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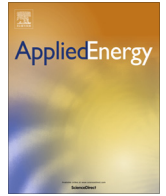
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# High-rate thermophilic bio-methanation of the fine sieved fraction from Dutch municipal raw sewage: Cost-effective potentials for on-site energy recovery



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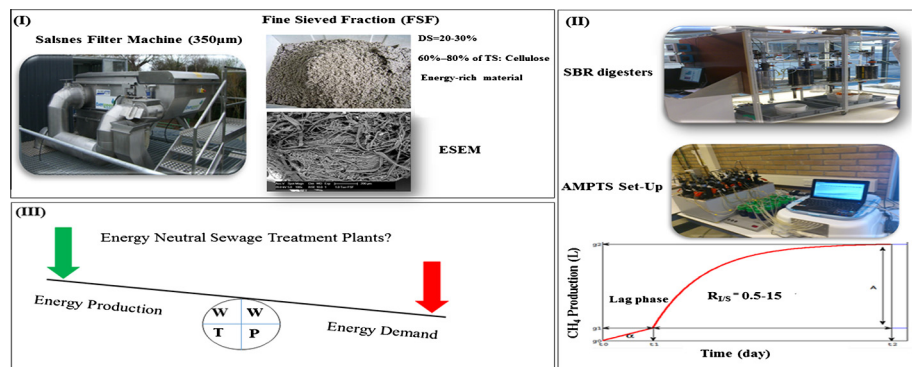
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## HIGHLIGHTS

- Fine sieved fraction (FSF) from raw municipal sewage was used a sole substrate.
- Various mesophilic and thermophilic BMP tests were run at different  $R_{I/S}$  ratios.
- Thermophilic digestion of FSF is highly efficient for on-site energy recovery.
- Biogas production rate of  $9.3 \text{ m}^3/\text{m}^3 \text{ d}$  at OLR of  $22.8 \text{ kgCOD}/\text{m}^3 \text{ d}$  is predicted.
- The net recoverable energy of 287 MJ/ton FSF and 237 kW h/ton FSF was found.

## GRAPHICAL ABSTRACT



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## ABSTRACT

Sieving of Dutch raw sewage over a  $350 \mu\text{m}$  screen, produces a cake layer called fine sieved fraction (FSF), an energy-rich material that contains mainly cellulosic fibers originating from toilet paper. The FSF bio-methane potential (BMP) was studied under both mesophilic ( $35 \text{ }^\circ\text{C}$ ) and thermophilic ( $55 \text{ }^\circ\text{C}$ ) conditions, whereas the stability of the fed-batch digesters at both  $35 \text{ }^\circ\text{C}$  and  $55 \text{ }^\circ\text{C}$  was researched by varying the inoculum to substrate ratios ( $R_{I/S}$ : 0.5–15). Results clearly showed advantages of thermophilic conditions over mesophilic conditions at all tested  $R_{I/S}$ . Stable digestion was even possible at an  $R_{I/S}$  of 0.5 at  $55 \text{ }^\circ\text{C}$ .

Following the results of the batch tests, a compact high loaded thermophilic digester for on-site energy recovery from FSF was proposed. Based on the results of the study, high biogas production rates at high organic loading rates (OLRs) were predicted. In the energy balance calculations, surplus heat production from combined heat and power (CHP) was utilized to dry the digestate sludge before transportation to an incineration plant or for use in pyrolysis or gasification processes. Overall results showed the potential of generating 46% of the required energy for wastewater treatment via high rate FSF digestion and subsequent conversion of the bio-methane into electricity and heat. The net recoverable energy from fine sieving, anaerobic digestion of FSF, dewatering of digestate sludge and drying of dewatered digestate sludge amounted 287 MJ/ton FSF and 237 kW h electric/ton FSF at 23% TS.

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## 1. Introduction

Energy recovery from raw municipal sewage for on-site use will minimize the fossil energy demand and contribute to the development towards energy neutral wastewater treatment plants (WWTPs). In principle this should be possible since the energy content of sewage is several times higher than the energy required for its treatment [1,2]. For indeed achieving energy neutrality, or even energy production, at WWTPs, the energy balance should be optimized which requires a dual approach. Firstly, the total energy consumption during wastewater treatment requires optimization, such as more energy-efficient aeration and less energy losses in pumping and sludge dewatering. Moreover, implementing enhanced primary sludge production and alternative routes for nitrogen removal can drastically reduce the use of fossil fuels for aeration. Secondly, the recovery of chemically bound energy should be maximized, requiring an upgraded anaerobic digestion (AD) technology as well as the implementation of AD at those WWTPs that so far are not served by AD, such as extended aerated biological nutrient removal plants [1–5]. The biggest energy gains per m<sup>3</sup> of sewage can be made in small WWTPs that are not equipped with primary clarifiers and that apply low sludge loading rates. In these systems, all incoming biochemical oxygen demand (BOD), as well as a large extent of the newly grown sludge is converted aerobically. With a biomass growth yield of 0.6 g volatile suspended solids (VSS)/g BOD and a sludge degradation efficiency of 30–50% during digestion, one can easily reason that a large part of influent BOD is lost for energy recovery during the activated sludge treatment process [6]. In Western industrialized countries, a significant part of the sewage BOD consists of cellulose (60–80% of total solids content), originating from the use of toilet paper [7,8]. Conventionally, a significant part of this cellulose fraction is removed in large conventional primary settlers. If primary settlers are absent, or only part of the cellulose is retained, the cellulose BOD is (partly) oxidized in the aeration tanks [8]. A very compact and efficient solution to minimize oxidation of filterable matter in extended aeration tanks is the recovery of cellulose-rich slurries from raw sewage with a fine-mesh (<500 µm) sieve. The derived fine sieved fraction (FSF) can then be used for on-site energy recovery through anaerobic digestion, instead of oxidation in the aeration tank. However, care should be taken that the required nutrient removal capacity remains unaffected.

At the WWTP Blaricum, the Netherlands, a 350 µm mesh size fine sieve (Salsnes Filter, Norway) for raw sewage mechanical pre-treatment is installed after the coarse screen (6 mm) as a pilot study. This sieve was implemented as a compact alternative to primary clarification taking into account that the composition of the material coming from the fine sieve deviates from conventional primary sludge [8]. At present, application of fine sieves receives growing interest in countries like the Netherlands, and water authorities are even exploring the recovery of cellulosic fibers for reuse. On the other hand, onsite bio-methanation of FSF at high dry solids contents, could contribute to the objective of drastically minimizing the fossil energy requirements at conventional WWTPs, eventually leading to energy neutral WWTPs [9]. The FSF is a heterogeneous substrate, sequestered from raw sewage, which mainly consists of partly dissolved toilet paper (with a high cellulose fraction), hair, lignin compounds such as leaves and shell of fruits as well as sands and undefined materials. FSF composition was determined to consist of 60–80% of cellulose, 5–10% of hemicellulose, 5–10% of lignin, 5–10% of oil and the rest accounted for inorganic salts (5–10%) [10,11].

For anaerobic digestion, thermophilic (50–60 °C) or mesophilic (30–40 °C) conditions can be chosen [12–14]. Mesophilic anaerobic digestion of organic solids is often reported as most convenient, stable and reliable form of substrate conversion leading to stable

methane production rates. However, mesophilic hydrolysis rates are lower than thermophilic conversion rates [15], since the rate of many, if not most, (bio)chemical reactions double as the reaction temperature increases by 10 °C [16]. On the other hand, thermophilic digestion requires higher energy input, and is regarded more sensitive to changes in operational conditions, such as changes in temperature and organic loading rates, as well as to changes in substrate characteristics [17,18]. The higher vulnerability could be due to a less diverse microbial community [19], persistence of propionate [20] and increased toxicity of intermediates at the thermophilic temperature range [18]. Lignocellulosic biomass, which has similar characteristics to FSF, has been widely used for bio-methanation by coupling cellulolytic microorganisms, fermenting bacteria and methanogenic archaea in one or two-stage anaerobic bioreactors [21,22]. Thermophilic anaerobic digestion of lignocellulosic biomass, such as FSF, might be more effective than mesophilic digestion [23]. Furthermore, high temperatures can also increase substrate solubility [24] and decrease the bulk liquid viscosity [25], leading to improved mixing performance and thus an increased hydrolysis of (hemi-)cellulose to monomers [26].

A filter cake containing very high dry solids concentrations (20–30%) without any chemical additions is one of the main advantages of fine sieving [7]. For comparison, primary and secondary sludge reach only 6% after thickening or 20% when polymer dosage is applied [27]. It is noteworthy that dewatering of FSF to 40–50% dry solids content is simply possible by applying mechanical pressure [8].

The high dry solids concentrations of FSF make the use of (semi) dry digesters possible, a technique that is usually applied in the digestion of the organic fraction of municipal solid waste (OFMSW) or food and yard waste digestion [23,28]. During past research in fed batch laboratory scale systems, digestion of FSF under thermophilic conditions has been shown to be more efficient and reliable than under mesophilic conditions [7]. Higher substrate doses could be applied and the measured higher reaction rate is expected to lead to a more efficient process with a lower retention time, thus leading to smaller reactor volumes [29–31]. The additional amount of required heat for thermophilic operation might be offset if higher biogas production yields are attained under these conditions [29]. In general, at a fixed solids retention time, a thermophilic digester indeed produces more methane per weight of biomass than the mesophilic counterpart [32–34].

