Pyrolysis for the Valorisation of Sludge and MSW Organic Fines – Reactor Characterisation and Product Distribution

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Abstract

Sludge from municipal wastewater treatment plants and organic fines from municipal solid waste (MSW) are two abundant waste streams. Wastewater sludge is commonly applied to agricultural land in the EU as a fertiliser. From an EU waste policy viewpoint, this is a desirable outlet. The screening of municipal solid waste produces a heterogeneous fine material, which is routinely stabilised through composting and destined to landfill sites. However, changing perceptions of risk in food production in society and a desire to avoid landfilling of materials have spurred interest in thermal conversion technologies that aim to recovery both energy and nutrients from waste while reducing any health and environmental risks associated with material re-use.

Pyrolysis yields three output products: solid char, liquid oils and gas. The relative distribution of each product depends heavily on process parameters which can be somewhat optimised depending on the application. The gas and char fractions can have a relatively high heating value making them attractive as potential fuels.

This study is part of a 32-month project investigating the potential of pyrolysis for the conversion of wastewater sludge (SS) and organic fine of MSW (OF) to a combustion gas and a carbon-rich char. Pyrolysis of SS and OF has been carried out using a laboratory fixed-bed reactor. Herein, the physical characterisation of the reactor is described and results on pyrolysis products yields are presented. Characterisation of feedstock and pyrolysis chars have been carried out using standard methods and pyrolysis gas composition was analysed using micro gas chromatography.

Product distribution (char/liquid/gas) from the pyrolysis of sewage sludge and composted MSW fines at 700°C for 10 min were 45/26/29 and 53/14/33 per cent respectively. The combustible fractions of pyrolysis gases ranged range from 36 to 54% for SS feedstock and 62 to 72% from OF. The corresponding lower heating value range of sampled gases were 11.8-19.1 MJ m^{-3} and 18.2-21.0 MJ m^{-3} respectively.

1. Introduction

Two abundant waste streams are sludge from municipal wastewater treatment plants (SS) and organic fines (OF) from the mechanical treatment of municipal solid waste (MSW). Wastewater sludge is the organic by-product of municipal wastewater treatment. It consists of the solids, which are removed from wastewater during the treatment process. Treatment methods can be mechanical, biological or chemical.

Sludge from wastewater treatment plants is commonly applied to agricultural land in the EU as a fertiliser. However, this is not universal as some countries, such as the Netherlands, incinerate all produced sludge. The fraction of total sewage sludge production from urban wastewater in selected Member States in depicted in Figure 1 [1]. The re-use of sludge is the most encouraged outlet, according to current EU waste policy objectives which also permit optional methods that provide the best overall environmental outcome [2].

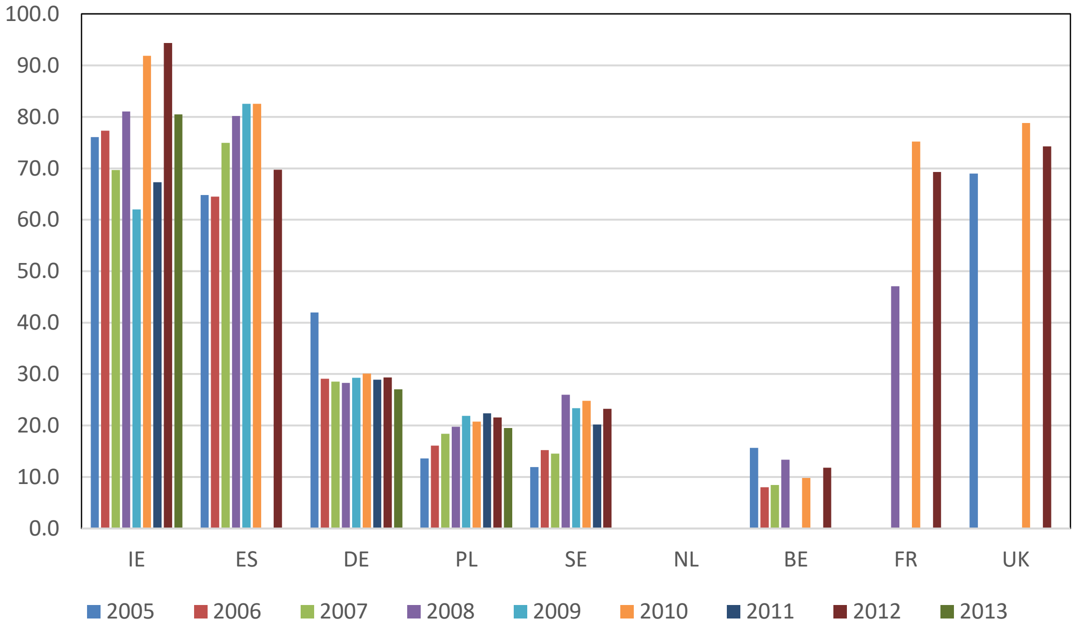

Figure 1. The re-use of sewage sludge in agriculture in select EU member states (2005-2013) expressed as fraction of total sewage sludge production from urban wastewater. Data is incomplete for ES, BE, FR and UK. Values for NL are zero [1].

