#### 1 The influence of temperature and SRT on high-solid digestion of municipal sewage 2 sludge

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8 Keywords ammonia inhibition, high-solid concentration, mesophilic digestion, methane production, removal 9 sludge retention time, temperature

10

#### 11 ABSTRACT

12 The influence of temperature and SRT on high-solid digestion of municipal sewage sludge 13 was investigated in laboratory scale reactors. Digestion with high-solid concentration reduces 14 the required digestion volume and is advantageous for urban areas. The experimental 15 conditions comprised TSS in digested sludge between 4.0 and 4.6 %, temperatures in a range 16 of 33 to 41 °C and the SRT between 10 and 25 d. High-solid digestion operates with increased NH<sub>4</sub>-N concentrations released from organic compounds. The anaerobic process can be 17 18 limited by high NH<sub>4</sub>-N concentration and toxic NH<sub>3</sub>. In this study a stable digestion was observed up to 2,000 mg L<sup>-1</sup> NH<sub>4</sub>-N and 75 mg L<sup>-1</sup> NH<sub>3</sub>. VSS and COD removal was 53 % 19 20 respectively 57 %. However, digestion with 10 d SRT led to a declined VSS removal of 49 %. 21 The removal at 41 and 37 °C showed minor differences, while reduced NH<sub>4</sub>-N release and methane production were observed at 33 °C. For economic reasons, high-solid digestion at 22 23 41 °C is not recommended, but will not impair VSS removal. The outcomes of this study 24 confirm that digestion with up to 7.8 % TSS in the feed is feasible for the tested temperatures 25 and SRT down to 15 d.

#### 26 **INTRODUCTION**

27 Anaerobic digestion is one of the major technologies for the stabilization of sewage sludge of 28 wastewater treatment plants (WWTPs). With a trend to more energy efficient wastewater 29 treatment and the desire to reduce GHG emissions, there is a strong development towards 30 anaerobic digestion for sewage sludge worldwide. Hereby the disposal and energy costs are 31 minimized by converting organic compounds into energy-rich methane gas and as a result 32 stabilize the sludge. Typically, wastewater treatment defines SRT as solids retention time. The 33 required digestion volume results from the raw sludge volume and SRT. Sludge thickening 34 before digestion is used to remove water and to ensure smaller sludge streams. As a result, a 35 reduced digestion volume and physical footprint are possible. Another benefit of thickening 36 can be seen in a reduced energy demand for sludge heating, as less water is required to be

- 37 heated.
- 38 High-solid digestion is characterized by 3 to 4 % TSS in the digested sludge, which results
- 39 from an increased solid content in the feed. There is limited information in literature about
- 40 optimum operating conditions for high-solid digester that can be used to inform the design.
- Most reports of high-solid digestion relate to the treatment of municipal organic waste streams 41
- 42 (Kayhanian 1994; Guendouz et al. 2008; Le Hyaric et al. 2011), whereas only few studies

exist for the high-solid digestion of municipal sewage sludge (Kapp 1984; Lay *et al.* 1997;
Fujishima *et al.* 2000; Duan *et al.* 2012). Because of the lack of information and for safety
reasons, the digesters of WWTP are often operated only with solid contents in a range of 2 to
3 %. Compared to well described studies about the digestion with lower TSS, this study focus
only on experiments with TSS in the digested sludge of about 4 %.

48 High-solid digestion shows a number of advantages especially for the economical sludge 49 treatment in urban areas. Besides an increased polymer demand for sludge thickening, it is 50 attractive for WWTPs that have a need to increase capacity and are constrained by space. However, high-solid sludge has different rheological flow characteristics compared to low 51 52 solid sludge. For this reason, the digester design requires adjusted hydraulic pipe design. 53 Moreover, the high-solid content in the digested sludge leads to increased NH4N 54 concentrations in the dewatered effluent. The returned nutrient load can be treated separate in 55 tanks or combined in the biological stage. No matter how, the treatment of turbid water lead 56 to enhanced aeration and energy demand. To reach an economic operation of high-solid 57 digestion, other costs must be kept low, especially increased disposal costs caused by a 58 potentially decreased sludge degradation. While a reduced methane production impairs the 59 energy recovery, a decreased removal can cause a declined dewaterability of digested sludge 60 and increased polymer demand or disposal costs. The influence of individual factors on the 61 degradability are described below.