Allowable substrate loading, bio-methanation rates as well as the maximum substrate conversion rates, are parameters required for the design and operation of a biogas plant [35,36]. Besides, the optimal inoculum to substrate ratio ( $R_{I/S}$ ) is considered a crucial parameter for design of batch-wise or plug flow operated solid state anaerobic digestion processes, since it indicates the allowable substrate loading [37]. Therefore, the hydrolysis kinetics, and optimal  $R_{I/S}$  were assessed using biochemical methane potential (BMP) tests. The BMP tests were conducted with well-adapted sludges at different  $R_{I/S}$  under both mesophilic (35 °C) and thermophilic (55 °C) conditions. Based on these results, the energy potential of FSF was found and the digestion of FSF for onsite energy recovery was evaluated towards energy neutrality at WWTPs using the design of a compact plug flow digester.

## 2. Materials and methods

### 2.1. Substrate

FSF was collected from a 350 µm mesh fine sieve (Salsnes Filter, Norway) at WWTP Blaricum, the Netherlands and was stored at 4 °C prior to conduct the BMP tests. Total solids (TS) and volatile

solids (VS) were measured on weight base (g/L) according to the standard methods for the examination of water and wastewater [38]. Chemical oxygen demand (COD) was measured using Merck photometric cell tests (500–10,000 mg/L, Merck, Germany). All analyses were done in triplicate.

## 2.2. Inoculum

Thermophilic inoculum was obtained from a plug flow dry anaerobic composting (DRANCO, OWS, Brecht, Belgium) digester [23], operated at a solid retention time (SRT) of 15 days and treating mainly vegetable, fruit and yard (VFY) wastes with a dry matter content of about 35% and a heterogeneous appearance. The thermophilic inoculum was sieved (4 mm mesh) prior to use. Mesophilic inoculum was taken from an anaerobic digester of a WWTP (Harnaschpolder, Delft, the Netherlands) that treats both primary and secondary sludge with a maximum solid content of 5% and which was operated at an SRT of 22 days [7]. A seven months adaptation time (over 200 days) was observed to be needed to obtain stable reactor operation for both thermophilic and mesophilic digesters in order to adapt to FSF as the sole substrate with varying dry solids content of 10–25% [7]. The inoculum consisted of well-adapted sludges, directly taken from mesophilic and thermophilic laboratory mixed fed-batch digesters after a stable reactor operation of 480 days. The laboratory reactors were at steady state either at 35 °C or 55 °C, at organic loading rate (OLR) of 2.5 and 5.5 kgCOD/m<sup>3</sup> d, respectively, at the time of inoculum sampling. The characterization of both inoculates was similar to the characterization of substrate, using the same methodology. The pH of the mesophilic and thermophilic sludge, prior to the experiments, were 7.0 ± 0.1 and 7.6 ± 0.2, under mesophilic and thermophilic conditions, respectively.

## 2.3. Volatile fatty acid (VFA)

Volatile fatty acids (VFAs) were quantified by Gas Chromatography (GC, Agilent Technology 7890A), using a flame ionization detector (FID) and a capillary column type HP-PLOT/U (25 m × 320 μm × 0.5 μm) with helium as the carrier gas at a flow rate of 67 mL/min and a split ratio of 25:1. The GC oven temperature was programmed to increase from 80 to 180 °C in 10.5 min. The temperatures of injector and detector were 80 and 240 °C, respectively, and the injected volume was 1 μL. Prior to GC analysis, 10 mL of digested samples was first centrifuged at 13,000 rpm for 15 min and then the supernatant was filtered over 0.45 μm filter paper. The filtered liquid was diluted 2 and 3 times with pentanol as internal solution (300 ppm) for mesophilic and thermophilic digestion samples, respectively. Finally, 10 μL of formic acid (purity >99%) was added to the 1.5 mL vials.

## 2.4. Specific Methanogenic Activity (SMA)

Specific methanogenic activity (SMA) assays were used to determine the rate capacity of methanogenic microorganisms to convert acetate into CH<sub>4</sub> in the anaerobic system. In this study, the SMA of the mesophilic and thermophilic sludge was determined using an Automated Methane Potential Test System (AMPTS-II) from Bioprocess Control (Sweden). The SMA was conducted using sodium acetate COD at a concentration of 2 g/L as the substrate, supplemented by a medium consisting of a mixture of macronutrients, trace elements and phosphate buffer solution [39]. The inoculum amount was determined by setting an inoculum VS to substrate COD ratio (I/S) of 2:1. SMA was calculated by using the steepest slope of the accumulating methane production curve (mL/d) divided by the amount of VS introduced in the bottle (inoculum), using the proper conversion factor from CH<sub>4</sub> to COD to

express the final values in gCOD-CH<sub>4</sub>/gVS d. Experiments were conducted in triplicate.

## 2.5. Biomethane potential (BMP) assays

The anaerobic biodegradability of the FSF was performed using the same AMPTS-II, applying adopted protocols suggested by Angelidaki et al. [40,41]. The 250 mL batch flasks containing inoculum and substrate were incubated in a temperature controlled rotational shaker (New Brunswick™ Biological Shakers Innova® 44/44R, USA) at 150 rpm, instead of using the AMPTS-II individual stirrers and waterbath. CO<sub>2</sub> and H<sub>2</sub>S gas were stripped from the biogas by leading the biogas through 100 mL bottles containing a 3 M NaOH solution. Hereafter the remaining gas, containing methane, flows into a gas flow cell with a calibrated volume. When the gas volume equals the calibrated volume of the flow cell, the gas was released and recorded as one normalized volume at time *t*. The test is finished at the moment gas production stops. Biodegradation experiments were performed in duplicate for all *R<sub>I/S</sub>* values. In each test, a blank for the inoculum was included in triplicate. Every batch flask contained the same amount of inoculum, meanwhile the desired *R<sub>I/S</sub>* was obtained using different amounts of substrate (duplicate measurements). After adding the required amounts of inoculum and substrate, each bottle was filled with a medium including macro-and micro-nutrients and buffer solution to maintain the designated volume of 0.2 L, according to the mentioned protocols above [39]. All batch tests including SMA and BMP blank were conducted in triplicate and tests with different *R<sub>I/S</sub>* were carried out in duplicate. It is noted that standard deviation for SMA and BMP blank and the error from average values for all assessed *R<sub>I/S</sub>* ratios of BMP tests under both thermophilic and mesophilic conditions were calculated to be less than 5%.

## 2.6. BMP analysis

The BMP is the net methane production per gram substrate VS added during the entire incubation period (subtracting the blank methane production) at standardized temperature and pressure (273 K, 100 kPa) which has the unit of mLCH<sub>4</sub>/gVS<sub>added</sub>.

## 2.7. Specific methane production rate (SMPR)

The SMPR (mLCH<sub>4</sub>/gVS<sub>inoc.</sub> d) was obtained by dividing the daily produced methane volume by the grams of inoculum VS added.

## 2.8. Anaerobic biodegradability (AnBD)

Anaerobic biodegradability (AnBD) was assessed as the measured ultimate methane production (expressed in COD) over the initial total COD of the substrate [42]. The relationship between AnBD and BMP [43] is expressed by Eq. (1)

$$\text{AnBD} = \frac{\text{BMP}(\text{mLCH}_4/\text{gVS})}{350 \times \text{COD}_{\text{substrate}}(\text{gCOD}/\text{gVS})} \quad (1)$$

Giving the conversion 1 CH<sub>4</sub> + 2O<sub>2</sub> → CO<sub>2</sub> + 2H<sub>2</sub>O, 1 gCOD equals 350 mL of CH<sub>4</sub> at standard temperature and pressure. It is noted that this theoretical approach does not take into account the needs for bacterial cell growth and their maintenance, which has been reported typically 5–10% of organic material degraded [39], meaning that not all biodegraded COD is transformed into methane. Moreover, during bioconversion non-methanised biodegradable or non-biodegradable intermediates may occur, lowering the actual methane yield of the substrate.

## 2.9. Hydrolysis constant ( $K_h$ )

Calculation of  $K_h$  was done according to the protocol published by Angelidaki et al. [40].  $K_h$  describes the velocity of degradation and typically follows first-order kinetics, assuming non-limited growth, meaning no inhibition, and no lack of macro or micro-nutrients [35,44,45]. By assuming that hydrolysis was the rate-limiting step, a first-order kinetic model was used for calculating the  $K_h$ , Eq. (2):

$$P = P_{max} \cdot [1 - \exp(-K_h \cdot t)] \quad (2)$$

where  $P$  = cumulative methane production from the BMP assay at time  $t$  (mL),  $P_{max}$  = ultimate methane yield from BMP assay at the end of the incubation time (mL),  $K_h$  = first-order hydrolysis rate constant (1/d).  $K_h$  can be derived from the slope of the linear regression line plotted for the net accumulated methane production against time at all  $R_{IJS}$  ratios.

## 2.10. Dissolved organic carbon (DOC)

All samples were stored at 4 °C after 0.45 µm filtration (Whatman, Dassel, Germany) to prevent biodegradation of organic matter and were characterized within 3 days after that the BMP experiments were finished. The concentration of bulk organic matter was determined as DOC by a total organic carbon analyzer (TOC-V<sub>CPN</sub>, Shimadzu, Japan) for both mesophilic and thermophilic digested samples.

## 2.11. F-EEM

Fluorescence spectra were collected using a Perkin-Elmer LS-50B luminescence spectrophotometer, which uses a 450 W xenon lamp. All samples were diluted with carbon-free electrolyte solution at pH about 7.5. Fluorescence excitation–emission matrix (F-EEM) spectroscopy was carried out at a concentration of 1 mg C/L to minimize the inner-filter effect. The acquisition interval and integration time were maintained at 0.5 nm and 0.1 s, respectively. Right-angle geometry was used for liquid samples in a 10 mm fused-quartz cuvette. Three dimensional spectra were obtained by repeatedly measuring the emission spectra within the range of 280–600 nm, with excitation wavelengths from 200 to 400 nm, spaced at 10 nm intervals in the excitation domain. Spectra were then concentrated into an excitation–emission matrix (EEM).