Treatment of wastewater is a costly but necessary process whose real cost in Ireland is somewhere in the range of 300 € t^{-1} and upwards per dry tonne of sludge produced [3]. Therefore, when evaluating the economic feasibility of different sludge use options, this level of investment exits in the history of the resource.

The fine fraction of MSW is an extremely heterogeneous material containing food waste, plastics, metals, paper, and glass [4]. After screening of MSW, the fine material is routinely stabilised through biological treatment (composting) after which it is used as a cover material at landfill sites [5]. In Ireland, where currently only a few landfills remain in operation, there are limited outlets for this material.

Pyrolysis in of interest in waste management because it can reduce health and environmental risks from problematic wastes [6] while providing an avenue for the recovery of energy and nutrients [4]. Moreover, societal perceptions of risk and quality-assurance schemes in food production have lessened the appeal of spreading treated sewage sludge on agricultural land.

Pyrolysis is the thermal conversion technique used to decompose organic materials into other products under inert atmosphere. Wood charcoal is a familiar product produced from pyrolysis of wood. Char, however, is only one of the products of pyrolysis. The process also yields liquids (oils and tars) and gases (syngas). The relative distribution of each product depends heavily on several process parameters whose influence follows a general trend for all organic feedstock [7]. These parameters can be somewhat optimised depending on the application. The three most obvious parameters are temperature, residence time and heating rate. Particle size of the feedstock and residence time of vapour-phase products are also important as these influence the contact between chars and gases, the extent of which affects char formation and decomposition of long-chain hydrocarbons gases. In follows, that there are important differences between batch and continuous pyrolysis processes.

The range of temperature used in pyrolysis can start as low as 220°C, as in the partial pyrolysis of wood [8], to up to 900°C [9, 10]. Residence times of feedstock vary from just a few seconds up to several hours. High temperature and a long residence time generally favour gas and char production while minimising the production of oils. High rates of feedstock heating, however, produces more vapour-phase products and decrease char yields [9]. From studies on MSW pyrolysis, for example, the heating rates of the feedstock can vary from 4°C min^{-1} to 670°C min^{-1}. High gas and char yields are desirable in a process, which recovery energy by using both these products as a fuel.

A 32-month Irish national project was launched in 2016 on the feasibility of pyrolysis in waste management. The project investigates the potential of pyrolysis as a conversion technology for the distributed treatment of two abundant waste steams in Ireland. It is viewed as an alternative to incineration. Waste is converted into a value-added solid product and a combustible gas to recovery energy and/or nutrients from the feedstock. Laboratory-scale pyrolysis experience is to be used to optimise a pilot-scale process being developed with technological partners in the project.

2. Materials and methods

2.1 Feedstock materials

Two feedstock types were used in the pyrolysis experiments herein; sludge from a municipal wastewater treatment plant (SS) and composted organic fines from MSW (OF). SS samples were pre-dried and in the form of pellets, supplied by Northumbrian Water, co. Cork, Ireland. They had a moisture content of 9.8% (wb). The average pellet size was approximately 10 mm in length and 4 mm in diameter (Figure 2). OF samples were supplied by Greenstar, co. Cork, Ireland. They had a maximum particle size of about 10 mm and had a moisture content of 8.5% (wb) after air-drying. The as-received moisture content varied between 17-25% (wb). Glass, which can make up to 20% mass of the material, and metals components were removed from the feedstock.

Figure 2. Photographs of feedstock materials used in this study. Sewage sludge (SS) pellets (left) and composted organic fines (OF) from municipal solid waste (right).

2.2 Experimental apparatus

The pyrolysis tube reactor (Figure 3) consists of a cylinder (45/50 mm, I.D./O.D) of quartz (H. Baumbach & Co Ltd, UK) whose exterior was evenly wrapping with Samox® heavily insulated heating tape whose maximum power output was 940 W (Omegalux, USA). Two layers of woven high-temperature insulation, having a combined thickness of approximately 10 mm, were wrapped on the outside of the heating tape and secured with aluminium-foil tape at either end. A model MC227 electrothermal power regulator (Cole-Parmer, UK) supplied the heating tape with AC electricity. The total length of the reactor cylinder is 600 mm and that of the heated section is approximately 350 mm. The reactor was held in place using clamps attached to two or more ring stands and was inclined from the horizontal by approximately 10°.

Figure 3. The quartz tube reactor with heating tape wrapped around its exterior (before the application of insulation).

Borosilicate (Pyrex®) jointed glassware, manufactured by Quickfit®, formed the remainder of the apparatus (Figure 4). One end of the reactor tube was open and sealable with a removable rubber stopper. The other end of the cylinder tapered to a ground glass fitting to match a reducer (XA43). A 90° bend then followed, leading to a condenser (C1/13/SC) whose outer jacket was cooled via circulation of a refrigerated liquid. The condenser section was inclined from the horizontal by approximately 45°. A twin-neck round-bottom 500 ml flask as onnected below the condenser to hold pyrolysis liquids. A 10 mm (O.D.) rubber tube, approximately 50 cm in length, was connected to the other neck of the flask above the liquid level. The other end of this tube was open to atmosphere and fitted with a plastic connector, suitable for attaching gas-sampling bags.