62 An important parameter for the anaerobic treatment is the SRT (eq. hydraulic retention time). 63 HRT is the time the water is in the digester, so it is not necessarily equivalent to SRT. It is 64 known, that with longer SRT a wider range of substrates, especially slowly degradable 65 carbohydrates can be hydrolyzed to easy available compounds resulting in high removal efficiency (Young et al. 2013). Kapp (1984) reported an increased VSS removal of high-solid 66 sludge at 30 d SRT compared to 15 d. The German design recommendations for digesters 67 68 name a minimum SRT of 15 d (DWA 2014). However, most digesters on WWTPs are 69 operated with SRTs of 25 d. SRT needs to be reduced in the cases of treating additional 70 organic waste streams (co-digestion), demographic increase or disturbances (decommissioning digester). Some authors report that a sufficient removal is possible also 71 72 with 10 d SRT (Nges & Liu 2010; Lee et al. 2011). Nevertheless, short SRT can decrease 73 VSS removal and dewaterability. This in turn increase sludge disposal costs and shows the 74 importance of a sufficient degradation (Skinner et al. 2015).

75 Another relevant operation parameter is the temperature. Mostly, mesophilic temperatures are 76 applied; although thermophilic digestion processes reach a higher reduction of organic 77 compounds, it is known to be more cost intensive. Additionally, thermophilic bacteria prefer a 78 narrow temperature range, which makes the process unsuitable for temperature shifts. 79 International textbooks name design temperatures for mesophilic fermenters in a range of 30 80 to 38 °C (Metcalf & Eddy 2004). For the mesophilic operation it is reported that the microbial community adapt on their environment when temperature was changed slowly. However, 81 82 until now it is not clear which effect temperature shifts will have for a digestion with highsolids, when more ammonium (NH4-N) is released compared to digestion with low solid 83 84 concentrations.

85 While operational settings like temperature or SRT result mainly from the design basis, the 86 NH<sub>4</sub>-N release relates mainly to the breakdown of protein containing compounds and is 87 adjustable only by controlling the solid concentration of the feed. Hence, NH<sub>4</sub>-N in digested 88 sludge correlates with the amount of supplied raw sludge and its solid concentration. It is 89 known that high NH<sub>4</sub>-N concentration can inhibit especially methanogenic bacteria (Hansen et al. 1998; Sung & Liu 2003; Rajagopal et al. 2013; Yenigün & Demirel 2013). Wiegant and 90 91 Zeeman (1986) as well as Angelidaki and Ahring (1993) found that acetoclastic methanogenic 92 are more resistant to high ammonia concentration, while hydrogenotrophic methanogenic 93 bacteria do not or only slowly adapt on increased concentration. Hydrogenotrophic 94 methanogenic bacteria are essential to keep hydrogen concentration low and thus to enable the 95 conversion of propionic into acetic acid and hydrogen (Fujishima et al. 2000). A negative impact on the digestion of sewage sludge was reported above 2,000 mg L<sup>-1</sup> NH<sub>4</sub>-N (Duan et 96 97 al. 2012; Hidaka et al. 2013). At the same time increased temperatures and pH strengthen the 98 inhibitory effect by forming toxic ammonia (NH<sub>3</sub>). Hence, the inhibition of high-solid 99 digestion by NH<sub>3</sub> is a result of the combination of NH<sub>4</sub>-N concentration, temperature and pH. 100 Lay et al. (1997) investigated the influence of moisture content and pH on the methane 101 production in high-solid fermentation. The authors observed at moisture contents of 90 to 102 96 % a pH range between 6.6 and 7.8. Optimally results appeared at pH 6.8.