## 2.12. Physicochemical inoculum and FSF characteristics

Tables 1 and 2 describe the physicochemical characteristics of the mesophilic and thermophilic inoculum as well as FSF, used as the sole substrate during all experiments. Information on the added FSF and inoculum amounts during the BMP assays under both temperature conditions is presented in Table 2. The tested  $R_{IJS}$  were 0.5, 1, 3, 5, 10 and 15, which were calculated by keeping a constant inoculum concentration at 27.6 and 30 gVS/L for mesophilic and thermophilic conditions, respectively. The substrate concentrations ranged from 55 ( $R_{IJS} = 0.5$ ) to 1.9 gVS/L ( $R_{IJS} = 15$ ) for the mesophilic conditions and from 60 ( $R_{IJS} = 0.5$ ) to 2 gVS/L

( $R_{IJS} = 15$ ) for the thermophilic conditions. Working volume of the batch digested bottles was 0.2 L and inoculum used in each bottle was 0.14 L.

## 2.13. Calculations for energy recovery potential at full scale

### 2.13.1. Plug flow reactor

For assessing the energy recovery potential at full scale, a horizontal plug flow reactor was considered with recirculation of the digested waste. Plug-flow operation was designed as a pulse fed batch reactor. Contact time ( $C_T$ ) in a plug flow reactor depends on the recirculation factor ( $R$ ).  $R$  can be defined from Eq. (3):

$$R = Q_R/Q_W \quad (3)$$

where  $Q_R$  (m<sup>3</sup>/day) is the amount of recirculated inoculum and  $Q_W$  (m<sup>3</sup>/day) is the amount of the waste fed to the reactor.

For a reactor with recirculation,  $C_T$  is defined according to Eq. (4):

$$C_T = V/(Q_w + Q_R) \quad (4)$$

where  $V$  is the reactor volume (m<sup>3</sup>). The solids retention time (SRT) for a reactor, which is operated with or without recirculation of digested waste, is defined as:

$$SRT = V/Q_W \quad (5)$$

The solids retention time can be calculated by substituting Eqs. (3) and (4) into Eq. (5), leading to:

$$SRT = C_T \cdot (1 + R) \quad (6)$$

### 2.13.2. Energy calculations

**2.13.2.1. Specific heat capacity.** The heating requirement of the incoming FSF was calculated based on the measured dry solids content of FSF (23% TS). FSF consisted of about 60–80% cellulose and the rest included hair, sands and clay [11]. It is noted that the sand trap at WWTP Blaricum is by passed when the fine sieve was in operation, explaining the relatively high fraction of inorganics and sand in the collected FSF (Fig. 5). However, in the below calculations it was assumed that FSF contained 80% cellulose and 20% clay. The specific heat capacity of cellulose ( $C_C$ ), water ( $C_W$ ) and clay ( $C_{Cl}$ ) is 1.55 kJ/kg °C, 4.20 kJ/kg °C and 0.92 kJ/kg °C, respectively, resulting in a specific heat capacity of FSF of 3.56 kJ/kg °C (23% TS). However, for the sludge entering the digester, i.e. the sum of incoming FSF sludge and return sludge at 50% recirculation with 9% dry solids content, an average specific heat capacity of 3.78 kJ/kg °C ( $C_F$ ) was calculated. It is noted that the heat capacity of solids in sludge ( $C_{P_{sludge}}$ ) was assumed to be 1.95 kJ/kg °C and was determined based on the range of heat capacities of general organic and inorganic substances [46]. This value is close to the value of 2.1 kJ/kg °C reported by Annadurai et al. [47].

**2.13.2.2. Heating and temperature control.** The heat requirement of sludge digesters generally depends on: (i) the temperature difference between incoming sludge flow and digester; (ii) heat losses through reactor walls, floor and roof [6]; (iii) heat losses that might occur through piping and (iv) biogas production. By appropriate construction, the heat losses in the piping can be minimized to the point where such losses can be neglected [48]. The measures

**Table 1**

Physicochemical characteristics of the mesophilic and thermophilic inoculum as well as FSF used as the sole substrate (mean values ± standard deviations of triplicates).

Component	COD (g/L)	TS (g/L)	VS (g/L)	VS/TS	COD/VS
Mesophilic_Inoculum	65.0 ± 3.2	50.0 ± 0.4	39.5 ± 0.4	0.79	1.64
Thermophilic_Inoculum	67.0 ± 2.9	52.0 ± 0.3	43.0 ± 0.3	0.82	1.56
FSF	342.0 ± 15.5	233.0 ± 1.5	220.0 ± 1.5	0.94	1.56

**Table 2**  
Mesophilic (M) and thermophilic (T) experiment set-up (mean values ± standard deviations of triplicates).

Components	$R_{IJS} = 0.5$		$R_{IJS} = 1$		$R_{IJS} = 3$		$R_{IJS} = 5$		$R_{IJS} = 10$		$R_{IJS} = 15$	
	M	T	M	T	M	T	M	T	M	T	M	T
FSF (g/bottle)	50.3 ± 0.5	54.6 ± 0.5	25.1 ± 0.3	27.3 ± 0.3	8.4 ± 0.2	9.1 ± 0.2	5.0 ± 0.2	5.5 ± 0.2	2.5 ± 0.1	2.7 ± 0.1	1.7 ± 0.1	1.8 ± 0.1
gVS/L (FSF)	55.0 ± 0.5	60.0 ± 0.5	27.5 ± 0.3	30.0 ± 0.3	9.0 ± 0.3	10.0 ± 0.3	5.5 ± 0.2	6.0 ± 0.2	2.8 ± 0.1	3.0 ± 0.1	1.9 ± 0.1	2.0 ± 0.1
gCOD/L (FSF)	86.0 ± 4.2	93.5 ± 4.5	43.0 ± 2.1	46.8 ± 2.3	14.4 ± 0.7	15.6 ± 0.7	8.6 ± 0.5	9.4 ± 0.5	4.3 ± 0.2	4.7 ± 0.2	2.9 ± 0.2	3.0 ± 0.2

and calculations for these heat losses are discussed one by one below.

**2.13.2.3. Heat exchanger.** To minimize heat losses through the incoming sludge, conventional counter current flow heat exchangers can be used to pre-heat the incoming sludge flow with the treated digestate [48]. The temperature of incoming sludge after applying heat exchanger can be calculated using Eq. (7):

$$T_F = (T_{OUT} - T_{IN}) \cdot \varepsilon_H + T_{IN} \quad (7)$$

where  $T_F$  (°C) is the temperature of feed sludge to digester,  $T_{IN}$  (°C) is the temperature of incoming sludge,  $T_{OUT}$  (°C) is the temperature of the digestate and  $\varepsilon_H$  is the efficiency of the heat exchanger, which was assumed at 70% [49]. Average temperature of incoming sludge was estimated at 15 °C.

**2.13.2.4. Heat requirement.** The required heat for increasing the temperature of the incoming sludge flow can be calculated using Eq. (8):

$$Q_H = Q_{(W+R)} \cdot C_F (T_{AD} - T_F) \quad (8)$$

where  $Q_H$  is the amount of heat required (kJ/d),  $Q_{(W+R)}$  the amount of waste fed to the reactor + the amount of digestate recycled to the entrance (kg/d),  $C_F$  the specific sludge heat transfer coefficient,  $T_{AD}$  the operating temperature of the digester which is 55 °C in this study, and  $T_F$  is the temperature of feed sludge (°C).

**2.13.2.5. Heat loss.** The amount of heat required to compensate the heat losses from the digester surface area is given by Eq. (9):

$$Q_L = U \cdot A \cdot (T_{AD} - T_a) \quad (9)$$

where  $Q_L$  is the reactor heat loss (J/s),  $U$  the heat transfer coefficient of the digester wall ( $W/m^2 \cdot ^\circ C$ ),  $A$  the Digester surface area ( $m^2$ ),  $T_{AD}$  the operating temperature of the digester (°C), and  $T_a$  the average ambient temperature outside of digester. For the heat loss due to heat transfer by the digester wall, it is assumed that the average ambient Dutch temperature is 15 °C. Furthermore a reactor wall of 10 cm concrete and 10 cm Styrofoam insulation has a calculated heat transfer coefficient of  $0.39 W/m^2 \cdot ^\circ C$  [50]. The area of the reactor wall is calculated by assuming a length (L) to diameter (D) ratio of 5 for the plug flow reactor.

**2.13.2.6. Electric energy requirement for mixing and pumping.** The electric energy requirement of the proposed anaerobic sludge digester consists of slow turning of agitators inside the digester (as e.g. proposed in the KOMPOGAS plug flow digester (<http://www.axpo.com/kompogas>, Accessed on 30 December 2015), pumping and mixing of the recycle flow with the incoming sludge. The material moves horizontally through the digester before it is discharged. A slowly turning agitator ensures that the digestate is optimally mixed and the biogas is released. The minimum power required for mixing in the anaerobic digester is 5–8  $W/m^3$  of digester volume and may be higher, if friction losses in the heat exchanger are high [51]. To be on the safe side, in this study, we assumed 16  $W/m^3$ . Based on the further-on calculated required digester volume of 52  $m^3$  (Section 3.6), an amount of 20 kW h/d is consumed.