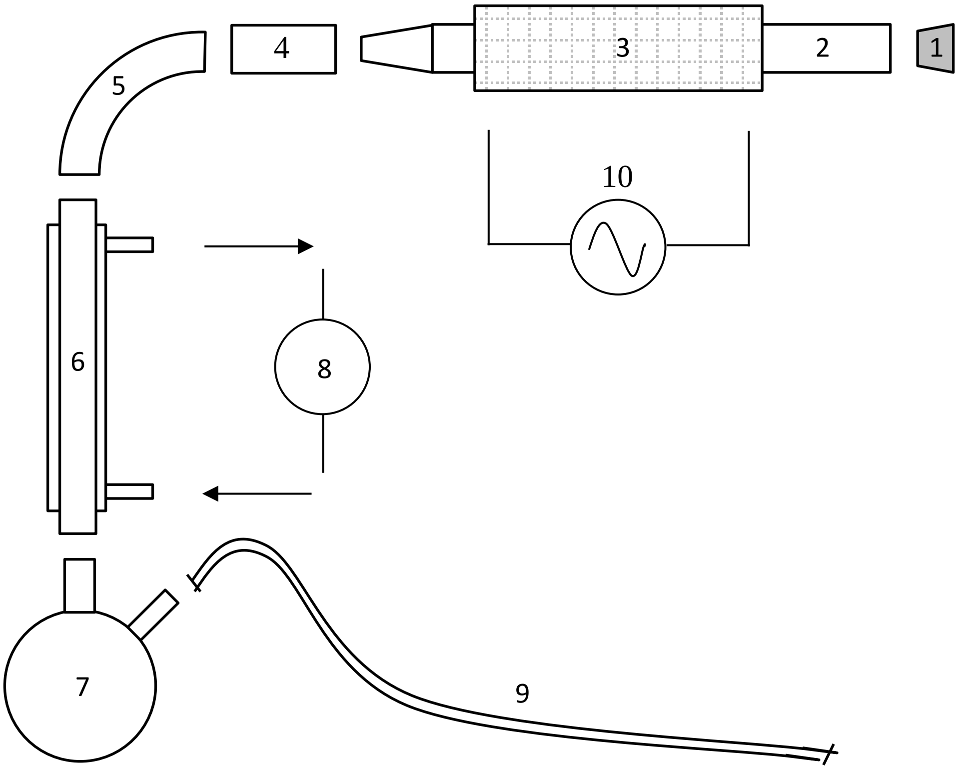

Figure 4. Exploded view of laboratory pyrolysis apparatus (not to scale) – 1. Rubber stopper 2. Quartz reactor tube 3. Heated and insulated section of reactor tube 4. Reducer 5. 90 degree bend 6. Condenser 7. Twin-neck round-bottom 500 ml flask 8. Refrigerated liquid controller 9. Exit tube for gas sampling 10. Power regulator (AC).

Temperature was measured using a sheathed K-type thermocouple of 1000 mm length which was inserted in the reactor via a feed-through hole in the rubber stopper. The hold was sealed on the outer side of the stopper using a small amount of Blu Tack®. Temperature was read within 1°C using a digital handheld display.

Feedstock samples were loaded into a purpose-made cylindrical steel-mesh basket having a length of about 250 mm and slightly smaller diameter than that of the inside of the reactor. The mesh size was about 0.25 mm.

2.3 Experimental procedure

The relative distribution of pyrolysis projects was determined by the principle of conservation of mass – the combined mass of pyrolysis products is equal to that of the initial sample feedstock. The primary assumption is that no liquids exit the apparatus in the vapour phase; all condensable products are present.

Before each pyrolysis run, the reactor, connected glassware, the empty sample basket and rubber stopper were weighed to within ± 0.01 g. Fine particles (< 380 µm) were screened from the sample beforehand. A sample of feedstock was poured into the basket and the basket was weighed again.

The reactor and glassware were assembled in a fume hood and the heating tape leads were connected to the power regulator and switched on. Ground glass connections were sealed with a few wraps of Teflon® tape. The condenser coolant tubes were connected and circulation of the refrigerated liquid (-5°C) commenced. After approximately 50 minutes the reactor is ready for use.

The thermocouple was inserted through rubber stopper to a suitable length that its tip was located in the middle of the heated section of the reactor tube. The sample basket and thermocouple together were inserted into the open end of the reactor in one smooth motion ending with the stopper being in place. The timer was started and the fume hood closed. After 10 or 15 minutes, the heating was switched off. After 30 minutes, the cooling was switched off. The reactor tube cooled to room temperature after about two hours. The sample basket and contained char were removed from the reactor and weighed. The reactor, glassware (containing condensed and liquid pyrolysis products), the empty basket and rubber stopper were weighed again.

