Process energetics for the hydrothermal carbonisation of human faecal wastes

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Abstract

Hydrothermal carbonisation (HTC) has the capability to convert wet biomass such as sewage sludge to a lignite-like renewable solid fuel of high calorific value. However, to date, assessment of the energy efficiency of the HTC process has not been fully investigated. In this work, mass and energy balances of semi-continuous HTC of faecal waste conducted at 200°C and at a reaction time of 30 min are presented. This analysis is based on recovering steam from the process as well energy from the solid fuel (hydrochar) and methane from digestion of the liquid product. The effect of the feedstock solids content and the quantity of feed on the mass and energy balance were investigated. The heat of reaction was measured at 200°C for 4 h with the wet faecal sludge, and the higher heating value was determined for the hydrochar. The results indicated that preheating the feed to 100°C using heat recovered from the process would significantly reduce the energy input to the reactor by about 59%, and decreased the heat loss from the reactor by between 50–60%. For feedstocks containing 15–25% solids (for all feed rates), after the process is in operation, energy recycled from the flashing off of steam and combustion of the hydrochar and would be sufficient for preheating the feed, operating the reactor and drying the wet hydrochar without the need for any external
sources of energy. Alternatively, for a feedstock containing 25% solids for all feed rates, energy recycled from the flashing off of steam and combustion of the methane provides sufficient energy to operate the entire process with an excess energy of about 19–21% which could be used for other purposes.

**Keywords:** Bio-energy; Biomass; Hydrochar; Renewable energy; Sewage sludge

1. Introduction

It is reported that about 1 billion tons of human faeces are generated each year [1], a figure which will increase in line with the projected growth in population. It is estimated that globally over 90% of sewage is discharged untreated [2] and that faecal contamination of water sources causes almost 4 billion cases of diarrhoea each year, killing nearly 2.2 million children under the age of five [3]. Even in those parts of the world where faecal waste routinely undergoes treatment management of sewage sludge continues to present environmental and health challenges. Management of faecal wastes, therefore, remains a critical problem requiring proper mitigation techniques.

Hydrothermal carbonisation (HTC) is an effective method for converting wet biomass at relatively mild reaction temperatures into a coal-like material commonly referred to as ‘hydrochar’ along with aqueous products and gases - primarily CO₂ [4-6]. HTC has been shown to produce substantial energy yields from various types of waste biomass of agricultural origin [6-8]. However, attention has recently turned towards the HTC of sewage sludges for energy generation [9-12]. One added benefit of this is that the process temperatures typically employed in HTC result in the destruction of any pathogens present in the sludge [13]. Most of the investigations cited above have concentrated on producing renewable solid fuels and analysing the combustion properties of such fuels [8-10,14,15]. In
addition, some of these studies have focused on evaluating the energy efficiency of the production of solid fuel by considering mechanical dewatering, drying and biofuel recovery ratios [11,12]. Furthermore, the prospects for recovering energy from the liquid by-products and the gas/vapour phase have also received considerable attention. The energy input of HTC could be improved by combining HTC with anaerobic digestion of water-soluble by-products [16-18] or their (and that of combustible gases and certain fractions of the hydrochar) wet air oxidation [19-22]. The latter has been reported to improve subsequent anaerobic biodegradability of the liquid products for enhance biogas yields.

HTC of biomass is widely reported [4,5,23] to be an exothermic process. In early reports [23,24], it was stated that about 20–30% of the energy stored in the biomass is released as heat during the HTC process, whilst between 60–90% of the heating value of the feedstock remains in the hydrochar [24]. Heating values of hydrochars following HTC of sewage and wastewater sludge range between 15–29 MJ kg$^{-1}$ [9-12,25] i.e. similar to that of lignite or sub-bituminous coal [26,27]. In order to maximise energy yield after the process is in operation, it is essential that the energy released during the process be recovered and utilised. The hydrochar can be directly combusted to provide additional energy, or as recommended in some studies, be blended with coal to improve the devolatilisation and ignition properties of coal [14,15]. Erlach and Tsatsaronis [28] reported that flashing off steam from hot slurry remaining in the reactor following treatment can improve the energy efficiency of the HTC process. Stemann and Ziegler [29] proposed recovering heat from the hot compressed water following the mechanical dewatering of the hydrochar to further improve the energy efficiency. However, the amount of energy recovered from the process by such strategies may not be sufficient to sustain the process, as energy is required to heat the faecal waste, which typically contains about 90% water, to the reaction temperature, and also to dry the hydrochar. Moreover, heat may be lost due to release of pressure at the end of the process.
(particularly in batch HTC plants) and mixing of the material in the reactor following treatment with the incoming cold feed (for semi-continuous and continuous plants). Heat losses from the HTC reactor will occur as a result of radiation and convection but there is no consensus as to their impact. Thorsness [30] reported significant heat losses during HTC of municipal solid waste, whereas Namioka et al. [31] claimed that only insignificant heat losses occurred during the HTC of sewage sludge. These conflicting claims emphasise the need for a thorough investigation of the energetics of HTC processes.

The work presented here provides a framework for estimating energy utilisation, losses and recovery within the HTC process for faecal waste treatment. Furthermore, consideration is given to optimisation of the feed rate and the solids content in the faeces to determine the best scale of operation for sustainability.

2. Materials and Methods

2.1. Materials

Primary sewage sludge (faecal sludge), was collected from Wanlip Sewage Treatment Works (Leicestershire, UK). The faecal sludge contained 4.3% (wt.) solids as received. The physical and chemical characteristics of the faecal sludge feedstock are shown in Table 1.