It is also assumed that the return sludge is externally mixed with the feed sludge (FSF) inside a mixing tank with a maximum volume of 20  $m^3$ , having a similar energy consumption for mixing, i.e. 16  $W/m^3$ , resulting in an energy consumption of 7.7 kW h/d. The mixed sludges are conveyed by gravity or pumped into the digester. Energy consumption for pumping can be calculated from Eq. (10):

$$E_P = 24 \cdot \frac{Q_{(R+W)} \cdot \rho \cdot g \cdot h}{(3.6 \cdot 10^6) \cdot \varepsilon_P} \quad (10)$$

where  $Q_{(R+W)}$  is the flow of incoming FSF sludge and returned sludge ( $m^3/d$ ),  $\rho$  the density of sludge ( $kg/m^3$ ),  $g$  the gravity acceleration ( $9.8 m/s^2$ ),  $h$  the differential head (height of reactor, m), the  $\varepsilon_P$  is the pumping efficiency (75%) and  $E_P$  the required pumping energy (kW h/day). The efficiencies for centrifugal pumps normally range between 50% to 85% [49]. Values of 0.45 kW h/d and 0.9 kW h/d have been calculated for pumping the recirculated sludge to the mixing tank and also to pump the sum of incoming feed and returned sludges to the digester, respectively.

**2.13.2.7. Energy consumption for FSF digestate dewatering and sludge drying.** Dewatering technologies vary between plants and the energy consumed is highly variable. A value of 0.11 kW h per kg dried matter was used for dewatering the digested sludge (9% TS) to 20% TS [52]. The energy consumption for drying ( $Q_{drying}$ ) of dewatered sludge was calculated using Eq. (11) [46]. The temperature difference employed in this equation was 90 °C because the collected digested sludge was assumed to be kept at 15 °C after dewatering and were dried at 105 °C.

$$Q_{drying} = M_{ws} \cdot W \cdot [(C_{p_{water}} \cdot \Delta T) + \Delta H_{vap}] + [M_{ws} \cdot (1 - W)] \cdot C_{p_{sludge}} \cdot \Delta T \quad (11)$$

where  $M_{ws}$  is unit mass of wet sludge using a basis of 1 kg,  $W$  the water fraction in the sludge,  $C_{p_{water}}$  the heat capacity of water,  $\Delta T$  the temperature difference between initial temperature of 15 °C and the temperature of drying at 105 °C,  $\Delta H_{vap}$  the latent heat for vaporization of water (2090 kJ/kg) and  $C_{p_{sludge}}$  the heat capacity of solids in sludge (1.95 kJ/kg °C). The energy consumption for drying ( $Q_{drying}$ ) was calculated to be 2008 kJ/kg as an average value.

### 3. Results and discussion

#### 3.1. Biomethane potential (BMP)

The BMP or ultimate methane yield tests giving the maximum amount of mL  $CH_4/gVS_{added}$ , were conducted under mesophilic and thermophilic conditions. Under mesophilic conditions, FSF digestion at  $R_{IJS} = 0.5$  failed (Fig. 1). The high substrate concentrations, reaching 55 gVS/L or 86 gCOD/L, resulted in an imbalance between hydrolysis/acidogenesis on the one hand, and acetogenesis and methanogenesis on the other hand. The batch reactor pH dropped to 5.2, and acetate and propionate accumulated to 8.1 g/L, which equaled to about 78% of the total VFA (10.38 g/L) at the end of this batch test, indicating acidification of the medium (Fig. 2). Based on the COD of acetate and propionate, 1.07 and

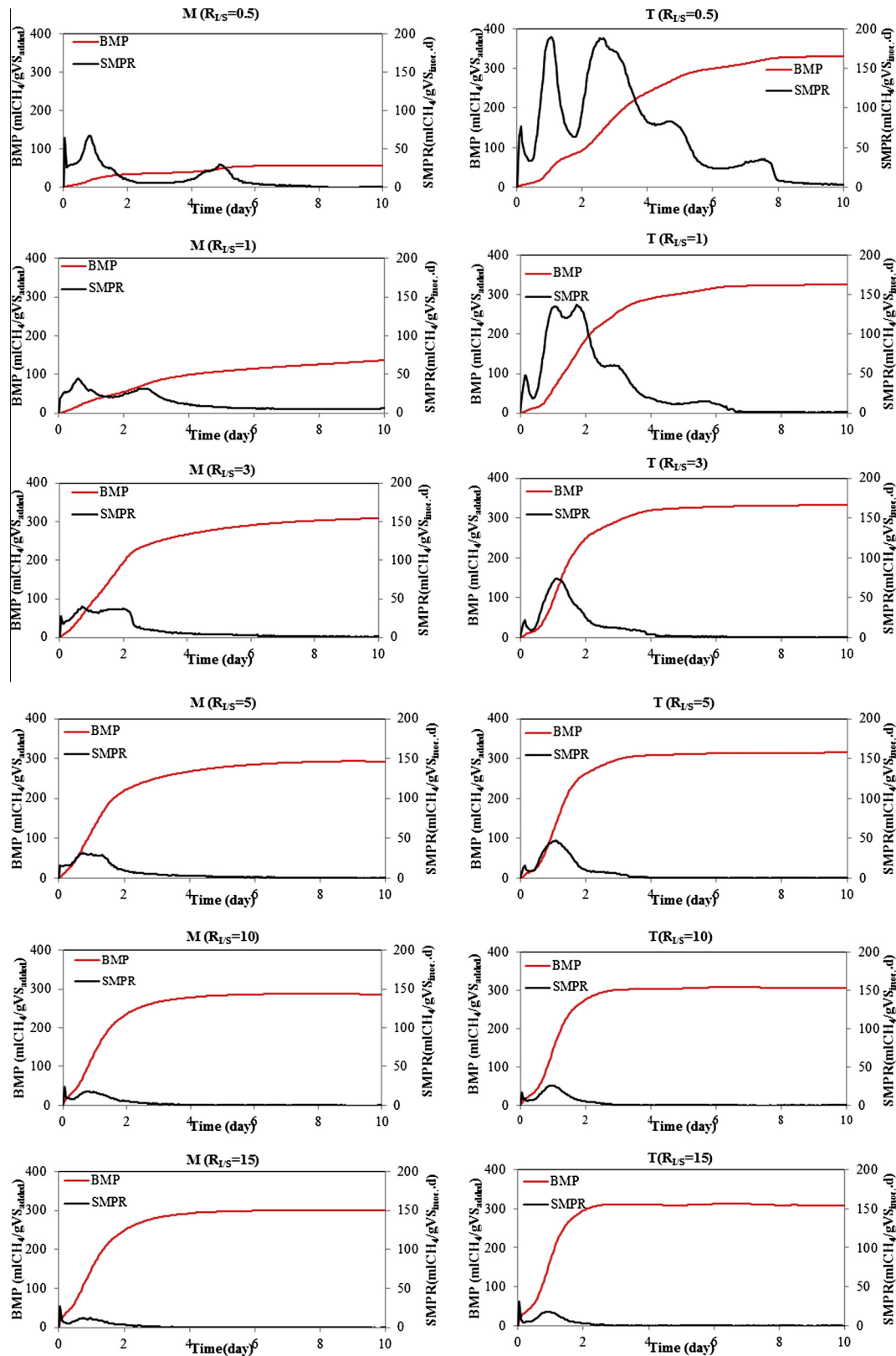


Fig. 1. BMP and SMPR at different  $R_{I/S}$  ratios at the mesophilic (M) (from left, row: 1, 2, 3, 4, 5, 6) and thermophilic (T) (from right, row: 1, 2, 3, 4, 5, 6) conditions (error from average values  $\leq 5\%$ ).

1.51 gCOD/g, respectively, this equals 12% of total influent FSF-COD (86 gCOD/L). An  $R_{I/S} = 1$  at 35 °C resulted in a long lag phase of almost 10 days. At the end of the batch tests only low VFA concentrations remained, whereas at higher  $R_{I/S}$  hardly any VFA could be

detected (Fig. 2, Table 3). Results indicate that mesophilic digestion of FSF at the ratio  $R_{I/S} = 1$  requires digestion times exceeding the 10 days that were standardized at our experiments (Fig. 1). With the increase in  $R_{I/S}$  to 1, 3, 5, 10 and 15, BMP values of 142, 309,

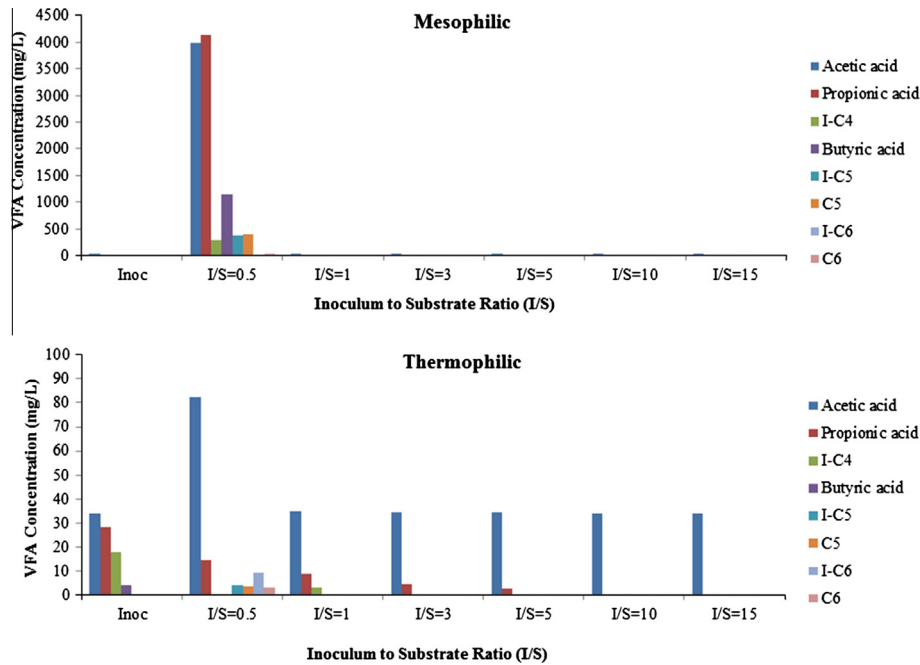


Fig. 2. VFA production at different I/S ratios under mesophilic (top) and thermophilic (bottom) conditions (error from average values  $\leq 5\%$ ); note difference in scale Y axis.