The initial sample mass was calculated as the difference between the empty and loaded basket before pyrolysis. The mass of char was calculated as the difference between the empty and loaded basket after pyrolysis. The liquid mass was the difference of mass of the apparatus (reactor and all components) before and after pyrolysis. The mass fraction of gas was calculated as the difference between 100% and the char and liquid mass. Pyrolysis runs were carried using 20 and 50 g samples at 600 and 700°C.

2.4 Physical characterisation of the reactor

In order to compare research results achieved with other apparatus, there are a number of physical quantities used to characterise a pyrolysis reactor. These describe heat transfer and operational parameters of the pyrolysis process.

The heating rate χ *($K\ s^{-1}$)* of a pyrolysis reactor is equal to the effective heating power *P ($J\ s^{-1}$)* divided by the heat capacity *C ($J\ K^{-1}$)* of the feedstock:

(1) $$\chi = \frac{P}{C}$$

P is the product of the heat flux *q ($W\ m^{-2}$)* and heat transfer surface area *A (m^2)* of the reactor wall (Equation 2). The heat flux is the product of the temperature difference *ΔT (K)* between the wall of the reactor and the feedstock and the heat transfer coefficient *α ($W\ m^{-2}\ K^{-1}$)* within the reactor (Equation 3). *C* in Equation 1 is the product of feedstock mass *m (kg)* and its specific heat capacity *C_p ($J\ kg^{-1}\ K^{-1}$)*.

(2) $$P = q \cdot A$$

(3) $$q = \propto \cdot \Delta T$$

The heat transfer coefficient is found by combining the above equations and solving for α, which yields Equation 4.

(4) $$\alpha = \frac{\chi \cdot m \cdot C_p}{A \cdot \Delta T}$$

With the possible exception of *A,* the factors in Equation 4 are strictly a function of time. The heating rate χ and *ΔT* are easily determined experimentally. χ is equivalent to the slope of the temperature versus time profile of the heated feedstock. By measuring the reactor wall temperature simultaneously, *ΔT* is determined.

The dimensionless *Biot* number *Bi,* is determined within the reactor by use of Equation 5, in which *r_p (m)* is the feedstock particle diameter (assumed to be spherical) and *λ ($W\ m^{-1}\ K^{-1}$)* is the thermal conductivity of the feedstock.

(5) $$Bi = \frac{\alpha \cdot r_p}{\lambda}$$

The Biot number is the ratio between the rate of heat convection (numerator) and conduction (denomination). A *Bi* value appreciably smaller than one indicates that heat transfer within the feedstock is rapid enough and resulting char is evenly cooked across the particle and that thermal control of the reactor is achieved.

2.5 Temperature profile, residence time and assumptions

The temperature profile within the reactor was measured as a function of distance from the internal wall of the quartz tube reactor. This varied from directly adjacent to the wall (outside the sample basket) to completely within the sample. To determine the great temperature difference between the reactor wall and the sample, an average temperature profile from several different number (Figure 5) of runs was calculated.

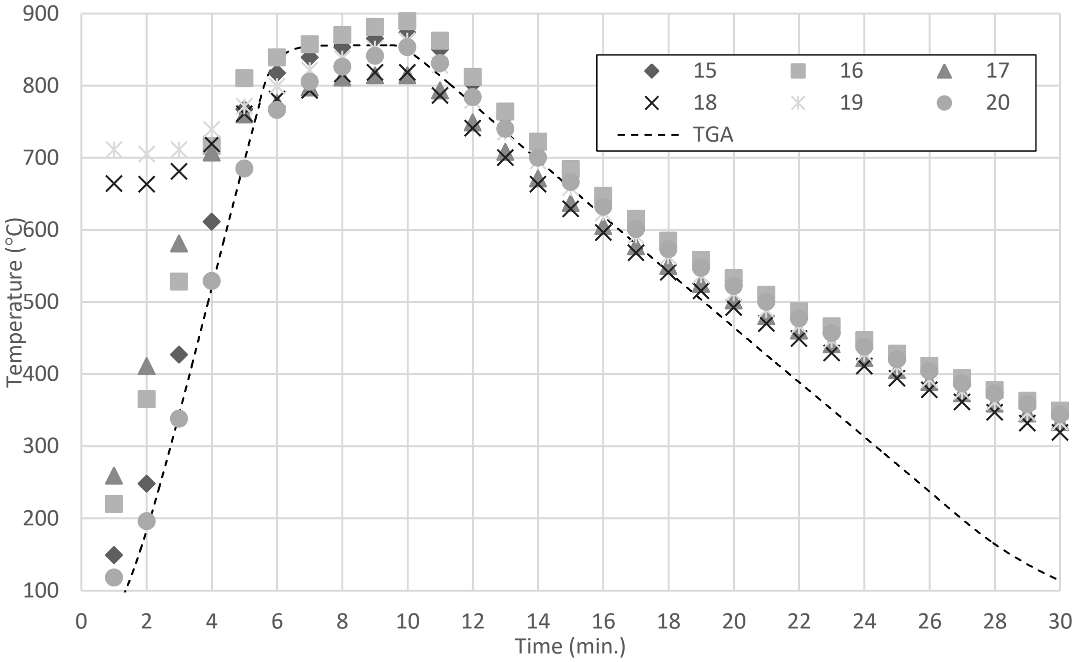

Figure 5. Measured temperature of 20 g of SS feedstock (15, 16, 17, 20) and reactor wall (18, 19) during six different pyrolysis runs (20 g at 700°C). The TGA simulated heating profile used for mass-loss determination (dashed line).