103 Due to the known negative influence of high NH<sub>4</sub>-N concentration to anaerobic bacteria, it 104 has to be considered to what extent NH<sub>4</sub>-N concentration result from the digestion of a high-105 solid feed and whether there is an impact on biodegradation or methane production. Particular 106 attention was therefore paid to released NH<sub>4</sub>-N and NH<sub>3</sub>. In this study the influence of 107 temperatures in a range of 33 to 41 °C and SRT of 10 to 25 d on high-solid digestion were 108 investigated to characterize the process stability in view of a large scale treatment. The 109 investigated temperatures cover the often named optimum of 37 °C for mesophilic operation 110 and temperatures with a trend to psychrophilic and thermophilic conditions. SRT was selected to compare the performance of values usually applied for digesters on WWTPs with shorter 111 112 SRT to find the optimal operation. Moreover, the removal of volatile suspended solids (VSS) 113 and chemical oxygen demand (COD) as well as the specific methane production according to 114 the tested conditions were evaluated. The aim of this study was to test different stress 115 conditions and to describe the expected impacts on the anaerobic conversion with higher solid 116 content. Acid concentrations were therefore used as tool to evaluate process stability.

# 117 MATERIAL AND METHODS

The experimental set-up consisted of three identical reactors with a volume of 3.0 liters. The reactors were inoculated with digested sludge from a municipal WWTP, digesting sewage sludge at low solid concentration under mesophilic conditions. All reactors were fed semicontinuously, once per day on five days per week with raw sludge. The raw sludge was taken from a static thickener of a WWTP. There, excess and primary sludge were thickened at once, while excess sludge is often thickened separate with mechanical techniques.

A heated water bath was used to control different temperatures. Temperature and pH were controlled every day by using a WTW SenTix20 pH meter. The dosage of chemicals to set optimal pH was not necessary. A stirring system ensured continuous sludge mixing. The reactors were directly attached to drum gas meter to measure the gas flow (Ritter, type

Bochum Langendreer 11066). Gas was analyzed by a GFM 400 gas analyzer to determine the methane content. Sludge samples were mainly analyzed for COD, total suspended solids (TSS), VSS, total kjeldahl nitrogen (TKN) and by standard methods (NH<sub>4</sub>-N and orthophosphate). Organic acids were measured by an isocratic high performance liquid chromatographic (HPIEC) method including the separation and quantitative analysis of organic acids. Organic acids were summarized as concentrations of formic, butyric, valeric, acetic, propionic and lactic acids.

135 The experiments were divided into five periods. Table 1 summarizes the applied temperatures and SRTs. After changing temperature or SRT, the reactors were allowed to stabilize for more 136 137 than one SRT before the evaluations were made for the individual periods. For each phase the 138 organic loading rate (OLR) and SRT were identical for all reactors. Raw sludge and digested 139 sludge samples were analyzed twice a week. The data were used to calculate the COD and VSS removal, specific methane production and nitrogen release. The measurements of every 140 141 period were controlled by mass balances for COD and total phosphorus (TP). Analytical data 142 of the raw sludge were compared to identify annual fluctuations in sludge quality. The ratio of 143 primary and excess sludge defines the biodegradability and gas production. Hence, slight 144 variations in the degradability of raw sewage sludge over longer periods are usual due to 145 variations in the sludge composition.

146 The TSS of the feed sludge varied between 6.7-7.8 %. Sludge samples were moreover 147 compared in terms of the nitrogen load; especially TKN and NH<sub>4</sub>-N. In the first experiments in raw sludge, mean NH<sub>4</sub>-N concentrations of about 700 mg L<sup>-1</sup> were observed, while in the 148 149 following periods the mean NH<sub>4</sub>-N concentrations were lower with 250 to 550 mg  $L^{-1}$ . 150 Moreover; a high ratio of TKN to VSS of about 7.3 was determined during the first period. 151 The ratio of TKN to VSS decreased during period 2 to 5 and was then found between 5.8 and 152 6.7. The differences can be explained by increased environmental temperatures during period 1, which took place in the summer period of June to August. Static thickening of raw sludge 153 154 with high environmental temperatures or longer storage time can favour the hydrolysis of 155 proteinaceous compounds and subsequently the total removal. Additionally; the proportion of 156 VSS and TSS of raw sludge was calculated. During the first experiments the ratio was about 157 1.96. A further hydrolysis of organic compounds caused by higher temperature is hereby 158 combined with decreasing VSS concentrations, which finally leads to a high ratio of 159 TSS/VSS. In the following months the temperatures were colder, resulting in a slight decreased ratio of TSS/VSS to finally 1.71. 160