Table 3

Variation of BMP, SMPR<sub>max</sub>, AnBD,  $K_h$ ,  $t_{90\%CH_4}$ , TVFA, DOC and pH with changes in  $R_{I/S}$  at mesophilic (top) and thermophilic (bottom) conditions (error from average values  $\leq 5\%$ ).

$R_{I/S}$	BMP $\frac{mLCH_4}{gVS_{sub}}$	SMPR <sub>max</sub> $\frac{mLCH_4}{gVS_{inoc} \cdot d}$	AnBD (%)	$K_h$ (1/d)	$t_{90\%CH_4}$ (day)	TVFA (g/L)	DOC (mg/L)	pH <sub>end</sub>
<i>Mesophilic conditions (35 °C)</i>								
0.5	56	67	10.4	0.15	5.2	10.38	6045	5.2
1	142	43	26.1	0.25	8.4	0.24	888	7.0
3	309	39	56.7	0.60	5.0	0.05	531	7.0
5	291	31	53.5	0.60	3.8	0.04	499	7.1
10	284	18	52.1	0.55	2.7	0.03	559	7.1
15	297	11	54.5	0.45	2.5	0.05	564	7.2
<i>Thermophilic conditions (55 °C)</i>								
0.5	334	189	61.4	0.40	6.1	0.12	1495	7.4
1	329	134	60.5	0.65	4.4	0.22	1209	7.5
3	338	73	62.1	0.85	3.3	0.18	1046	7.5
5	316	46	58.2	1.20	2.6	0.13	1014	7.5
10	297	25	54.6	1.60	2.0	0.09	987	7.5
15	299	17	55.0	1.80	1.8	0.07	990	7.5

291, 284 and 297  $mLCH_4/gVS_{added}$ , respectively, were obtained (Table 3). Previous studies have shown that decreasing the  $R_{I/S}$ , may have a negative impact on the ultimate methane potential of the substrate [53–55]. In our tests a clear deviation was only found at the ratio  $R_{I/S} = 1$ , while all other observed BMP values were more or less similar. The BMP value at the ratio  $R_{I/S} = 1$  cannot be regarded a BMP value, since the long lag phase, viscosity and possible accumulated intermediates apparently determined the outcome. Diluting the samples might have resulted in higher BMP values for the  $R_{I/S}$  of 1. However, for the application of FSF in a compact highly loaded bioreactor, the values presented in this study are considered most representative.

Under thermophilic conditions, all applied  $R_{I/S}$  ratios resulted in a good degradation of the FSF. Following BMPs were obtained: 334, 329, 338, 316, 297 and 299  $mLCH_4/gVS_{added}$  at  $R_{I/S}$  0.5, 1, 3, 5, 10 and 15 (Table 3) with initial substrate concentrations of 60, 30, 10, 6, 3 and 2  $gVS/L$ , respectively (Table 2). Contrary to the mesophilic conditions, there was only a slight VFA accumulation measured at all ratios (Fig. 2, Table 3), apparently there was neither substrate inhibition nor a pH drop, even at the lowest  $R_{I/S}$  during thermophilic digestion. Our results indicate a better balance

between hydrolysis and acidification and the activity of the methanogens under thermophilic conditions. Furthermore, the SMA tests indicated higher methanogenic activity in the thermophilic digester sludge prior to the BMP experiment, reaching values of  $0.5 \pm 0.05 gCOD-CH_4/gVS d$  compared to an SMA of  $0.2 \pm 0.03 gCOD-CH_4/gVS d$  of the mesophilic sludge. In fact, thermophilic digestion showed higher BMP values than mesophilic digestion under all tested conditions. This indicates that under thermophilic conditions, a higher biogas production per gram substrate can be expected. The fact that the thermophilic digester remained stable even at the lowest  $R_{I/S}$  value means that higher substrate loading rates can be applied under thermophilic conditions compared to mesophilic conditions.

### 3.2. Specific methane production rate (SMPR)

The SMPR varied over time following the batch degradation of the substrate at the different  $R_{I/S}$  under both mesophilic and thermophilic conditions (Fig. 1). At  $R_{I/S} = 0.5$  and 1 under mesophilic and thermophilic conditions, fluctuating methane production rates were recorded over time. The SMPR under thermophilic conditions



was always higher than during mesophilic digestion. The increased SMPR reflects increased hydrolysis rates at higher temperatures, considering SMA was not rate limiting at the lower  $R_{I/S}$  values. The observed fluctuations in SMPR might be caused by different hydrolyses steps in the degradation of FSF, which could be attributed to its heterogeneous nature. In addition, the characteristic drop in the SMPR between days 1.5 and 2 at the low  $R_{I/S}$  of 0.5 and 1, observed at 35 °C and particularly 55 °C, may result from substrate inhibition as experienced by Hashimoto [56] and Raposo et al. [57], or from depletion of readily degradable substrate after 1.5 days and ‘delayed hydrolysis’ of less readily degradable substrates.

The higher the  $R_{I/S}$  the lower the SMPR peaks and the shorter the time interval between the different methane production rates, finally resulting in a stabilized SMPR at the highest  $R_{I/S}$ . Prashanth et al. [58] explained the stable SMPR at high  $R_{I/S}$  by the presence of a large pool of the different required enzymes that are necessary for complete biodegradation of the substrate. The highest observed SMPR values amounted 67 and 189 mLCH<sub>4</sub>/gVS<sub>inoc. d</sub> ( $\approx 0.19$  and 0.54 gCOD/gVS<sub>inoc. d</sub>) for mesophilic and thermophilic conditions, respectively, and were found at the lowest  $R_{I/S} = 0.5$ . Table 2, presents SMPR values under both conditions, showing higher values under thermophilic conditions at all  $R_{I/S}$ .

The net cumulative methane production and the different  $R_{I/S}$  applied, are plotted against the added FSF expressed in gVS, for both mesophilic and thermophilic conditions in Fig. 3. Under mesophilic conditions the relation between methane production and the batch-fed substrate load (in gVS) is linear for  $R_{I/S} \geq 3$ . While under thermophilic conditions, there is a linear relationship ( $R^2 = 0.999$ ) between the net produced methane and gVS added until the lowest  $R_{I/S}$  of 0.5. The slope of the line gives an average methane yield coefficient (BMP) of 333 mLCH<sub>4</sub>/gVS<sub>added</sub>.

### 3.3. Hydrolysis constant ( $K_h$ )

By assuming that hydrolysis is the rate-limiting step, the hydrolysis constant ( $K_h$ ) was calculated using a first order kinetic model as described by Angelidaki et al. [40]. The  $K_h$  has been derived from the slope of the linear regression line plotted for the net accumulated methane production against time for all  $R_{I/S}$  ratios under both mesophilic and thermophilic conditions. Initial observed lag phases were disregarded, which were between 0.5–1 day for all conditions.

The observed apparent  $K_h$  at all  $R_{I/S}$  was higher under thermophilic conditions compared to mesophilic conditions (Table 3). A gradual increase in  $K_h$  was observed with increasing  $R_{I/S}$  ratios under thermophilic conditions, whereas the maximum  $K_h$  was

observed at  $R_{I/S}$  of 3 and 5, for the mesophilic digesters. The observed maximum  $K_h$  was 0.60 (1/d) at  $R_{I/S}$  of 3 and 1.80 (1/d) at  $R_{I/S}$  of 15 under mesophilic and thermophilic conditions, respectively. The  $K_h$  of 0.40 (1/d) of the thermophilic sludge at a high substrate dose ( $R_{I/S}$  of 0.5), is considerable, as at this ratio mesophilic digestion failed due to VFA accumulation. However, considering the possible increased accumulation of VFA intermediates during the first days of FSF digestion at  $R_{I/S}$  of 0.5 and 1 under thermophilic conditions, the calculated  $K_h$  value might be an underestimate of the maximum possible values. A clear inhibition by VFA was shown during mesophilic digestion of FSF at  $R_{I/S}$  of 0.5 (Figs. 1 and 2, Table 3). Therefore, only the obtained  $K_h$  values at  $R_{I/S} \geq 1$  should be used for process evaluation.

### 3.4. Anaerobic biodegradability (AnBD)

The biodegradation efficiency was calculated for both mesophilic and thermophilic conditions at all  $R_{I/S}$  (Table 3). Highest efficiencies were found at an  $R_{I/S}$  of 3 for both conditions, while at all ratios the thermophilic batches revealed the highest efficiency.

Table 3 also shows the required incubation time to achieve 90% of the maximum cumulative methane production ( $t_{90\%CH_4}$ ), which is another factor characterizing the bioavailability of organic matter [59]. Conform expectations, the  $t_{90\%CH_4}$  was shorter under thermophilic conditions compared to mesophilic conditions, likely because of higher metabolic rates at higher temperatures as was also indicated with overall SMPR in the thermophilic batches. The BMP, SMPR, AnBD,  $K_h$ ,  $t_{90\%CH_4}$ , TVFA, DOC and pH values at  $R_{I/S}$  of 0.5 under mesophilic conditions (marked *italic*) indicated digestion failure owing to the high substrate dose. In general, the required incubation period for our BMP experiments was considerably shorter than that described in the conventional BMP methodology (30–50 days) [36,60,61]. Very likely, the use of well-adapted inoculum for FSF digestion [7] resulted in rapid and stable substrate conversion.