2.2. Experimental procedures

Triplicate batch HTC experiments were conducted using a 250 mL stainless steel reactor (BS1506-845B, BTL Ltd, England, UK) immersed in an oil bath (B7 Phoenix II, Thermo Scientific, UK) containing “THERMINOL ® 66” heating oil. HTC of primary sewage sludge containing about 5% solids were carried out at 200°C for 30 min. The time taken for the reactor to reach the reaction temperature was about 15 min. Further details of carbonisation experiments are described in a previous work [16].
Table 1
Proximate and ultimate analysis of feedstock and hydrochar generated

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Basis</th>
<th>Feedstock (dried)</th>
<th>Hydrochar</th>
<th>Liquid product</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Proximate analysis a</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Moisture</td>
<td>%</td>
<td>8.17 ± 0.25</td>
<td>4.58 ± 0.21</td>
<td></td>
</tr>
<tr>
<td>Ash</td>
<td>% db</td>
<td>27.54 ± 0.63</td>
<td>36.29 ± 0.30</td>
<td></td>
</tr>
<tr>
<td>Volatile Matter</td>
<td>% db</td>
<td>68.56 ± 0.83</td>
<td>62.30 ± 0.86</td>
<td></td>
</tr>
<tr>
<td>Fixed Carbon b</td>
<td>% db</td>
<td>3.90 ± 0.05</td>
<td>1.42 ± 0.56</td>
<td></td>
</tr>
<tr>
<td>HHV</td>
<td>MJ kg⁻¹(db)</td>
<td>17.79 ± 0.09</td>
<td>18.49 ± 0.56</td>
<td></td>
</tr>
<tr>
<td><strong>Ultimate analysis c</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>C</td>
<td>%</td>
<td>37.63 ± 1.60</td>
<td>37.85 ± 0.25</td>
<td></td>
</tr>
<tr>
<td>H</td>
<td>%</td>
<td>5.79 ± 0.26</td>
<td>5.39 ± 0.15</td>
<td></td>
</tr>
<tr>
<td>O d</td>
<td>%</td>
<td>51.30 ± 1.69</td>
<td>53.72 ± 0.18</td>
<td></td>
</tr>
<tr>
<td>N</td>
<td>%</td>
<td>5.29 ± 0.17</td>
<td>3.04 ± 0.06</td>
<td></td>
</tr>
<tr>
<td>Hydrochar yield</td>
<td>%</td>
<td></td>
<td>66.83 ± 1.00</td>
<td></td>
</tr>
<tr>
<td>Carbon recovery e</td>
<td>%</td>
<td></td>
<td>57.05 ± 0.37</td>
<td></td>
</tr>
<tr>
<td>Fixed carbon recovery f</td>
<td>%</td>
<td></td>
<td>24.65 ± 9.81</td>
<td></td>
</tr>
<tr>
<td>Energy recovery g</td>
<td>%</td>
<td></td>
<td>70.64 ± 2.25</td>
<td></td>
</tr>
<tr>
<td>COD</td>
<td>g L⁻¹</td>
<td></td>
<td>21.30 ± 2.52</td>
<td></td>
</tr>
<tr>
<td>a ASTM D7582-10.</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>b 100 – (Moisture + ash + volatile matter).</td>
<td>ASTM D5373-08.</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>d Calculated as difference between 100 and total C/H/N.</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>e (%C in hydrochar * hydrochar mass / %C in feedstock * dry feedstock mass) * 100 [16].</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>f (%fixed carbon in hydrochar * hydrochar mass / %fixed carbon in feedstock * dry feedstock mass) * 100 [32].</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>g HHV of hydrochar * hydrochar mass / HHV of feedstock * dry feedstock mass) * 100 [32].</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

108 2.2. Experimental procedures

109 Triplicate batch HTC experiments were conducted using a 250 mL stainless steel reactor (BS1506-845B, BTL Ltd, England, UK) immersed in an oil bath (B7 Phoenix II, Thermo Scientific, UK) containing “THERMINOL ® 66” heating oil. HTC of primary sewage sludge containing about 5% solids were carried out at 200°C for 30 min. The time taken for the reactor to reach the reaction temperature was about 15 min. Further details of carbonisation experiments are described in a previous work [16].

116 2.2.1. Feedstock and product analysis

117 Proximate and ultimate analyses were conducted to characterise both the feedstock and hydrochar. Residual moisture, ash and volatile matter were analysed using a
thermogravimetric analyser (TA Instruments Q5000IR, Elstree, UK), according to ASTM method D7582-10 [33]. Carbon (C), hydrogen (H) and nitrogen (N) contents were analysed using a CHN Analyser (CE-440 Elemental Analyser, Exeter Analytical Inc., Coventry, UK), according to ASTM D5373-08 [34]. Energy content of the PSS feedstock and hydrochars was determined using a bomb calorimeter (CAL2K, Digital Data Systems, Randburg, South Africa). The specific heat capacity of dry sewage sludge was determined theoretically by the method of Namioka et al. [31] and that of the hydrochar was the value used by Stemann and Ziegler [29]. Chemical oxygen demand (COD) in the liquid product was measured using a COD analyser (Palintest 8000, Palintest Ltd, UK) at a wavelength of 570 nm, in accordance with Standard Methods 5220 D – Closed Reflux Colorimetric Method [35]. All determinations were conducted in triplicate.

2.2.2. Heat of reaction measurement

The heat of reaction was measured in triplicate using a heat flux differential scanning calorimeter (DSC-Q10, TA Instruments, Crawley, UK) at 200°C for a reaction time of 4 h, being the reported time for a complete reaction [36]. Approximately 8 mg of faecal sludge (4.3% solids content) was heated in stainless steel high pressure capsules (TA Instruments, USA). Empty sealed pans were use as the reference capsules. The experiments and evaluation of the results followed ISO11357-5:1999 and ISO11357-1:2009 using the isothermal method [37,38]. For comparison, additional measurements were conducted at reaction temperatures of 160 and 180°C for 4 h. Before the heat of reaction measurements, the DSC was calibrated using 10 mg of standard grade indium metal (LGC, Teddington, Middlesex, UK). The nitrogen purge gas flow was set at 0.5 ml/min. The cooler temperature was held at 40°C by DSC re-generated cooling system (TA Instruments).