Item		Unit	period 1	period 2	period 3	period 4	period 5
	n*	[-]	10	5	7	9	6
adjustment	t <sub>R1,R2,R3</sub>	[°C]	39;37;35	41;37;33	41;37;33	41;37;33	41;37;33
	SRT	[d]	25	25	20	15	10
	TSS	[%]	6.7±1.1	6.8±0.2	7.0±0.4	7.1±0.5	7.6±0.6
mean sludge composition	TSS/VSS	[-]	$1.96 \pm 0.1$	$1.81\pm0.1$	$1.75\pm0.1$	$1.74\pm0.1$	1.71±0.1
	COD/VSS	[-]	$1.34\pm0.0$	$1.27\pm0.0$	$1.25\pm0.0$	$1.22\pm0.0$	1.25±0.1
	TKN/VSS	[%]	7.32±0.5	$6.68\pm0.4$	$5.78 \pm 2.4$	$5.75 \pm 2.0$	$6.52 \pm 0.5$
	org. N	[g L <sup>-1</sup> ]	3.20±0.8	2.99±0.2	3.49±0.4	3.30±0.3	3.59±0.2
	NH <sub>4</sub> -N	[mg L <sup>-1</sup> ]	716±388	550±240	245±134	365±180	365±170

161 Table 1: Operation adjustments and mean analytical data of used raw sludge

162 \* number of measurements

### 163 **RESULTS AND DISCUSSION**

## 164 1. NH<sub>4</sub>-N concentration

165 An essential point of evaluation concerns the production of NH<sub>4</sub>-N within the digested sludge. Figure 1 depicts the NH<sub>4</sub>-N concentration of raw sludge and digested sludge samples. NH<sub>4</sub>-N 166 concentrations peaked up to 2,000 mg  $L^{-1}$  especially during the first phase. This was mainly 167 168 caused by increased NH<sub>4</sub>-N concentration in the feed. A reason for this is the increased 169 hydrolysis during thickening due to increased temperatures during the months June to August. 170 In the following two periods, NH<sub>4</sub>-N in digested sludge stabilised at 1,600 and 1,860 mg  $L^{-1}$ due to lower concentrations in the feed. However, the TKN load remained almost the same, 171 172 which represented a constant total nitrogen load. Moreover, the lowest concentrations in the 173 input were found during period 3 due to low temperatures of the months November and December. The findings of this study demonstrate that the NH<sub>4</sub>-N concentrations were mainly 174 175 affected by the degree of hydrolysis and not by TSS concentration, since the TSS in digested sludge was almost stable around 40 g  $L^{-1}$  (38.0 to 46.0 g  $L^{-1}$ ). However, this observation refers 176 to TSS in the feed of 6.7-7.8 %. Thus, NH<sub>4</sub>-N concertation of 2,000 mg L<sup>-1</sup> can naturally 177 occur by treating sewage sludge with higher TSS and an increased hydrolysis during pre-178 179 treatment.



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Figure 1: Mean NH<sub>4</sub>-N and TKN concentration of raw sludge and NH<sub>4</sub>-N concentration of digested sludge samples

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184 2. NH<sub>3</sub> concentration

Table 2 summarizes the main conditions resulting for the applied temperatures and SRTs in the three reactors as average values.  $NH_3$  was calculated based on equations from Hobiger (1996). Taking into account that  $NH_3$  formation relates to temperatures; the highest concentrations occurred still at 41 °C. However,  $NH_3$  was all time below than 75 mg L<sup>-1</sup>. Duan *et al.* (2012) reported a moderate inhibition at mesophilic temperatures by free ammonia

concentrations of 400 mg L<sup>-1</sup>. Sung and Liu (2003) found a reduced methane production of 190 39 % and 64 % by total ammonia concentrations of 4,920 and 5,077 mg L<sup>-1</sup>. The comparison 191 192 of NH<sub>4</sub>-N and NH<sub>3</sub> to references from literature indicates that there were no critical values 193 during the experiments which would cause inhibition of organisms. It has to be mentioned, 194 that the results relate to the digestion of sewage sludge with TSS of about 7.8 %. 195 Nevertheless, NH<sub>4</sub>-N has to be considered in view of applying co-digestion. Generally, co-196 digestion is therefore suitable for substrates which have very low or no nitrogen due to the 197 resulting impact on the water stream of a WWTP.

