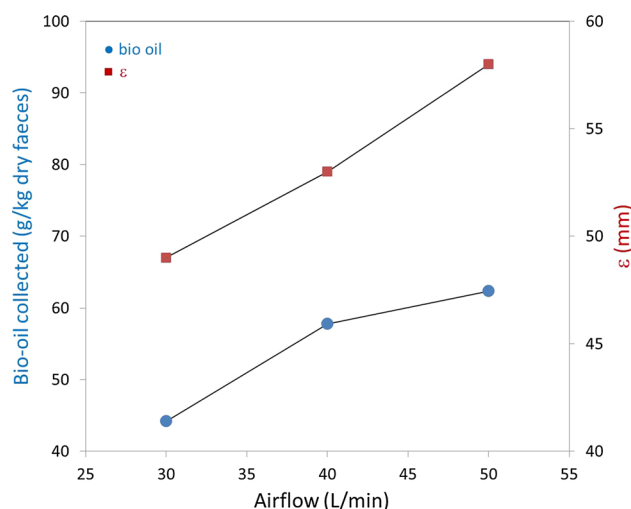

Fig. 6 Mass of bio-oil collected (g/kg dry faeces) as a function of the airflow rate, at 20 S/F (dry basis) and 20 % MC
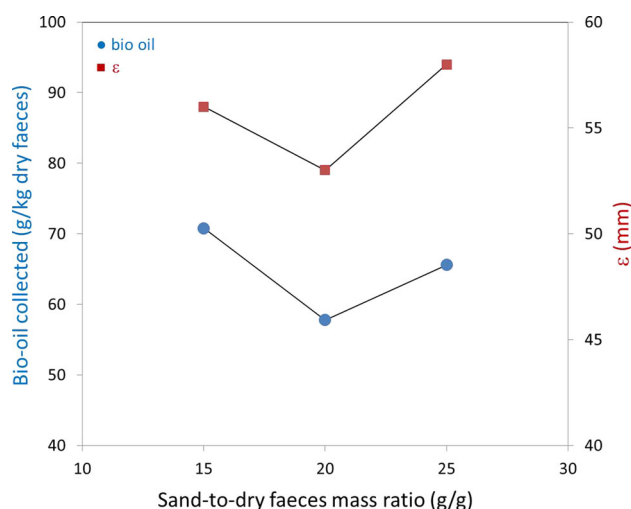

Fig. 7 Mass of bio-oil collected (g/kg dry faeces) as a function of the sand-to-faeces mass ratio (dry basis), at 20 % MC and airflow 40 L/min

The results showed in Figs. 5, 6 and 7 demonstrate that there is an interrelationship between the extent of the pyrolysis zone and the amount of bio-oil produced. Although further study is required, the results presented here demonstrate two important things: (1) the feasibility of producing bio-oil through smouldering combustion of faeces, and (2) the variation of the bio-oil yields with the smouldering conditions such as airflow rate and relative amount of sand.

The composition of the bio-oil collected during the different experiments does not vary substantially. Table 3 presents the average values obtained from the moisture content and proximate analysis as well as the higher heating value (HHV) from calorimetry. The bio-oil

exhibited an average water content of 56 wt%. The typical water content of bio-oil is 15–30 %, although it has been reported up to 60 % [28, 29]. The presence of water is not desired because it lowers the heating value. However, the water could help to decrease the viscosity, enhancing fluidity, which is desired for its atomization in an engine [29].

Ultimate analysis of the bio-oil showed the following values: 74.1 wt% C, 18.4 wt% O, 2.1 wt% H and 1.0 wt% N. No sulphur was present in the bio-oil, which is not surprising since it was not present in the surrogate faeces. The oxygen content is another important parameter to evaluate the oil quality. Low oxygen content is desired because it leads to higher energy density. The oxygen content of bio-oil is typically 35–40 % while the higher heating value is reported within the range 15–20 kJ/g [30]. In this case, the oxygen content found in the oil collected from the experiments is lower than the typical values (18.4 %), which coincides with the HHV that is above the typical values reported for bio-oil (27.6 kJ/g).