As described by ISO 11357-1:2009, the sample and reference capsules were reweighed after each run to determine if there were changes in mass that could have disturbed the
instrument baseline or created additional thermal effects [38]. The heat of reaction during the isothermal stage was estimated by integrating the area between the peak and the baseline. The results were evaluated using the method proposed by Funke and Ziegler [39], by defining the interval of integration in order to reduce uncertainties in determining the virtual baseline.

2.3. Modelling

The model developed here was based on a semi-continuous HTC plant comprising eight components; a feed tank (and pre-heater), a reactor, a flash vessel, a pressure filter, a solids dryer, an anaerobic digester (AD), and two combustion units. The plant capacity was varied between 4.00–400.00 kg/day of wet faecal waste, representing faeces generated by between 10 and 1000 people per day. The model assumed solids content in the faecal waste to vary between 5%, 15% and 25%. The faecal waste was heated to 200°C for 30 min (via a heating unit) for sterilisation. The treatment time used here was selected on the basis of results from previous study [16], which indicated that HTC at 200°C for 30 min produced hydrochars having optimal characteristics; with the HHV of the hydrochar at those conditions similar to the HHV of sub-bituminous coal. Although batch or continuous systems are typically applied in a HTC plant, continuous or semi-continuous systems promote efficient utilisation of the heat of reactions well as effective application of adjacent equipment, and also pressure changes in the reactor are prevented [29].

2.3.1 Heat recovery routes

The two processes that require energy inputs are: (1) heating of the faecal waste to the reaction temperature of 200°C; (2) drying of the wet hydrochar to less than 5% moisture before combustion to generate energy to power the plant. A previous study on filterability of slurry following HTC showed that for slurry from carbonisation conducted at 200°C for 30 min, hot-filtering the slurry at about 100°C resulted in hydrochars having water contents of
about 50% [40]; such hydrochars would require drying before combustion. No additional or external source of energy was required for dewatering the hydrochar.

After the HTC process has been initiated, energy can be recovered from three mechanisms: (1) steam from the flash tank; (2) combustion of the hydrochar; (3) combustion of biogas produced from anaerobic digestion (AD) of the liquid product. Estimated methane yields from previous work showed that a yield of 52% was attainable from such liquid products following HTC at 200°C for 30 min [16]. The overall energy recycled will be used to preheat the feedstock, dry the hydrochar, and heat the reactor.

2.3.2 Mass and energy balances

The total mass balance was evaluated for the operations taking place within the boundaries shown in Figure 1 as follows:

\[
F = m_t + m_{L,D} + m_{w,D} + m_{V,F}
\]  

(1)

where \(F\) is the mass of wet faecal feedstock (kg), \(m_t\) is mass of dried hydrochar obtained at reaction time \(t\) (kg), \(m_{L,D}\) is the mass of liquid after filtration of the carbonised slurry (kg), \(m_{w,D}\) is the mass of water evaporated from the wet hydrochar during drying (kg), and \(m_{V,F}\) is the mass of steam or water vapour recovered from the flash vessel (kg).

![Figure 1 – Basis of mass balance calculations.](image-url)
The energy balance was modelled as follows:

\[
\text{Energy input} = \text{Energy to heat reactor} + \text{Energy to heat faecal sludge} + \text{Energy to heat water} + \text{Heat of reaction} - \text{Heat loss}
\]  

(2)

That is, energy balance before energy is recovered from the process is

\[
V_0 \rho_o c_{p,o} (T - T_0) + (H_U \times \tau_h) = [m_r c_{p,r} (T - T_0)] + [m_{FS} c_{p,FS} (T - T_0)]
\]

\[
+ [m_w (H_{L(T)} - H_{w(T_0)})] + m_0 \Delta H_R - [A_r U_r t_h (T - T_0)]
\]

(3)

where: \(c_{p,o}\) is the specific heat capacity of heating oil (kJ kg\(^{-1}\) K\(^{-1}\)); \(c_{p,FS}\) is the specific heat capacity of dry sewage sludge (kJ kg K\(^{-1}\)), used to represent that of faecal sludge; \(c_{p,r}\) is the specific heat capacity of the reactor (kJ kg K\(^{-1}\)); \(\rho_o\) is the density of heating oil (kg m\(^{-3}\)); \(m_{oil}\) is the mass of heating oil (kg); \(m_r\) is the reactor mass (i.e. density of reactor material x reactor volume, kg); \(m_{FS} = m_0\), is the mass of solids in the wet sludge as (kg); \(m_w\) is the mass of water in the sludge (kg); \(H_{w(T_0)}\) is the enthalpy of water at initial temperature (kJ kg\(^{-1}\)); \(H_{L(T)}\) is the enthalpy of water at the saturated liquid temperature, \(T\) (kJ kg\(^{-1}\)); \(H_U\) is heating unit utility (kW); \(\tau_h\) is oil holding time (min); \(\Delta H_R\) is the heat of reaction during holding period (kJ kg\(^{-1}\)); \(U_r\) is the overall reactor heat transfer coefficient (W m\(^{-2}\) K\(^{-1}\)); \(A_r\) is the reactor heat transfer area (m\(^2\)) and \(t_h\) is reaction time – including heat up time (45 min).

The reactor heat transfer area is given by

\[
A_r = \pi L (D + t_I) + 2\pi r^2
\]

(4)

where: \(D\) is reactor diameter (m); \(L\) is reactor height (m); \(r\) is the reactor radius (m); and \(t_I\) is the insulation thickness (m).