SDT	reactor	Ø TSS	Ø NH <sub>4</sub> -N	Ø NH <sub>3</sub>
SKI	(temperature)	[g L <sup>-1</sup> ]	[mg L <sup>-1</sup> ]	[mg L <sup>-1</sup> ]
	R1 (39 °C)	40.7±2.68	2,023±101	69.3
25 d	R2 (37 °C)	41.0±2.13	1,903±43	57.1
	R3 (35 °C)	39.9±3.66	1,933±156	47.3
	R1 (41 °C)	38.3±4.37	$1,859\pm58$	75.3
25 d	R2 (37 °C)	37.7±4.28	1,760±64	51.1
	R3 (33 °C)	38.4±4.27	1,851±43	33.6
	R1 (41 °C)	41.1±1.15	1,752±79	72.5
20 d	R2 (37 °C)	$40.8 \pm 1.24$	$1,627\pm56$	48.6
	R3 (33 °C)	$40.5 \pm 1.85$	1,701±71	31.0
	R1 (41 °C)	40.1±1.20	1,742±95	75.3
15 d	R2 (37 °C)	39.5±1.35	$1,653{\pm}100$	50.3
	R3 (33 °C)	39.5±1.59	1,781±74	33.0
	R1 (41 °C)	46.0±2.18	1,578±83	55.9
10 d	R2 (37 °C)	44.0±2.66	1,437±58	35.7
	R3 (33 °C)	46.0±2.20	1,547±81	23.4

198 Table 2: Mean analytical parameters of the reactors at temperatures of 33 to 41 °C and 10 to 25 d SRT

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## 200 3. Nitrogen concentration

201 An overall lower removal can also occur when an incomplete hydrolysis of poorly degradable 202 substances appears. Especially cellulose as carbohydrate-rich compounds is difficult to break 203 down, while proteins are more available for organisms (Young et al. 2013). Hydrolysis is a 204 process of the first order and independent from the anaerobic or aerobic conditions. It is 205 known that SRT and temperature influence hydrolysis. (Miron et al. 2000; Ferreiro & Soto 206 2003). To consider the breakdown of organic compounds, NH<sub>4</sub>-N release was calculated in 207 relation to the removed COD. This relation could be applied to identify the more reduced 208 organic groups and the progress of hydrolysis. Hydrolysis is often found to be the rate 209 limiting step of particulate substances in the anaerobic conversion and therefore important for 210 the process efficiency (Miron et al. 2000; Ferreiro & Soto 2003; Mahmoud et al. 2004). Table 211 3 shows the NH<sub>4</sub>-N release, calculated as percentage from the total supplied organic nitrogen 212 load and the ratio of released NH<sub>4</sub>-N to degraded COD at the tested conditions. Hereby, the 213 calculated ratio of released NH<sub>4</sub>-N to degraded COD at 15 to 25 d SRT occurred in a narrow 214 range. A clear increased ratio was observed with 10 d SRT, while more nitrogen compounds 215 were released than carbon removed representing a reduced hydrolysis of carbon compounds.