The aqueous phase is cloudy and brown in colour (see Fig. 8). The aqueous fraction of bio oil may be useful to process as it contains sugars, organic acids, phenols and other organic species that can potentially be converted to valuable products. However, the presence of organic acids can impose more demands on the construction of equipment (e.g. vessels) and on the upgrading process [31]. However, these organic acids can be easily extracted from the bio-oil by adding water and further phase separation. Table 4 shows the VFA concentration in the aqueous phase of the bio oil. A total of 60.46 g of VFAs are produced per litre of aqueous phase. Lighter acids (formic and acetic) are present in significantly higher concentrations, and concentrations tend to decrease with increasing length of the carbon chain of the acid. These concentrations are similar to reported values for conventional methods of bio-oil production [29].

Fig. 8 Photograph of two beakers containing aqueous and non-aqueous condensate phases collected from the exhaust of a smouldering experiment. The aqueous phase is highly diluted by the water initially present in the water bubbler used to collect these emissions

Sand Reutilization and Post-treatment Matrix

Smouldering consumes all of the organic matter and the inert sand matrix is found to be clean after each experiment. A change in colour from brown to red is observed in the sand after the first test (Reg Sand and Test #1 in Fig. 9). This is attributed to the oxidation of the iron compounds in the sand [13]. The inorganic species, primarily the calcium phosphate added to the surrogate formula, are not consumed during smouldering and are retained in the sand.

This treatment technology aims to continually reuse the sand matrix mixing the sterilized, clean sand from the last batch with fresh faeces for the next treatment. As the sand is hot after the experiments, it could contribute to pre-drying the sand-faeces mixture, decreasing the initial moisture content of the next batch. Towards this end, the effects of the ash accumulation in the sand matrix on smouldering was investigated over five successive tests with the same batch of sand. The smouldering conditions were kept constant at 7.5 g/g S/F, 50 % MC, and

Table 3 Ultimate analysis performed on the oil condensed from the exhaust gas during smouldering

Property	
HHV (kJ/g)	27.6
Water content (wt%)	56
Elemental analysis (dry, wt%)	
C	74.1
O	18.4
H	2.1
N	1.0
S	0.0

The ultimate analysis does not match 100 % because are average values

Table 4 Amount of volatile fatty acids (VFA) present in the aqueous phase

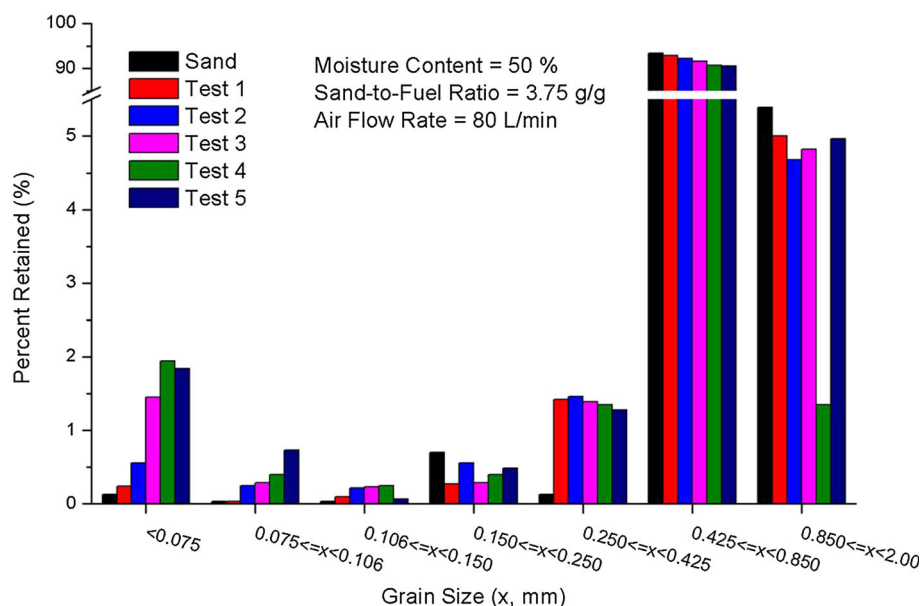| Compound	g/L
Acetic acid	19.60
Formic acid	24.71
Propionic acid	5.05
Isobutyric acid	1.26
Butyric acid	2.37
Isovaleric acid	1.07
N-valeric acid	1.54
Isocaproic acid	2.31
N-caproic acid	1.63
Heptanoic acid	0.92
Total	60.46