The two most extreme temperature profiles, that of the reactor wall and that within the bulk of the sample, were used to calculate ΔT and the resulting heat transfer coefficient. This value is interpreted as a minimum value because α is inversely proportional to ΔT (Equation 4). The Biot number was then calculated.

The residence time of a sample was determined through averaging the temperature data from several runs, as in Figure 5. For a 20 g sample, the residence time was 9.8 min at 700°C and 6.2 min at 800°C. A 50 g sample had a residence time 8.2 min at 700°C and 4.1 min at 800°C.

The change of mass of the feedstock during pyrolysis (Equation 4) was estimated using a mass-loss curve from analysis using a TGA (PerkinElmer Pyris). The heating profile of the feedstock in reactor was simulated in the TGA (Figure 5) with a sample mass of 7.5 mg, initial heating rate of 150 K min^{-1} and nitrogen flow of 20 ml min^{-1}. A value of 1950 J kg^{-1} K^{-1} was used for the heat capacity of dried sewage sludge [11]. For the thermal conductivity of the sludge, a value of 0.1 W m^{-1} K^{-1} was used.

SS feedstock was used to characterise the pyrolysis reactor because of its homogeneity in composition. Temperature profiles using OF showed greater variation between individual runs.

2.6 Characterisation of feedstock and pyrolysis char

Proximate analysis of feedstock and pyrolysis chars were carried out according to standard methods for heating value (EN 15400), ash content (EN 14775), moisture content (EN 15414) and volatile content (EN 15402). Ultimate analysis of feedstock and char was carried out by Celignis Analytical, Ireland. The selected chars were from SS (50g, 700°C) and OF (50g, 700°C).

2.7 Pyrolysis gas analysis

Pyrolysis gas sampling was done using Tedlar ® 0.5 litre bags with a polypropylene valve (Restek, Ireland). The bags were filled via the exit tube of the reactor (Figure 4). Gas samples were taken at regular intervals during the batch pyrolysis run with each sampling lasting from 15-30 s depending on the filling rate. Samples were analysed using a micro gas chromatograph (Agilent 3000).

The reactor tube contained air at the start of a run. To determine the extend of carbon oxidation in the sample due this amount of oxygen, the required stoichiometric volume (ideal gas) of air required for combustion $(C + O_2 \rightarrow CO_2)$ of the feedstock carbon was calculated. At 600°C, the required volume of air was 152 litres while the volume of the reactor tube is one litre. Therefore, an inert atmosphere can be assumed.

The lower heating value *LHV (MJ* m^{-3}*)* of the pyrolysis gas was calculated using the volumetric fraction f_x *(dimensionless)* of the combustible gas component *x* and the lower heating value of that component LHV_x *(MJ* m^{-3}*)* as in Equation 6 [12].

(6) $$LHV = \sum f_x \cdot LHV_x$$

$$LHV = f_{CH4} \cdot 35.883 + f_{CO} \cdot 12.633 + f_{H2} \cdot 10.783 + f_{C2H4} \cdot 59.457 + f_{C2H6} \cdot 63.79$$

3. Results and Discussion

3.1 Heat transfer within the reactor

The temperature of the reactor wall and feedstock, whose difference represents ΔT, is depicted in Figure 6 along with the calculated Biot number. The heat transfer coefficient α over this time period ranged from 2.6 to 18.1 W m^{-2} K^{-1} for SS feedstock. The heating rate ranged from 160 to 5 K min^{-1}. The undulating appearance of the Bi curve in Figure 6 is a result the difficulty of evaluating the heating rate χ (dT/dt) from the temperature curve. The important point is that *Bi* has a value well below unity over the run, even for feedstock having a 4 mm particle diameter (SS). For smaller particle sizes, *Bi* decreases accordingly (Equation 5). This indicates that heat transfer within the sample is rapid enough for sufficient thermal control of the process.