216 For the evaluation of the breakdown; the NH<sub>4</sub>-N release was further expressed as ratio to the 217 total supplied organic nitrogen load. As mentioned before, the nitrogen released during the 218 first experiments was slightly enhanced because of higher environmental temperatures and 219 subsequently stronger hydrolysis during static thickening. The NH<sub>4</sub>-N released for 220 temperatures of 37 and 41 °C with 15 to 25 d SRT fluctuated only slightly and reached about 221 42 (±2) %. Beside low differences between 37 and 41 °C; the lowest NH<sub>4</sub>-N release was 222 always found at  $33^{\circ}$ C with  $39 (\pm 2)$  %. The observation clarifies the correlation between 223 temperatures and hydrolysis as reported by Ferreiro and Soto (2003). A lower release of NH<sub>4</sub>-224 N for all temperatures was even determined in the tests with 10 d SRT, that was mainly 225 caused by and insufficient degradation of organic load by short retention time.

ODT	reactor	$Ø \Delta NH_4$ -N/ $\Delta COD$	Ø NH4-N release [%]	
SKI	(temperature)	[mg g <sup>-1</sup> ]		
	R1 (41 °C)	24.6	43.8	
25 d	R2 (37 °C)	24.8	44.4	
	R3 (33 °C)	23.1	40.4	
20 d	R1 (41 °C)	27.7	43.6	
	R2 (37 °C)	27.3	42.4	
	R3 (33 °C)	26.5	40.0	
15 d	R1 (41 °C)	23.6	40.7	
	R2 (37 °C)	24.0	42.6	
	R3 (33 °C)	22.0	38.5	
10 d	R1 (41 °C)	37.5	35.5	
	R2 (37 °C)	37.1	33.6	
	R3 (33 °C)	33.0	30.5	

226 Table 3:  $NH_4$ -N release of the reactors at temperatures of 33 to 41 °C and SRTs of 10 to 25 d

4. Removal performance and gas production

228 Table 4 summarizes the results of degradation for the different operating conditions. VSS and 229 COD removal were calculated from the total initial load, the total effluent load and changed 230 amount in digested sludge over the operation period. The first period with 25 d SRT showed 231 an increased removal compared to the following experiments. As mentioned before, an 232 intensified hydrolysis during the thickening and subsequently a higher degradation was 233 expected. After the first period, the experiments were interrupted and the reactors refilled with 234 fresh sludge. In this context, an overall reduced VSS removal of 49 % was observed with 235 likewise 25 d SRT. It can be assumed, that the differences result from an insufficient start-up 236 period and incomplete adaptation of biomass to the increased temperatures and TSS. The 237 importance of a sufficient acclimation of bacteria to higher ammonia nitrogen concentration 238 was also reported by Fujishima et al. (2000). The results of these investigations with 25 d 239 SRT were therefore not considered in detail. Similar results as in period 1 were found with a 240 SRT of 20 and 15 d. The removal here was about 53 % (VSS) and 57 % (COD). A stable 241 operation was reached with 15 to 25 d SRT and OLR in a range of 2.0 to 3.8 g VSS m<sup>-3</sup> d<sup>-1</sup>.

A declined removal was seen by reducing SRT to at least 10 d, while COD and VSS removal was about 7 % (absolute) lower compared to the test with 15 d SRT. A lower removal efficiency was also seen in an increased TSS in digested sludge of about 44 to 46 g L<sup>-1</sup>. Moreover, organic acid concentrations during the experiments with 10 d SRT appeared up to

810 mg L<sup>-1</sup>. Similar observations were reported by Kapp (1984), who found a VSS reduction 246 247 of 50 to 54 % at TSS of 4.5 % and SRT of 15 to 30 d, while enhanced TSS of about 7 % in 248 digested sludge leads to a reduced VSS removal of 45 to 48 %. A clear influence of SRT and 249 OLR on VSS reduction was also recognized by Duan et al. (2012). In this study, the increase 250 of OLR and the reduction of SRT, lead to increasingly acid concentration. The accumulation can be explained by a delayed degradation of acids formed during acidogenic phase. Since the 251 252 conversion rate of acidogenic microorganisms are higher compared to the conversion rate of 253 methane forming microorganisms, more acids are produced than consumed when the load 254 exceeds an upper limit. During the experiment with 10 d SRT, OLR was about 5.5 g VSS m<sup>-3</sup> d<sup>-1</sup>. For the reason of acid accumulation, the alkalinity in digested sludge is 255 important, while a low alkalinity is known to induce the appearance of not dissociated acids 256 257 which inhibit bacteria. Normally sewage sludge has a high alkalinity that buffers pH shifts. In 258 this study, pH dropped by changing SRT from 15 d to 10 d SRT from about 7.35 to 7.25 and 259 was despite the increased acid concentration in an optimal range.