Fig. 9 Photograph showing the sand appearance with ash accumulation over five consecutive experiments. Reg sand corresponds to the clean dry sand before the first test

80 L/min airflow. As shown in Fig. 9, a shift in overall colour from red to white was observed as a function of the iteration, which is attributed to the build-up of inorganics in the sand.

The results of sieve analyses, for particle sizes between 0 and 2 mm, performed on the sand after each test are presented in Fig. 10. The analysis shows that approximately 90 % of particles in these tests are between 0.45 to 0.85 mm in diameter; these are the unchanged sand grains. The particle size of the ash is below 0.425 mm and the ash concentration is observed to increase after each test. The amount of increased ash in each successive test matches the amount of inorganics in the faeces. However, the overall particle size distribution of the sand does not change significantly. No impact was observed on the smouldering behaviour, as temperatures and smouldering velocities were consistent from test 1 to test 5. This demonstrates that the sand can be re-used after each test and it is expected it will not need to be changed until a very substantial build-up of fines occurs, affecting permeability.

Conclusions

The feasibility of producing bio-oil through the smouldering combustion of faeces mixed with sand was demonstrated. Additionally, the influence of the experimental parameters (original moisture content of the faeces, the sand-to-faeces mass ration and airflow rate) on the yield of bio-oil production was studied. It was shown that the original moisture content of the faeces seems not to affect the bio-oil production, while the airflow rate increase the amount of bio-oil produced. In the case of the amount of sand, the mass of bio-oil collected fluctuated within the range studied.

A method to estimate the length of the pyrolysis zone during smouldering was presented, and the extent of this zone was calculated for each experiment. In all cases, there seems to be a correlation between the extent of the pyrolysis zone calculated and the amount of bio-oil produced. Under the experimental conditions studied, up to 70 g of bio-oil per kg of faeces (HHV = 26.4 kJ/g) can be

Fig. 10 Sieve analysis of sand-ash mixture over five consecutive experiments between 0 and 2 mm. Sand grains (black bars) are mainly 0.425–0.850 mm in diameter

produced. As the results shown, this yield can be increased further by increasing the airflow rate and changing the relative amount of sand used. The necessity of energy to sustain the smouldering process—coming from exothermic combustion- makes the levels of CO_2 high, as compared to traditional pyrolysis methods, which reduces the pyrolysis products that can be obtained. While the bio-oil yield is low compared with other methods [29], the method presented here does not require external energy, or the utilization of high pressure, and thereby avoids using more complex equipment and higher initial investment. Moreover, smouldering is a robust and easy-to-operate process that can applied in a wide range of situations. The bio-oil produced by smouldering of faeces would be an additional benefit to the treatment and disinfection method, making the process even more sustainable. For example, the energy of the bio-oil produced here is enough to achieve ignition of the smouldering process, which is between 600 and 1000 kJ.

The sand used in the process is recovered clean and the only change is a very slow build-up of inorganics with minimal impact on permeability or smouldering behaviour. It was demonstrated the clean sand recovered after smouldering can be re-utilized for at least five consecutive batches.

It is acknowledged that this is a preliminary study and further investigation is required; especially because the study was based on a surrogate waste. The assessment of the quality of the pyrolysis products should be evaluated and studied for real faeces; and the implementation of this technology may require further treatment or upgrade of the oil produced. Further research should be conducted to study the implementation of this technology with other organic waste with high moisture content, such as kitchen or agricultural waste, which can shed light on the implementation of an integrated waste treatment technology. The utilization of other organic residues (e.g. lignocellulosic) could enhance the yield and quality of the bio-oil produced. The scale and robustness of this approach make this technology ideal for application in slums, rural schools or hospitals.

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