Ignoring fouling factors, the overall heat transfer coefficient of the reactor is:

\[
\frac{1}{U_r} = \frac{1}{h_m} + \frac{1}{h_l} + \frac{1}{h_A + h_R}
\]

(5)

where: \(h_A\) is the heat transfer coefficient at the reactor wall (W m\(^{-2}\) k\(^{-1}\)); \(h_l\) is the conduction coefficient of insulation (W m\(^{-2}\) k\(^{-1}\)); \(h_m\) is the conduction coefficient of the metal walls of the reactor (W m\(^{-2}\) K\(^{-1}\)); \(h_R\) is the radiation coefficient of side walls (W m\(^{-2}\) K\(^{-1}\)).
The heat transfer coefficient at the reactor wall was calculated by modifying the equation proposed by Kato et al. [41] as follows:

\[
h_A = 0.138 \times (N_{Gr})^{0.36} \times [(N_{Pr})^{0.175} - 0.55] \times k_{air}/L \tag{6}
\]

\[
N_{Gr} = (L^3 \times \rho_{air}^2 \times g \times \beta \times \Delta T)/\mu_{air}^2 \tag{7}
\]

\[
N_{Pr} = c_{p_{air}} \times \mu_{air}/k_{air} \tag{8}
\]

where: \(N_{Gr}\) is the Grashof number, \(N_{Pr}\) is the Prandtl number, \(k_{air}\) is the thermal conductivity of air at 25°C (W m\(^{-1}\) K\(^{-1}\)); \(c_{p_{air}}\) is the specific heat capacity of air at 25°C (kJ kg\(^{-1}\) K\(^{-1}\)); \(\mu_{air}\) is the viscosity of air at 25°C (kg m\(^{-1}\) s\(^{-1}\)); \(\rho_{air}\) is the density of air at 25°C (kg m\(^{-3}\)); \(\beta\) is the coefficient of thermal expansion of air at 25°C (K\(^{-1}\)); \(g\) is the gravitational constant (m s\(^{-2}\)), \(L\) is the reactor height (m); \(\Delta T\) is the average temperature (°C).

The average temperature drop is described in Eq. (9) using the expression proposed by Kumana and Kothari [42].

\[
\Delta T = (T - T_A)/4 \tag{9}
\]

where \(T_A\) is the outside air temperature (°C).

The conduction coefficient of the metal wall and insulation are defined as follows:

\[
h_m = k_M/t_M \tag{10}
\]

\[
h_I = k_I/t_I \tag{11}
\]

where: \(k_M\) is the thermal conductivity of the metal of the reactor at 200°C (W m\(^{-1}\) °C\(^{-1}\)); \(k_I\) is the thermal conductivity of the insulation used (calcium silicate at 200°C, W m\(^{-1}\) °C\(^{-1}\)); \(t_M\) is the reactor wall thickness (m).

The radiation coefficient of side walls is defined by Perry and Chilton [43] as follows:

\[
h_R = 0.1713 \times \varepsilon \times \left[\frac{((T_{IS}+460/100)^4-(T_A+460/100)^4)}{T_{IS}-T_A}\right] \tag{12}
\]

\[
T_{IS} = T_A + 0.25(T - T_A) \tag{13}
\]
where: \( \varepsilon \) is the surface emissivity of the aluminium jacket, \( T_{IS} \) is the outside temperature of the insulated surface (\(^\circ\)C), 460 is the temperature conversion factor from K to \(^\circ\)F. \( T_A \) and \( T_{IS} \) were converted from \(^\circ\)C to K during calculation of \( h_R \).

After the process is started, energy recycled from steam is calculated using the following equation as:

\[
E_s = [m_w \times (\hat{H}_{w(T)} - \hat{H}_{w(T_0)})] - m_0 \times HHV_f
\]  

(14)

where: \( E_s \) is the steam energy (kJ h\(^{-1}\)); \( HHV_f \) is the heating value of the solids in the sludge (kJ kg\(^{-1}\)); \( \hat{H}_{w(T)} \) is the specific enthalpy of water at the reaction temperature, T (kJ kg\(^{-1}\)); and \( \hat{H}_{w(T_0)} \) is the specific enthalpy of water at reference temperature, 25\(^{\circ}\)C (kJ kg\(^{-1}\)).

The total mass of steam produced from flashing, \( m_{V,F} \) was then calculated as:

\[
m_{V,F} = \left( 1 - \frac{\hat{H}_{S,T} - \hat{H}_{w(T)}}{\hat{H}_{S,T} - \hat{H}_{w(120^{\circ}C)}} \right) \times m_w
\]  

(15)

where: \( \hat{H}_{S,T} \) is the specific enthalpy of steam at reaction temperature, T (kJ kg\(^{-1}\)); and \( \hat{H}_{w,120^{\circ}C} \) is the specific enthalpy of water at 120\(^{\circ}\)C (kJ kg\(^{-1}\)).

The energy from the steam for preheating the feedstock, assuming negligible heat losses, is given by:

\[
m_{V,PF}H_{V,(T)} = [m_{FT} \cdot c_{p,FT}(T_f - T_0)] + [m_{FS} \cdot c_{p,FS}(T_f - T_0)] + [m_w(H_{w(T_f)} - H_{w(T_0)})]
\]  

(16)

where: \( c_{p,FT} \) is the specific heat capacity of feed tank (kJ kg\(^{-1}\) K\(^{-1}\)); \( m_{V,PF} \) is the mass of steam from flash vessel for preheating feed (kg); \( m_{FT} \) is the mass of feed tank; \( T_f \) is the feed pretreatment temperature (\(^\circ\)C); and \( H_{w(T_f)} \) is the enthalpy of water at pretreatment temperature (kJ kg\(^{-1}\)).

The energy for drying wet hydrochar, assuming negligible heat losses, is given by:

\[
m_{V,D}H_{V,(T)} = [m_{H,D} \cdot c_{p,H}(T_D - T_H)] + (m_{W,H} \times h_{vap})
\]  

(17)
where: $m_{\nu,D}$ is the mass of steam for drying (kg); $m_{H,D}$ is the mass of wet hydrochar fed to
the drying process (kg); $m_{W,H}$ is the mass of water in hydrochar (kg); $c_{p,H}$ the specific heat
capacity of hydrochar (kJ kg$^{-1}$ K$^{-1}$); $h_{\text{vap}}$ is the latent heat of vaporisation of water (kJ kg$^{-1}$);
$T_D$ is the drying temperature ($^\circ$C); and $T_H$ is the temperature of wet hydrochar entering the
dryer ($^\circ$C).