### 3.5. Protein matter and humic-like substances

It was hypothesized that the difference in BMP and conversion rates at the two conditions could be due to a different enzyme (protein) production rate by the microorganisms, or due to a change in the bioavailability of organic components such as humic-bound biodegradable compounds. Therefore F-EEM spectroscopy was used to determine differences in protein-like (aromatic and tryptophan-like) and humic-like substances, which are considered the main fluorophores in sludge [62,63]. In this study the observed peaks were identified by comparing their

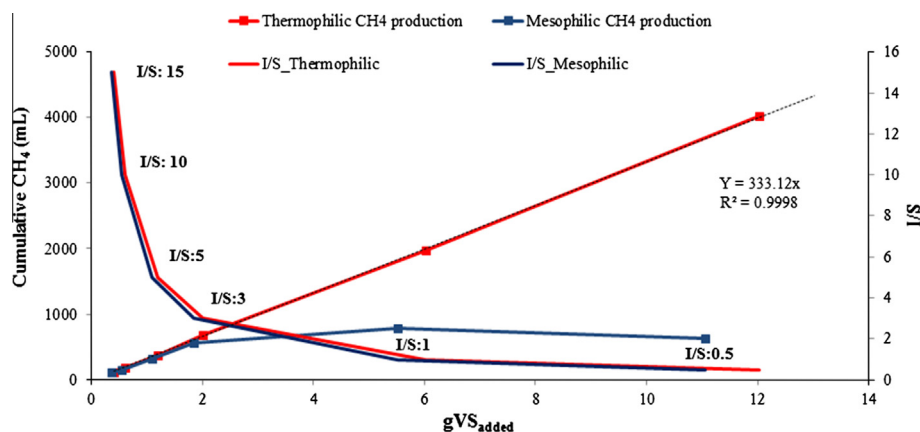
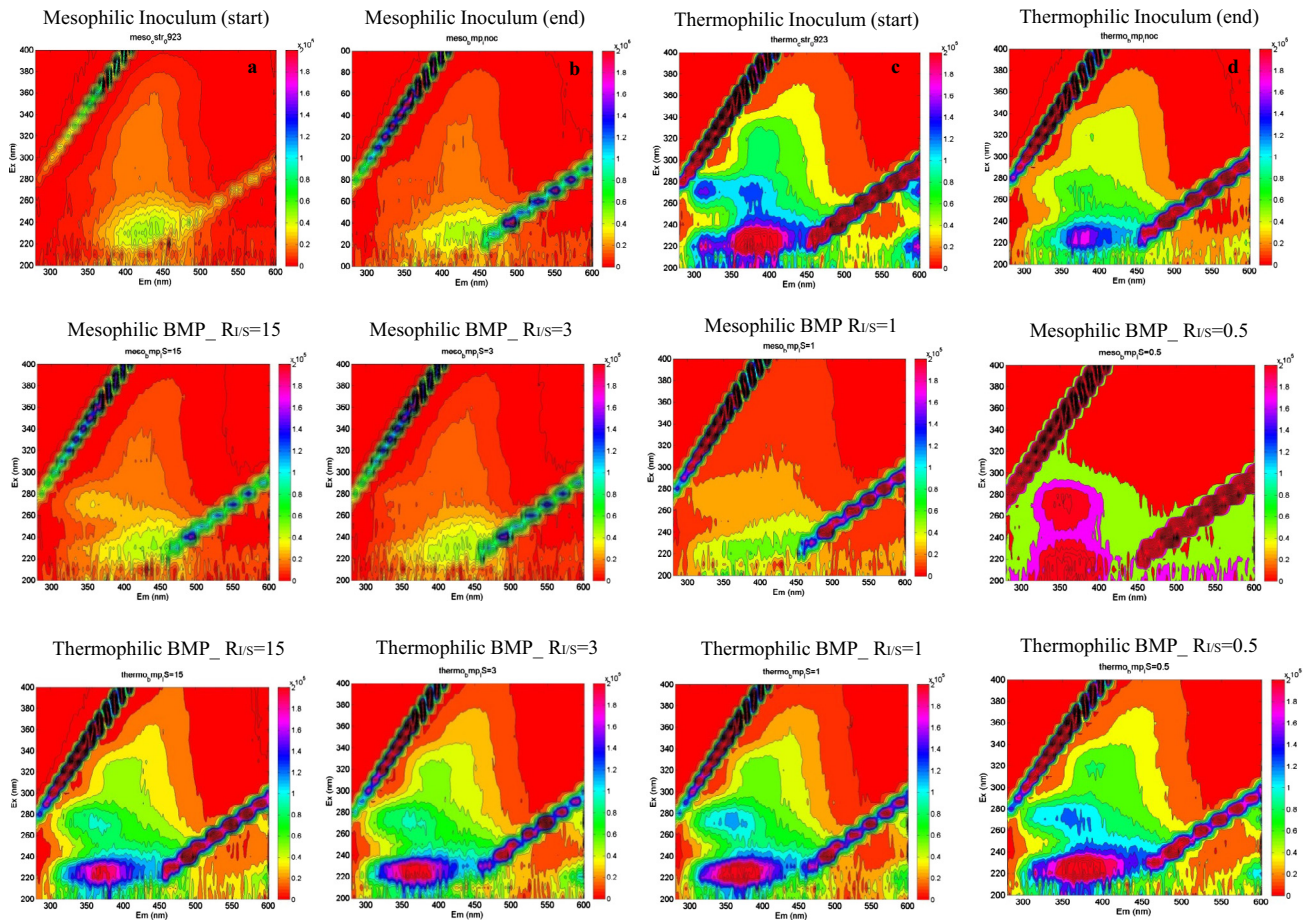


Fig. 3. Net cumulative CH<sub>4</sub> production vs. gVS added per bottle at different I/S ratios (error from average values  $\leq 5\%$ ).



**Fig. 4.** F-EEM images of the mesophilic inoculum at the start and the end of the incubation time (first row, a and b), the thermophilic inoculum at the start and the end (first row, c and d), mesophilic digested FSF at  $R_{I/S} = 15, 3, 1, 0.5$  at the end of digestion (second row, left to right) and thermophilic digested FSF at  $R_{I/S} = 15, 3, 1, 0.5$  at the end of digestion (third row, left to right).

fluorescence properties (excitation/emission (Ex/Em)) with those of pure compounds, such as aromatic protein, tryptophan protein and humic and fulvic acids. The main intensities in four region peaks determined by F-EEM were tryptophan protein-like (Ex/Em = 270–280/320–350 nm), aromatic protein-like (Ex/Em = 220–240/320–350 nm), humic-like (Ex/Em = 330–350/420–480 nm), and fulvic-like (Ex/Em = 250–260/380–480 nm), respectively [62–64].

F-EEM measurements were conducted with the supernatant of fresh mesophilic and thermophilic inoculum (Fig. 4, first row) as well as all  $R_{I/S}$  ratios, at the beginning and end of the experiment, to observe changes in protein and humic-like substances. At both temperatures, measured spectra for  $R_{I/S}$  of 15, 10 and 5 were similar, therefore only F-EEM results of  $R_{I/S} = 15$  are presented. F-EEM spectroscopy revealed the presence of more fluorescent organic matter, especially protein-like substances under thermophilic conditions. It also indicates that humic-like and fulvic-like substances gradually decreased relative to the protein amount, during thermophilic digestion, while during mesophilic digestion hardly any change can be seen. The changes in fluorescence intensity became more apparent during digestion at low  $R_{I/S}$ . The fluorescence intensity of protein-like substances slightly increased with a decrease in  $R_{I/S}$  in the thermophilic digestion (Fig. 4, third row). In contrast, there was no significant formation of protein-like substances observed under mesophilic conditions and the relative intensity of fulvic-like substances was higher except at  $R_{I/S}$  of 0.5, which failed due to VFAs accumulation. Very likely, the high intensity of protein-like substances observed at the ratio  $R_{I/S} = 0.5$  was related

to cell lysis of decaying biomass (Fig. 4, second row). Although not very visible, the fulvic-like substances may also be present in the thermophilic assays, but if so, they are masked by the high intensity of the protein like substances. Results from the F-EEM analysis indicate that protein-like substances are more produced under thermophilic conditions. Considering that these proteins may relate to hydrolytic enzymes than these results agree with the higher activity at thermophilic temperatures.

### 3.6. Energy recovery from municipal raw sewage

#### 3.6.1. Biogas production and electricity recovery by AD of FSF

Energy recovery from raw sewage by using fine sieves coupled to FSF digestion could be a feasible alternative to primary sludge digestion in conventional extended aeration WWTPs, avoiding the construction of large primary clarifiers. The results obtained with the BMP tests were used to quantify the potential energy recovery that could be gained from FSF digestion (Table 4).

For calculating the energy recovery from FSF, the methane production rate per reactor volume ( $\text{m}^3/\text{m}^3 \text{ d}$ ), the lower heating value (LHV) of methane (50 MJ/kg) and its density at standardized temperature and pressure ( $0.716 \text{ kg}/\text{m}^3$ ,  $T = 0^\circ \text{C}$  and  $P = 1 \text{ atm}$ ) as well as the required incubation time to achieve  $t_{90\%}\text{CH}_4$ , were taken into account for each assessed  $R_{I/S}$ . The biogas production rate was calculated based on an average methane composition of 53% and 57% for both thermophilic and mesophilic conditions, respectively [7]. Table 4 presents the normalized methane and biogas production per ton of FSF (wet weight basis at 23%TS) ( $\text{N m}^3/\text{t-FSF}$ ) translated

**Table 4**  
Average energy recovery and electricity generation estimation (error from average values  $\leq 5\%$ ) from mesophilic (top, 35 °C) and thermophilic (bottom, 55 °C) digestion of FSF.