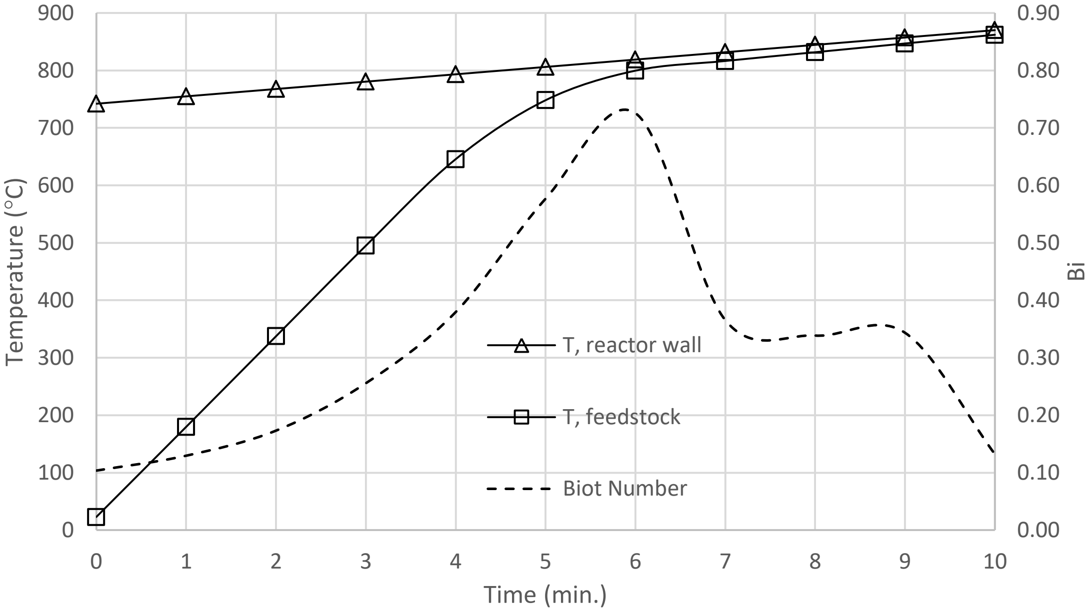

Figure 6. Modelled temperature profile of reactor wall and feedstock with calculated Biot number for 4 mm particle diameter.

3.2 Mass balance

The distribution of pyrolysis products for four series of runs is shown in Figure 7. Each series was an average of five separate runs whose standard deviation is listed in Table 1. Focusing first on the differences between the two feedstock, the char yield for SS ranged from 45 to 48%, while that of OF was 52%. The liquid yield of SS runs had an average of 25-32% while that of OF was 14%. Correspondingly, the gas yield from OF was the highest observed being 33% while gas fractions from SS had a range of 19 to 29%.

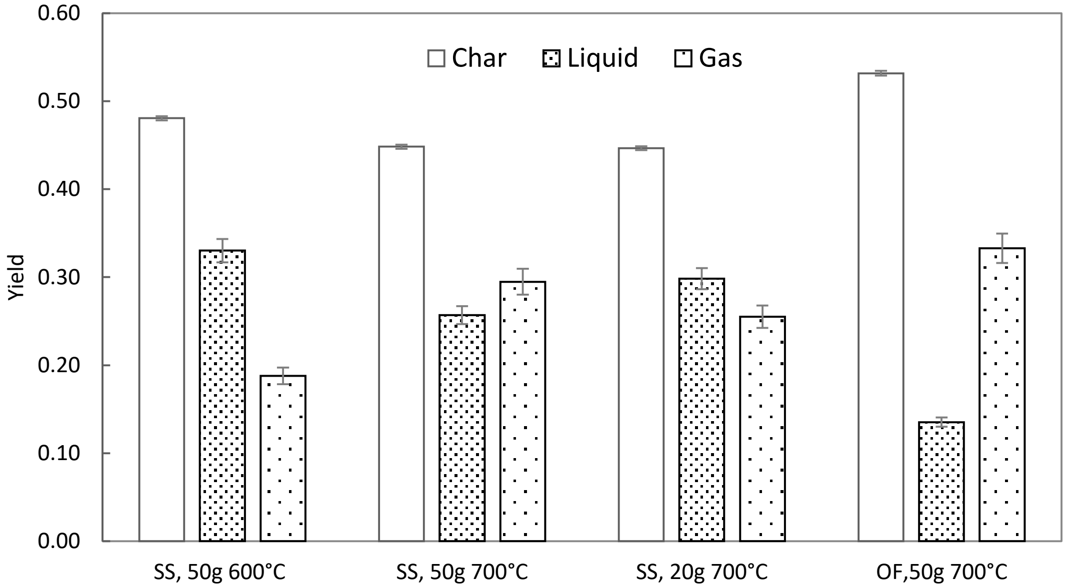

Figure 7. Yields of Laboratory-Scale Pyrolysis with experimental uncertainties

Error bars represent experimental uncertainty in mass determination. The experimental uncertainty in char yield was low (0.5%) because the sample and char was confined to the sample basket and their mass was directly measured on the laboratory balance. Moreover, screening of the sample ensured that fine particles did not escape the basket.

Liquid and gas yield amounts had inherently greater uncertainty. For example, a loss or a gain of liquid while disassembling the apparatus or through condensation of water on the condenser and seepage into collection flask, respectively was observed. Furthermore, smaller absolute yields, with constant balance uncertainty, increased errors in measurement.