The energy required to heat the reactor and contents after completion of one process
cycle is given by:

\[
Q_{\text{char}} + Q_{\text{CH}_4} = [m_r \cdot c_{p_r}(T - T_r)] + [m_{FS} \cdot c_{p_FS}(T - T_f)] + \Delta H_R m_0
\]

\[
+ [m_w(H_{L,T} - H_{w,(T_f)})] - [A_r U_r t_h(T - T_f)]
\]  \hspace{1cm} (18)

where: $Q_{\text{char}}$ and $Q_{\text{CH}_4}$ are the energies produced from combustion of dry hydrochar and
methane (kJ), respectively; $T_f$ is the temperature of preheated feedstock ($^\circ$C); and $T_r$ is the
temperature of the reactor body after completion of a process cycle (120$^\circ$C); and $H_{w(T_f)}$ is the
enthalpy of water at the preheat temperature, $T_f$ (kJ kg$^{-1}$).

\[
Q_{\text{char}} = m_t \times \text{HHV of char}
\]  \hspace{1cm} (19)

\[
Q_{\text{CH}_4} = m_{\text{CH}_4} \times H_{\text{CH}_4}
\]  \hspace{1cm} (20)

where $H_{\text{CH}_4}$ is the heat of combustion of methane (50125 kJ kg$^{-1}$) from stoichiometric
combustion equation. $m_{\text{CH}_4}$ is the mass of methane (kg) obtained from the relationship
between mass of CH$_4$ generated and that of COD removed during anaerobic digestion (1 g
COD removed = 0.25 g CH$_4$ produced, which is equivalent to 1.4 L CH$_4$ at STP [44-46], and
on the basis that 90% of the COD was converted to CH$_4$ [47]. That is by proportion: {mass of
CH$_4$ produced (kg) = mass of COD (kg) x 0.9 x 0.25 (kg)}/1 kg COD. Table 2 gives the
physical and thermodynamics properties of the HTC reactor, faecal waste and products.

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<table>
<thead>
<tr>
<th>Parameter</th>
<th>Notation</th>
<th>Unit</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Reactor and other units</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Specific heat capacity of stainless steel</td>
<td>$c_{p,r}$; $c_{p,PT}$</td>
<td>kJ kg$^{-1}$ K$^{-1}$</td>
<td>0.502</td>
</tr>
<tr>
<td>Density of stainless steel reactor (at 25°C)</td>
<td>$\rho_r$</td>
<td>kg/m$^3$</td>
<td>8027.2</td>
</tr>
<tr>
<td>Reactor diameter$^a$</td>
<td>$D$</td>
<td>m</td>
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<td>$D_j$</td>
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<td>Insulation thickness</td>
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<tr>
<td>Volume of heating oil</td>
<td>$V_o$</td>
<td>m$^3$</td>
<td></td>
</tr>
<tr>
<td><strong>Feedstock and products</strong></td>
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</tr>
<tr>
<td>Specific heat capacity of dry sewage sludge</td>
<td>$c_{p,FS}$</td>
<td>kJ kg$^{-1}$ K$^{-1}$</td>
<td>1.7$^c$</td>
</tr>
<tr>
<td>Specific heat capacity of hydrochar</td>
<td>$c_{p,H}$</td>
<td>kJ kg$^{-1}$ K$^{-1}$</td>
<td>1.45$^d$</td>
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<td>$c_{p,w}$</td>
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<td>kJ kg$^{-1}$</td>
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<tr>
<td>Specific enthalpy of water at 25°C</td>
<td>$H_{w(T_o)}$</td>
<td>kJ kg$^{-1}$</td>
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<tr>
<td>Specific enthalpy of water at 100°C</td>
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<tr>
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<td>$H_{V(T_f)}$</td>
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<td>kJ kg$^{-1}$</td>
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<td>Temperature of steam from flash tank</td>
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<td>Temperature of hydrochar to dryer</td>
<td>$T_H$</td>
<td>°C</td>
<td>100</td>
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<tr>
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<td>K$^{-1}$</td>
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<td>Gravitational constant</td>
<td>$G$</td>
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</table>

$^a$ Varies based on reactor size or the number of faeces to be fed.  
$^b$ Calcium silicate.  
$^c$ [31].  
$^d$ [29].  
$^e$ Heat up time and reaction time.
3. Results and Discussion

3.1. Mass balance

Figure 2 summarises the mass balance for the HTC process carried out at 200°C and for a reaction time of 30 min using faecal sludge with solid concentrations of between 5–25%, on a per day basis and assuming a 12 h process operation. The hydrochar yield was about 67% of the initial solids in the faeces following carbonisation under these conditions. After drying, the hydrochar contained about 5% water (using the results of residual moisture content in

Figure 2 – Energy balance on a semi-continuous HTC system based on heat recycled from both the process and products using feedstock with 25% solids and a daily feed rate equivalent to 400.00 kg.
As the quantity of faeces treated increases from 4.00 to 400.00 kg per day, the amount of hydrochar produced after drying increases from 0.12–13.80 kg, 0.48–41.52 kg, and 0.72–69.12 kg when the solid content in the feedstock increases from 5%, 15% and 25%, respectively (Table A.1 in the appendix). The amount of steam from the flash tank increases significantly as the quantity of faeces treated increases from 4.00 to 400.00 kg per day, but decreased as the solids content in the faeces increased or the liquid fraction decreased. The amount of steam released from the flash tank ranged from 0.60–70.56 kg for 5% solids concentration, 0.60–63.24 kg for 15% solid concentration, and 0.60–55.68 kg for 25% solids concentration (Table A.1).