$R_{IJS}$	Methane (N m <sup>3</sup> /t-FSF)	Biogas (N m <sup>3</sup> /t-FSF)	MJ/t-FSF (CHP gross)	kW h/t-FSF (Eff. 40%)	Methane (m <sup>3</sup> /m <sup>3</sup> d)	Biogas (m <sup>3</sup> /m <sup>3</sup> d)
<i>Mesophilic conditions (35 °C)</i>						
0.5	12	22	444	49	0.6	1.1 (Failed)
1	31	55	1121	125	0.5	0.8
3	68	119	2430	270	0.6	1.0
5	64	112	2293	255	0.4	0.7
10	62	110	2236	248	0.3	0.5
15	65	115	2337	260	0.2	0.4
<i>Thermophilic conditions (55 °C)</i>						
0.5	74	139	2635	293	3.3	6.2
1	72	137	2595	288	2.2	4.2
3	74	140	2664	296	1.0	1.9
5	70	131	2495	277	0.7	1.4
10	65	124	2343	260	0.4	0.8
15	66	124	2360	262	0.3	0.6

**Table 5**  
Operational conditions and biogas production from mesophilic and thermophilic digestion of FSF.

Parameters	BMP results (Mesophilic)	BMP results (Thermophilic)
Operating temperature (°C)	35 ± 1	55 ± 1
Dry matter (%)	23	23
pH (after BMP)	7.0 ± 0.1	7.4 ± 0.1
Retention time (day)	2.5–5 ( $R_{IJS} = 3$ –15)	1.8–6.1 ( $R_{IJS} = 0.5$ –15)
Gas yield (N m <sup>3</sup> /ton)	119 ( $R_{IJS} = 3$ )	139–140 ( $R_{IJS} = 0.5$ –3)
Maximum gas production rate (m <sup>3</sup> /m <sup>3</sup> reactor day)	1 ( $R_{IJS} = 3$ )	6.2 ( $R_{IJS} = 0.5$ )
Organic loading	9 kgVS/m <sup>3</sup> or 14.4 kgCOD/m <sup>3</sup> ( $R_{IJS} = 3$ ) (58% conversion)	60 kgVS/m <sup>3</sup> or 93.5 kgCOD/m <sup>3</sup> ( $R_{IJS} = 0.5$ ) (61% conversion)

to energy production as heat (MJ/t-FSF) and electricity generation (kW h), taking an electric and heat conversion efficiency of 40% and 50% using a modern combined heat and power (CHP) unit, heat losses are about 10% [3,51,65]. Furthermore, the rate of normalized methane and biogas production per reactor (working) volume (m<sup>3</sup>/m<sup>3</sup> d) were calculated.

As shown in Table 4, thermophilic digestion of FSF presents higher values for all parameters compared to the mesophilic conditions. Maximum values of biogas production rates were 1 and 6.2 m<sup>3</sup>/m<sup>3</sup> d for mesophilic and thermophilic FSF digestion at  $R_{IJS}$  ratio of 3 and 0.5, respectively. Typical values obtained for mesophilic sludge digestion at WWTPs are in the range of 0.5–1.0 m<sup>3</sup>/m<sup>3</sup> d [4]. Mesophilic digestion under high substrate loading ( $R_{IJS} = 0.5$ ) was shown to be impossible due to VFA accumulation and mixing problems owing to higher viscosity of the reactor broth [11].

Semi-dry or dry thermophilic digestion using plug flow reactors could be of interest for FSF digestion, since dewatering (mechanically pressing) of FSF to 40–50% dry solids is possible [8]. Batch and plug flow reactors also have a significant potential to produce biogas with low capital costs and high efficiencies [66]. Based on the batch tests results (Table 3) the applicable  $R_{IJS}$  ratio for FSF digestion in such reactor could be as low as 1–0.5 for thermophilic conditions. Substrate loading rates of 60 kgVS/m<sup>3</sup> or 93.5 kgCOD/m<sup>3</sup> could be feasible based on the relatively short retention times needed for 90% conversion (3–6 days, Tables 4 and 5). Such operational conditions would result in a very small and compact thermophilic reactor design. Especially when compared to the conventional anaerobic digestion systems treating sludge from wastewater with maximum dry solids content of 9% and a long retention time (12–30 days). Therefore, the possible on-site energy recovery with thermophilic digestion of FSF was evaluated for the situation at WWTP Blaricum, the Netherlands. The average data used for this WWTP were: a dry weather flow (DWF) of 8000 m<sup>3</sup>/d; influent wastewater COD of 424 mg/L; FSF COD of 342 g/kg at TS

of 23% dry solids, and 35% COD removal efficiency by fine sieving [8].

The total mass flow of COD and flow rate of FSF that would enter the plug flow digester were calculated at 1187 kgCOD/d and 3.47 m<sup>3</sup>/d, respectively. This would result in a design based on 15 days SRT and thus a flow rate of FSF of 3.47 m<sup>3</sup>/d. As a result, the volume of the digester would become 52 m<sup>3</sup> (e.g. L:D = 5; L = 11.85 m and D = 2.35 m). During the batch experiments, an FSF biodegradability of 61% was found, leading to a calculated methane production of 255.5 m<sup>3</sup>/d with the proposed digester. The biogas production rate would become 9.3 m<sup>3</sup>/m<sup>3</sup> d (53% CH<sub>4</sub>) under an OLR of 22.8 kgCOD/m<sup>3</sup> d.

The estimated minimal recirculation rate for the thermophilic digestion of FSF would be approximately 50% with  $C_T$  of 7.5 days ( $Q_R = 3.47$  m<sup>3</sup>/d and  $R = 1$ ). Using higher recirculation rates could result in incomplete substrate digestion, leading to a lower methane production per kg of FSF. It is recommended to study this assumption in a continuously operated pilot experiment. If such pilot digester could be operated with even shorter retention times or lower recycle ratios as were derived from the batch tests ( $R_{IJS} = 0.5$ ), overall dimensions could be significantly reduced.

**Table 6**  
Overall energy balance on FSF digestion in the plug flow digester.

Parameter	Unit	Value
CHP gross energy (heat and electricity)	MJ/d	9146
CHP gross electrical output (40% efficiency)	kW h/d	1016
Electrical requirement of digester	kW h/d	–29
Net energy output as electricity	kW h/d	987
CHP gross heat output (50% efficiency)	MJ/d	4573
Heat requirement of digester	MJ/d	–445
Net energy output as heat	MJ/d	4128
Lost CHP energy (10%)	MJ/d	–915

**Table 7**  
Total electricity consumption at WWTP Blaricum [52] and integration of FSF and anaerobic digestion (FSFAD) as proposed in this study.

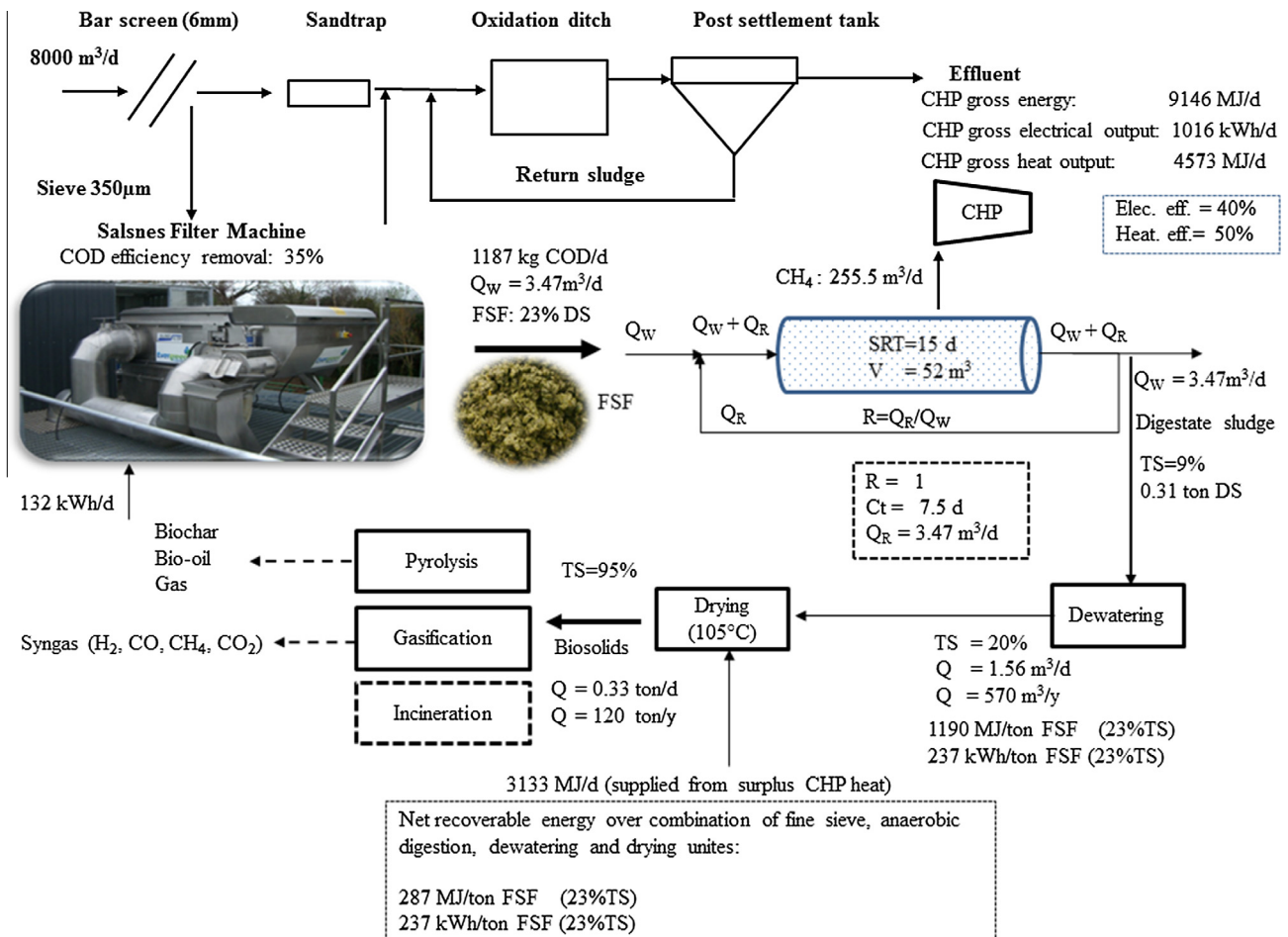
Components	Unit	Reference	Fine sieve	FSFAD
Fine sieving	kW h/d	0	132	132
Aeration	kW h/d	1655	1159	1159
Other consumptions	kW h/d	1340	1307	1307
Digestion of FSF	kW h/d	0	0	-987
Total	kW h/d	2995	2598	1611

The calculated overall daily energy balance of FSF digestion in the proposed plug flow digester is presented in Table 6. Calculations were based on Eq. (7)–(10) as well as suggested values to calculate, for instance, the mixing energy, efficiency of pumps and heat exchanger. The heat requirement of the digester (MJ/d) is composed of the total heat required to heat the incoming FSF and the heat loss from the digester surface areas. Electricity consumption (kW h/d) for mixing and pumping is presented as electrical requirement of digester in Table 6.