Additionally, gas sampling bags often contained condensed tars. This indicated that some condensable pyrolysis products exited the apparatus as a vapour but this amount was not quantified. The error bars in Figure 7 do not include this error. Consequently, based on the conservation of mass, the calculated liquid fraction in the results is interpreted as the minimum liquid fraction while the calculated gas fraction is interpreted as a maximum value.

Table 1. Mass yields of pyrolysis products including standard deviation (SD).

Run Series	Char	SD	Liquid	SD	Gas	SD
SS, 50g 600°C	0.48	0.00	0.33	0.08	0.19	0.07
SS, 50g 700°C	0.45	0.00	0.26	0.04	0.29	0.04
SS, 20g 700°C	0.45	0.00	0.30	0.08	0.26	0.07
OF,50g 700°C	0.53	0.02	0.14	0.02	0.33	0.03

Standard deviation (SD) of char yields from SS was below 0.5%, which reflects the homogeneous composition of the feedstock. SD for OF runs, however, was almost 4%. The particle size of the SS samples did not affect yields as runs, using ground SS pellets (20 g 700°C) with 1 mm screen size, resulted in the same distribution of product yields (within SD).

3.3 Proximate and ultimate analysis of feedstock and char

Table 2. Proximate analysis of feedstock and pyrolysis char (dry basis)

Sample	HHV (MJ kg^{-1})	Volatiles (%)	Ash (%)	Fixed C (%) *
SS	16.9	52.8	32.1	15.1
OF	15.6	42.2	41.5	16.4
SS char	11.7	3.2	69.4	27.4
OF char	12.2	-	64.2	-

Table 3. Ultimate analysis of feedstock and pyrolysis char (dry basis)

Sample	C (%)	H (%)	N (%)	S (%)	O (%) *
SS	37.28	5.50	5.67	1.01	17.94
OF	37.48	4.13	2.48	1.44	16.12
SS char	28.68	0.09	1.94	0.41	-0.41
OF char	34.96	0.42	1.13	2.41	-3.14

3.4 Pyrolysis gases

Results of gas analysis from SS and OF feedstock are presented in Figure 9 and 10, respectively.

Five samples, one every two minutes, were taken for SS pyrolysis. For SS pyrolysis, the combustible gas fractions, whose combined volume fraction range from 36 to 54% of observed gases, were predominantly carbon monoxide (CO), methane (CH_4), ethylene (C_2H_4) and ethane (C_2H_6). The LHV of the gas (Equation 6) ranged from 11.8 to 19.1 MJ m^{-3}. The fraction of carbon dioxide (CO_2) declined from an initial 2% and CO ranged from 14 to 22%, which indicates that carbon oxidation of the sample was insignificant.

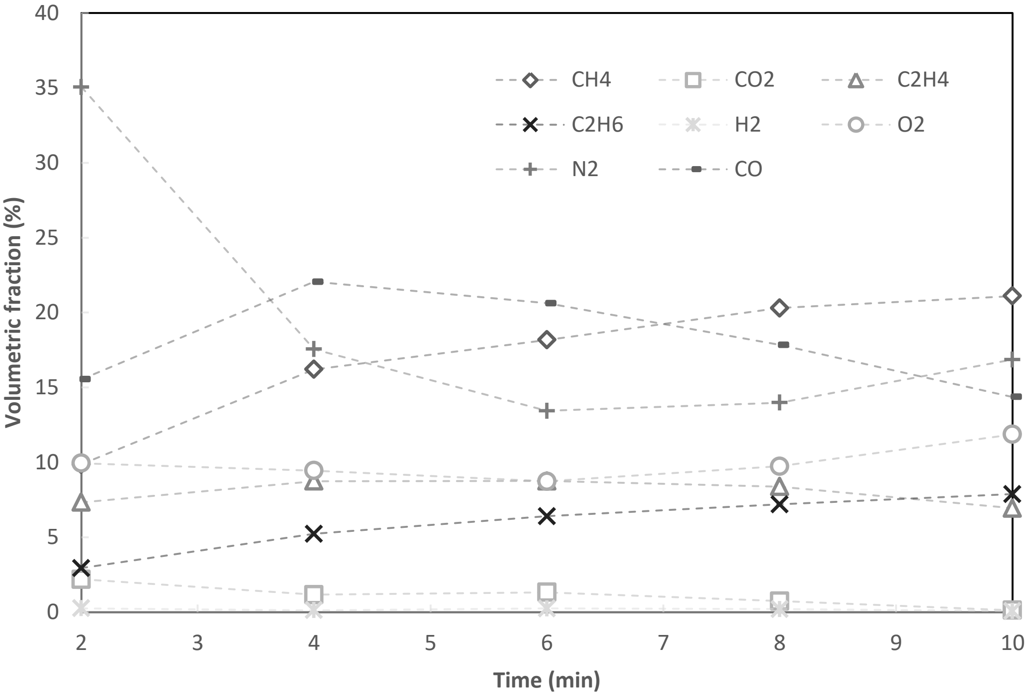

Figure 9. Volumetric fraction of pyrolysis gases from 50 g of SS at 600°C.