As shown in Figure 2, the amount of steam released from the Flash tank represents about 15% of the total quantity of material (slurry) fed to the Flash tank from the HT reactor, while the remaining 85% consists of solids and water. For feedstock containing 15–25% solids, only about 47% of the amount of steam released is required to preheat the feed to 100°C and dry the hot hydrochar containing about 50% moisture. Therefore, 53% of the steam generated is available for other purposes. The amount of water evaporated during drying of the wet solids ranged between 0.12–12.96 kg, 0.36–38.64 kg, and 0.60–64.56 kg for faeces containing 5%, 15%, and 25% solids respectively as the quantity of faeces treated was raised from 4.00–400.00 kg per day (Table A.2). The mass of estimated methane yields decreased as the solids content in the feedstock increases and ranged from 0.01–1.42 kg, 0.01–1.13 kg, and 0.01–0.85 kg for faeces with 5%, 15%, and 25% solids respectively. Previous studies reported that HTC of biomass wastes followed by AD could enhance methane yields from 72–222% [16-18]; whilst carbonising digestate from AD by HTC has been reported to improve energy recovery from biomass wastes [11,25,48] with the combined AD-HTC doubling the energy recovery compared to AD process alone [48].
The mass of liquid waste that remained after anaerobic digestion ranged between 1.44–143.28 kg, 1.20–114.60 kg, and 0.84–85.92 kg for faeces containing 5%, 15%, and 25% solids respectively; representing about 48% of the liquid filtrate fed to the anaerobic digester, which is recycled. The mass of condensed steam following preheating of the faecal feed decreases as the solids content in the faeces was reduced as a result of the decrease in heat energy required at higher solids contents (Section 3.2). For further processing, the water vapour must be condensed and the condensates sent to the evaporator and sorption stage for recovery of inorganic salts. The mass of ash after combustion of the hydrochar was obtained by multiplying the ash content of the hydrochar following HTC at 200°C for 30 min (about 36% d.b., Table 1) by the mass of hydrochar after drying (Table A.2).

3.2 Energy balance

The energy balance of the HTC process is summarised in Figure 2 and the details presented in Tables A.3 and A.5 in the appendix. The data in Table A.3 are based on the assumption that no heat was recovered from the process or the products, and that the only energy required was that used to heat the reactor to the reaction temperature and for completion of the process; whilst that in Table A.5 are based on energy recovery from the process and the products when the process is in operation. The results clearly indicate that the total amount of energy required to heat feedstock containing a lower amount of solids was higher than that required for feedstock with a higher solids content. This was particularly so for feedstock containing 5% solids. Also, the total energy input to the reactor increases as the quantity of feedstock increases. Although the energy required to heat the reactor increased from 13.92–1458.96 MJ as the quantity of faeces treated per day was increased from 4.00–400.00 kg, there was no change when the solids content in the faeces increased. Of the total energy input, when no heat was recovered from the process or the products the energy required to heat the reactor represented about 63–61% (for 5% solids in faeces), 64–62% (for
15% solids in faeces), and 65–63% (for 25% solids in faeces) of the total energy input; that is a slight decrease as the faeces treated per day was increased, and increased as the solids content of the faeces increases (noting that energy input decreased as the faeces solid content increased, as explained earlier). The energy required for heating all the faecal material represents about 21–20% (for faeces with 5% solids), 20–19% (for faeces with 15% solids), and 19–18% (for faeces with 25% solids) of the total energy input to the reactor, that represents an increase with increases in the feed water content. These results are in keeping with those of previous studies. For example, Thorsness [30] found that energy input in the form of steam increased by approximately 15% as the MSW feed water content increased from 25 to 35%. Stemann and Ziegler [29] also reported that the amount of energy required to heat biomass to the reaction temperature depended significantly on the water content of the biomass. In their study on the energetic assessment of the HTC of woody biomass, increasing the water content of the feedstock resulted in increases in energy input of between 2.2% and 7.3% of the energy of the hydrochar. Heat losses from the insulated reactor increased as the number of person equivalents increased, but did not change with increases in the solids content and accounted for between about 0.5–3% of the total energy input. This serves to indicate the importance of thermal insulation, and heat losses on a commercial scale may become significant if proper insulation is not provided, and this would adversely affect the overall energy efficiency of the process. Thorsness [30] reported that heat loss effects from the walls of the reactor were significant, with an increase in input steam flow rate requirement of about 40% due to adiabatic conditions. Stemann and Ziegler [29] reported that heat losses from the reactor ranged from 0.005–0.2 MW, which accounts for about 0.2% of the system power of the HTC plant, and falls within the range of values obtained in this study. The heat transfer parameters used to
estimate the heat loss from the insulated reactor are presented in Tables 2 and A.4 (in the appendix).

The heat of reaction measured over an interval of 4 h using the DSC were –0.20 MJ kg\(^{-1}\) (±0.01) at 160\(^\circ\)C, –0.32 MJ kg\(^{-1}\) (±0.03) at 180\(^\circ\)C, and –0.70 MJ kg\(^{-1}\) (±0.08) at 200\(^\circ\)C. The heats of reaction were measured over a period of 4 h, as this was the time previously reported [36] as being the time for complete reaction. Funke and Ziegler [36] reported that the amount of energy released increases under severe carbonisation conditions. In their study because cellulose required severe reaction conditions to carbonise, it took longer for the heat to be released than the 30–40 min reported for wood and glucose. For a treatment time of 30 min, the use of such a value measured in 4 h may represent a slight overestimate but in the overall scheme the error involved would be insignificant. The heat of reaction measured at 200\(^\circ\)C for 4 h was closer to the value of –0.79 MJ kg\(^{-1}\) reported for HTC of digestate from anaerobic digested waste that was estimated based on measured higher heating value (HHV) and combustion reactions [25], but significantly lower than the value of –1.6 MJ kg\(^{-1}\) reported for cellulose [5,25], and –1.07 MJ kg\(^{-1}\) and –1.06 MJ kg\(^{-1}\) for cellulose and glucose using DSC measurements [36]. The reaction heat increased as the amount of faeces undergoing treatment was increased, and was calculated by multiplying the mass of faeces fed into the reactor by the heat of reaction measured at 200\(^\circ\)C. The heat of reaction alone cannot sustain the carbonisation reaction as it represents only about 19–20% of the total energy required if the feed had not been preheated (Table A.3 in the appendix), and between 33–35% of the energy if the feed was preheated to 100\(^\circ\)C (Table A.5 in the appendix).