The total electricity consumption at WWTP Blaricum for the situations without fine sieve (reference), with fine sieve as well as combination of fine sieving and anaerobic digestion of FSF are presented in Table 7. It was estimated that the installation of a fine sieve would reduce the aeration energy in the WWTP Blaricum by 30% [8]. The total electricity consumption of the WWTP including the fine sieve, aerators and other consumptions (Table 7) were obtained from previous studies [52].

The total electricity consumption at the WWTP Blaricum could be reduced with about 13% by the use of a FSF, without digestion of the FSF. The net electricity production from the anaerobic digestion of the FSF in combination with the reduced energy consumption by installing a fine sieve, would lead to a total of 46% lower (fossil) energy consumption at the WWTP Blaricum. In this calculation the reduction of sludge volume to be transported from WWTP Blaricum to the incineration plant (approximately 45 km) is not taken into account.

At WWTP Blaricum the activated sludge is only thickened upon transport. Therefore energy requirement for excess activated sludge dewatering is not included in this balance. However, removal of FSF from the sludge matrix might impact the energy consumption for dewatering. STOWA (The Foundation for Applied Water Research) in the Netherlands [52], reported an energy consumption of 108 kW h/d and 49 kW h/d for secondary sludge dewatering to 20% TS in absence of fine sieve (reference) and with one fine sieve, respectively. Different values have been reported on WWTP electricity consumption (kW h/m<sup>3</sup> of wastewater treated) showing the high variety among different facilities and countries [67]. Average values reported varied from 0.30 to 0.78 kW h/m<sup>3</sup> [67,68]. For WWTP Blaricum 0.37 kW h/m<sup>3</sup>, 0.32 kW h/m<sup>3</sup> and 0.20 kW h/m<sup>3</sup> were calculated for the reference, fine sieve and FSFAD respectively. These numbers excluded secondary sludge treatment that results in energy consumption for dewatering that might be off-set when the sludge is digested.



**Fig. 5.** Schematic view of WWTP Blaricum combined with fine sieve, plug flow digester, CHP, dewatering and drying units and final destination of biosolids. Figure also contains further details about the mass flow rate of FSF, volume of digester, gross energy (heat and electricity) and net recoverable energy per ton of FSF at 23% TS.

### 3.6.2. Heat production by AD of FSF

Heat production of the CHP exceeds the amount of heat required to heat up the mixed sludges (incoming FSF sludge and returned sludge) to thermophilic conditions and the heat loss from digester surface area. For WWTP Blaricum, the net heat production was calculated for the proposed thermophilic digester according to Eqs. (7)–(9). The extra available heat production from FSF digestion (4128 MJ/d, Table 6) by the CHP unit can be utilized at the WWTP facility or its surroundings for e.g. heating water and buildings. In addition, also the heat requirement to maintain the mixing tank temperature where returned sludge is mixed with inflow FSF sludge can be covered. Currently, WWTP Blaricum consumes, on average, 26 GJ/y net heat energy (71 MJ/d) which can be supplied from the heat recovery of the CHP.

To reduce the volume of sludge to be transported and/or to create a possibility for more advanced technology for digestate processing, excess heat can be used for sludge drying [46,69]. The required amount of heat to dry the flow of dewatered digestate (1.56 m<sup>3</sup>/d) at 20% dry solids content at 15 °C (assumed minimum temperature after dewatering) to 95% dry solids content at 105 °C, was calculated to be 3133 MJ/d using Eq. (11). This amounts equals about 76% of the net heat production (4128 MJ/d) by the CHP.

By applying a drying process, the weight of transported biosolids to the gasification or pyrolysis plant amounts 120 ton/y, which is considerably lower compared to the amount of undigested FSF (1267 ton/y at TS ≈ 23%) (Fig. 5). Current practice for utilization of FSF from WWTP Blaricum is transporting FSF at 23% TS to waste incineration plant (AEB) in Amsterdam (distance approx. 45 km) to generate heat and electricity off-site.

Since costs for transportation, dewatering and incineration approximately amounts 60–100 euros per ton of solids cake in the Netherlands [70], therefore, the final cost of sludge treatment could be reduced over 76,000–127,000 euros per year. In the case of combining fine sieving and digestion, on-site dewatering of the digestate and drying the dewatered digestate, the costs of fuel consumption for 120 ton/y biosolids could reach €150/year (4277 MJ/year) if the biosolids are transported to gasification or pyrolysis plant (assumed the same distance as incineration plant). By this means, transport fuel for 38 trucks (30 tonnes) per year can be saved as well. In the calculation of fuel consumption cost, the capacity of a trucks (30 tonnes or 30 m<sup>3</sup>), diesel fuel consumption (0.33 L/km), net heating value of diesel fuel (36 MJ/L), approximate distance of transportation (45 km), and current price of diesel fuel in the Netherlands (€1.27/L) were taken into account.

Summarizing the overall energy (heat and electricity) use in a combination of fine sieving (TS of the FSF 23%), anaerobic digestion of FSF, dewatering of digestate sludge and drying of the dewatered digestate sludge, a net energy recovery can be calculated. Fine sieving (132 kW h/d), anaerobic digestion (29 kW h/d and 445 MJ/d), digestate dewatering from 9% to 20% TS (34 kW h/d or 0.11 kW h per kg dry solids) and sludge drying (3133 MJ/d), leads to a net recoverable energy of 287 MJ/ton FSF and 237 kW h electric/ton FSF.

## 4. Other routes for energy recovery

In recent years, several valorisation routes have been practiced to find a more sustainable use of digestate sludge, such as the potential use of solids digestate as solid fuel [71] and production of bioethanol [72]. These routes are more promising by means of energy and resource recovery than the conventional management and valorisation routes like landfilling, composting and incineration. Currently, there is growing attention towards the application of thermal processing techniques such as pyrolysis and gasification to treat sewage sludge [46,73–75] or coupling anaerobic digestion

to pyrolysis [76] or gasification processes to generate syngas (H<sub>2</sub>, CO, CH<sub>4</sub>, CO<sub>2</sub>) [77].

Monlau et al. [76] investigated the feasibility of combining anaerobic digestion and pyrolysis processes in order to increase the energy recovery from agricultural residues, which could be applicable to FSF too. It was reported that excess heat production from CHP could cover the drying needs for the solid fraction of the digestate, whereas pyrolysis of this fraction at 500 °C resulted in 8.8 wt.%, 58.4 wt.% and 32.8 wt.% of syngas, oil and char, respectively. The LHV of syngas was 15.7 MJ/N m<sup>3</sup>, whereas pyrolysis oil exhibited a higher heating value (HHV) of 23.5 MJ/kg after water extraction. Integrating these two processes and by using the heat production for sludge drying, could increase the production of electricity by 42% compared to anaerobic digestion as stand-alone plant [76].

Since digestion of FSF does not need any pre-treatment process and dewatering of FSF to 40–50% dry solids is simply possible [8], it is speculated that a very compact high loaded system can be applied for semi-dry or dry thermophilic digestion of FSF in plug flow reactors. Moreover, it is predicated that coupling anaerobic digestion and gasification/pyrolysis systems, considering the economic analysis, legislation and incentives, would further increase the on-site energy recovery approaching to the level of energy neutrality or even energy positive STPs.

## 5. Conclusions

The outcomes of this study revealed promising biogas production rates from FSF digestion under low  $R_{f/S}$ , which were translated to a design of a compact thermophilic plug flow digester with high OLR. It was calculated that 46% reduction in electricity use could be reached when on-site digestion of FSF at WWTP Blaricum would be applied. Surplus heat production from the CHP would be enough to dry the digestate before transport to the final utilization unit. The net recoverable energy from FSF (23% TS) was estimated at 287 MJ/ton FSF and 237 kW h/ton FSF.

Based on the results of the batch tests, it can be concluded that thermophilic adapted biomass is more appropriate for FSF degradation than mesophilic adapted sludge. Higher SMA, BMP, SMPR,  $K_h$  and AnBD values were found under thermophilic conditions for all  $R_{f/S}$  ratios compared to mesophilic digestion. Physicochemical analysis of the reactor broth showed that protein-like substances were present in higher concentrations under thermophilic conditions than under mesophilic conditions at all applied  $R_{f/S}$ , indicating an increased amount of enzymes and thus higher substrate conversion rates at high temperatures.

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