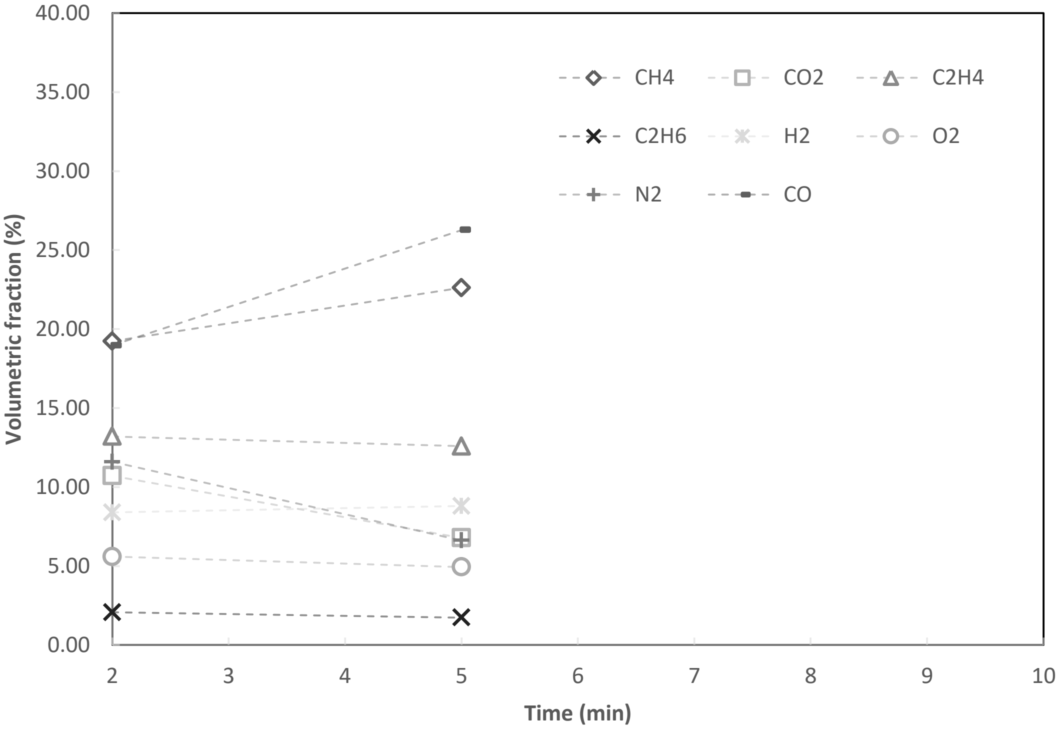

Figure 10. Volumetric fraction of pyrolysis gases from 50g of OF at 700°C.

For OF pyrolysis, the combustible gas fractions ranged from 62 to 72% of observed gases and were CO, CH_4, C_2H_4 and hydrogen (H_2). The calculated LHV ranged from 18.2 to 21.0 MJ m^{-3}.

The fraction of carbon dioxide was some 7 to 11%, significantly higher than in SS pyrolysis, whereas the oxygen and nitrogen fraction were markedly lower.

Observations on pyrolysis gas composition are qualitative because the absolute amounts of each gas species, produced from the feedstock cannot be determined without information on gas-flow rates from the reactor during the pyrolysis run.

4. Conclusions

Physical characterisation of a laboratory reactor gives detailed information on pyrolysis process parameters which are essential in interpreting and comparing experimental results. Heat transfer conditions can be described using the sample heating rate, heat transfer coefficient and Biot number.

Pyrolysis yields from wastewater sewage sludge and organic fines of municipal solid waste were determined according to the principle of conservation of mass. Initial results on chars from these feedstock and pyrolysis gas yields show potential for use as fuels.

Nomenclature

MSW	Municipal solid waste
OF	Organic fines
SS	Sludge from wastewater treatment plant
χ	Heating rate of the feedstock $(K\ s^{-1})$
P	Effective heating power $(J\ s^{-1})$
C	Heat capacity $(J\ K^{-1})$
q	Heat flux $(W\ m^{-2})$
A	Heat transfer surface area (m^{2})
ΔT	Temperature difference between the wall of the reactor and the feedstock (K)
α	Heat transfer coefficient within the reactor $(W\ m^{-2}\ K^{-1})$
m	Feedstock mass (kg)
C_p	Specific heat capacity $(J\ kg^{-1}\ K^{-1})$
Q	Energy required for pyrolysis (J)
t_R	Batch run time (s)
Bi	*Biot* number
r_p	Particle diameter of the feedstock (m)
λ	Thermal conductivity $(W\ m^{-1}\ K^{-1})$
LHV	Lower heating value of pyrolysis gas $(MJ\ m^{-3})$
LHV_x	Lower heating value of pyrolysis gas x $(MJ\ m^{-3})$
f_x	Volumetric fraction of pyrolysis gas x

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