Energy recovered from the steam in the flash tank increased as the solids content in the faeces and mass of faecal sludge was increased. About 4.1, 0.9, and 0.8% of the total energy recovered from steam was used to dry the hydrochar to approximately 5% moisture content for faeces containing 5, 15, and 25% solids respectively; whilst 65 and 33% of the energy
was used to preheat faecal sludge with solid contents of 15 and 25% respectively (Table A.3 in the appendix). Preheating the feed reduced the energy required to heat the reactor by about 59%. However, energy recovered from steam for faeces containing 5% solids was not sufficient to preheat the feed before it was fed to the HTC reactor. For faeces containing 25% solids, about 63–64% of the total energy from combustion of the hydrochar and methane were used to power the reactor; indicated that the surplus energy could be utilised for other purposes.

Alternatively, the energy generated from combustion of the methane and the excess energy recovered from the flashing off of steam alone (71–81%) were sufficient for powering the entire HTC system; hence, the hydrochar can be used for other applications such as addition to soil as a soil conditioner and carbon sequestration or combustion for syngas production. It must be noted that higher methane yields were obtained when the solids content of the faeces was low. Preheating the faeces to 100°C before it was fed to the reactor reduced the heat losses from the reactor to between 50–60%, and also decreased the total heat input required to heat the reactor and faecal content to the reaction temperature of 200°C by 59% (Table A.5 in the appendix). Zhao et al. [12] reported that about 48% of the heat generated from hydrochar combustion could be recovered, while the total energy recovery for HTC processing at temperatures above 200°C was approximately between 40% and 60% if the reactor was preheated and when ignoring preheating, respectively. However, this could be lower as heat losses were not considered in their study.

3.3. Sensitivity analysis

The amount of faeces to be treated, and the concentration of solids in the faeces significantly determined the overall process energetics (Tables A.3 and A.5 in the appendix). The latter were varied as the input parameters in the HTC process from 4.00–400.00 kg per day for the feed rate, and 5–25% for the solids content. For a higher solids content (15–25%)
sufficient energy is recovered from flashing off the steam, which can be used to preheat the
feed to 100°C and drying the wet hydrochar with 50% moisture to 5%, with excess energy of
up to 34% (for 15% solids) and 66% (for 25% solids in faeces). For faeces with 25% solids
content energy from combustion of the hydrochar was enough to operate the reactor, leaving
a surplus of between 33–35%. This decreased as the amount of faecal waste increased from
4.00–400.00 kg/day. For HTC of feedstock with 15% solids, using the energy from
combustion of the hydrochar and the surplus energy from steam (10.08–1021.08 MJ per day,
Table A.5) would be sufficient to operate the HTC reaction. A feedstock containing 5%
solids produces the highest amount of methane (Table A.2 in the appendix), about 125 and
167% more than that produced from feedstock with 15 and 25% solids respectively; and
consequently generating more energy from its combustion. For all solids contents and
feedstock rates, the amount of energy generated from the combustion of methane alone was
insufficient to operate the reactor. However, for a feedstock containing 15% solids the excess
energy from flashing off of steam and the energy from combustion of both methane and the
hydrochar were sufficient to operate the HTC reactor with surplus energy of about 21–22%.
Also, for a feedstock containing 25% solids energy from combustion of methane and the
hydrochar were sufficient to operate the reactor leaving excess of about 36–37%. It must be
noted that the methane yield was based on an empirical estimation and that prediction of the
percentage of CH₄ in biogas is difficult and depends on the pH in the anaerobic digestion
reactor, which is influenced by the equilibrium CO₂. This is because carbon dioxide is
partially soluble in water, and so is partly dissolved in the liquid phase or converted to
bicarbonate depending on the pH; but the CH₄ produced is practically insoluble in water and
is mostly present in the gas phase. As a result, the estimated CH₄ yield will generally be
lower than the fraction of CH₄ in biogas produced from experimental anaerobic digestion
tests.
4. Conclusions

The solids contents of the feedstock and the amount of feed material had a significant effect on the material and energy balances of the HTC of faecal sludge. Although feedstocks of lower solids content produced more steam, the steam energy from feedstock with 5% solids was not sufficient for preheating the feed although it was enough for drying the wet hydrochar. In a process where the liquid products were not digested for methane production and for feedstocks containing 15 and 25% solids, once the process has started energy recovery from flashing off steam, and combustion of the char would be sufficient for operating the entire process without the need for any external sources of energy. Alternatively, for a feedstock with 25% solids content and all feed rates, 79–81% of the energy from combustion of methane and the excess energy recovered from flashing off of steam were sufficient for sustaining the process, and the remaining 19–21% could be utilised for other purposes; hence the hydrochar could be used for carbon sequestration when applied to soil or for other applications such as gasification for syngas production. Further investigations would need to be conducted at different reaction temperatures to fully establish the effect of temperature on the energetics of the process. Also, studies into a detailed life-cycle and economic analysis of the process would be useful to confirm the sustainability of the process.

Acknowledgements

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References


### Table A.1
Mass balance of faecal sludge HTC as a function of feedstock quantity and solids content

<table>
<thead>
<tr>
<th>Feedstock * (kg)</th>
<th>Faecal Sludge (kg)</th>
<th>Flashing (kg)</th>
<th>Dewatering (kg)</th>
<th>Drying (kg)</th>
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<tbody>
<tr>
<td></td>
<td>Solids</td>
<td>Water</td>
<td>Slurry</td>
<td>Steam</td>
</tr>
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<td></td>
<td>Solid cake</td>
<td>Liquid</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Hydrochar</td>
<td>Evaporated water</td>
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<tr>
<td>4.00</td>
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<td>12.96</td>
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</tbody>
</table>

| 4.00             | 0.60              | 3.40          | 3.12            | 0.60        |
|                  |                   |               | 0.84            | 2.28        |
|                  |                   |               |                 | 0.48        |
|                  |                   |               |                 | 0.36        |
| 8.00             | 1.20              | 6.80          | 6.36            | 1.32        |
|                  |                   |               | 1.56            | 4.80        |
|                  |                   |               |                 | 0.84        |
|                  |                   |               |                 | 0.72        |
| 40.00            | 6.00              | 34.00         | 31.68           | 6.36        |
|                  |                   |               | 8.04            | 23.64       |
|                  |                   |               |                 | 4.20        |
|                  |                   |               |                 | 3.84        |
| 200.00           | 30.00             | 170.00        | 158.52          | 31.56       |
|                  |                   |               | 40.08           | 118.44      |
|                  |                   |               |                 | 20.76       |
|                  |                   |               |                 | 19.32       |
| 400.00           | 60.00             | 340.00        | 317.04          | 63.24       |
|                  |                   |               | 80.16           | 236.88      |
|                  |                   |               |                 | 41.52       |
|                  |                   |               |                 | 38.64       |

| 4.00             | 1.00              | 3.00          | 3.12            | 0.60        |
|                  |                   |               | 1.32            | 1.80        |
|                  |                   |               |                 | 0.72        |
|                  |                   |               |                 | 0.60        |
| 8.00             | 2.00              | 6.00          | 6.24            | 1.08        |
|                  |                   |               | 2.64            | 3.60        |
|                  |                   |               |                 | 1.44        |
|                  |                   |               |                 | 1.20        |
| 40.00            | 10.00             | 30.00         | 31.08           | 5.52        |
|                  |                   |               | 13.32           | 17.76       |
|                  |                   |               |                 | 6.96        |
|                  |                   |               |                 | 6.36        |
| 200.00           | 50.00             | 150.00        | 155.64          | 27.84       |
|                  |                   |               | 66.84           | 88.8        |
|                  |                   |               |                 | 34.56       |
|                  |                   |               |                 | 32.28       |
| 400.00           | 100.00            | 300.00        | 311.16          | 55.68       |
|                  |                   |               | 133.68          | 177.48      |
|                  |                   |               |                 | 69.12       |
|                  |                   |               |                 | 64.56       |

* On a per day basis, and assuming that the plant operates 12 hours a day. Solids in the faeces reduced by 66.8% following carbonisation.
Table A.2
Mass balance of faecal sludge HTC resulted from recovered and waste materials

<table>
<thead>
<tr>
<th>Feedstock (kg)</th>
<th>Feed stock Pre-heating (kg)</th>
<th>Anaerobic Digestion (kg)</th>
<th>Methane ( ^b ) Combustion (kg)</th>
<th>Char Combustion (kg)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Condensed Steam COD</td>
<td>Methane Waste Water</td>
<td>Water vapour</td>
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<tr>
<td>4.00</td>
<td>0.28</td>
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\( ^a \) On a per day basis, and assuming that the plant operates 12 hours a day. \( ^b \) From reaction stoichiometry: 1 kg CH\(_4\) makes 2.25 kg (16/36) kg H\(_2\)O.
### Table A.3
Energy balance of faecal sludge HTC without heat recovery

<table>
<thead>
<tr>
<th>Feedstock (kg)</th>
<th>5% Solids in faeces</th>
<th>Hydrothermal Treatment (MJ)</th>
<th>Flashing (MJ)</th>
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<tbody>
<tr>
<td></td>
<td>Total input</td>
<td>Energy to heat reactor</td>
<td>Energy to faeces &amp; water</td>
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<tr>
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<td>17.88</td>
<td>0.12</td>
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<td>0.60</td>
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<td>1458.96</td>
<td>894.96</td>
<td>6.00</td>
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<table>
<thead>
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<th>15% Solids in faeces</th>
<th>Hydrothermal Treatment (MJ)</th>
<th>Flashing (MJ)</th>
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<td>Total input</td>
<td>Energy to heat reactor</td>
<td>Energy to faeces &amp; water</td>
</tr>
<tr>
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<table>
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<th>Hydrothermal Treatment (MJ)</th>
<th>Flashing (MJ)</th>
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<td>Total input</td>
<td>Energy to heat reactor</td>
<td>Energy to faeces &amp; water</td>
</tr>
<tr>
<td></td>
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<td>faeces</td>
</tr>
<tr>
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<td>13.92</td>
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<td>17.88</td>
<td>0.60</td>
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<td>29.76</td>
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On per day basis, and assuming that the plant operates 12 hours a day. The faeces are not preheated before fed to the reactor.

### Table A.4
Heat transfer parameters

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<thead>
<tr>
<th>Feedstock (kg)</th>
<th>L (m)</th>
<th>D (m)</th>
<th>A (m²)</th>
<th>N_GR</th>
<th>N_P</th>
<th>h_L (w m⁻² K⁻¹)</th>
<th>h_M (w m⁻² K⁻¹)</th>
<th>h_A (w m⁻² K⁻¹)</th>
<th>h_G (w m⁻² K⁻¹)</th>
<th>U_L (w m⁻² K⁻¹)</th>
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<td>0.09</td>
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<td>534.59</td>
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<td>0.87</td>
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<td>534.59</td>
<td>4.14</td>
<td>0.16</td>
<td>0.87</td>
</tr>
<tr>
<td>40.00</td>
<td>0.32</td>
<td>0.22</td>
<td>0.36</td>
<td>1.86 x 10⁸</td>
<td>0.77</td>
<td>1.10</td>
<td>534.59</td>
<td>4.32</td>
<td>0.16</td>
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<tr>
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<td>1.86 x 10⁹</td>
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<td>534.59</td>
<td>4.59</td>
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*On a per day basis, and assuming that the plant operates 12 hours a day.*
Table A.5
Assessment of energy balance of faecal sludge HTC with heat recovery

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a Includes energy to heat feed tank, faeces and water to 100°C. b Energy from combustion of the hydrochar and methane. c Only part of the energy is used to preheat the feedstock and dry the char, and the remainder represents a surplus; “deficit” indicates that the energy is used only to dry the char but is insufficient to preheat the feed. d Surplus energy recovered from steam, and from combustion of both char and methane after using part of the energy to operate the reactor. On per day basis, and assuming that the plant operates 12 hours a day.