260 Comparing the VSS and COD removal at the investigated temperatures; there were only 261 minor differences. As a result of this study a low degradation, such as that observed here 262 when applying a 10 d SRT, can cause decreased dewaterability with higher polymer demand 263 and disposal costs. This hypothesis is supported by findings from Miron et al. (2000), who 264 found a clearly decreased dewaterability of digested sludge by reducing SRT from 15 to 10 d. 265 Furthermore, a reduced dewaterability by changing SRT from 30 d to 8 d was observed by Kapp (1984). The optimal results of dewaterability for mesophilic temperatures were reported 266 267 by Mahmoud et al. (2006) with SRT in a range on 15 to 20 d.

Table 4Additionally, the methane production was calculated in relation to the supplied VSS. 268 269 Methane production is important for the energy recovery by WWTPs. Methane production of about 380 NmL g  $VSS_{IN}^{-1}$  were observed in the first experiments, which correlate to an 270 271 increased removal and were mainly caused by higher environmental temperature. The 272 methane yields at the following tests with 25 to 15 d SRT were much lower, while the highest 273 amounts of 350 to 360 NmL g VSS<sub>IN</sub><sup>-1</sup> were determined at 37 °C. A 2.9 to 10.1 % lower methane production was observed at 33 °C, while the methane yield at 41 °C was only 1.5 to 274 275 4.8 % lower compared to 37 °C. This result corresponds with research from Bouskova et al. 276 (2005), while in the situation of changing the temperature from 37 to 42 °C only minor 277 changes in gas production were seen. However, for economically reasons temperatures of 278 41 °C should not be applied at large-scale.

279 Moreover, the investigated methane production for 15 to 25 d confirm findings from Nges and Liu (2010); they observed 0.314 to 0.348 °Nm<sup>3</sup> CH<sub>4</sub> kg VSS<sub>IN<sup>-1</sup></sub> at SRT between 12 and 35 d; 280 while an unstable process was found at 9 d SRT. For this study a decreased methane 281 282 production was found for all tested temperatures at 10 d SRT; while the conversion with 283 41 °C was not that strong influenced like it was observed for 33 and 37 °C. This outcome is 284 similar to findings from Nges and Liu (2010). They found that under thermophilic conditions the VSS removal showed better performance at shorter SRTs (15-5 d); while mesophilic 285 286 digestion reached higher VSS removal at longer SRT (35-20 d). It can be assumed that higher 287 temperatures at short SRT improve the breakdown of biopolymers and are responsible for the 288 comparatively higher methane production and removal.

289 An important sludge property that was not investigated in this study, but is significant for 290 WWTP, is the dewaterability of digested sludge. Skinner et al. (2015) described the link 291 between VSS concentration and dewatering behavior by comparing fifteen sludges with VSS 292 concentration of 40 to 80 %. As a result of this study a low degradation, such as that observed 293 here when applying a 10 d SRT, can cause decreased dewaterability with higher polymer 294 demand and disposal costs. This hypothesis is supported by findings from Miron et al. (2000), 295 who found a clearly decreased dewaterability of digested sludge by reducing SRT from 15 to 296 10 d. Furthermore, a reduced dewaterability by changing SRT from 30 d to 8 d was observed 297 by Kapp (1984). The optimal results of dewaterability for mesophilic temperatures were 298 reported by Mahmoud et al. (2006) with SRT in a range on 15 to 20 d.

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Table 4:Mean organic acid concentrations, VSS and COD removal and specific methane yield at<br/>temperatures of 33 to 41 °C and SRT of 10 to 25 d

SRT	reactor (temperature)	VSS removal	COD removal	spec. CH <sub>4</sub> prod.	Ø acids
		[%]	[%]	$[mL gVSS_{IN}^{-1}]$	[mg L <sup>-1</sup> ]
25 d	R1 (39 °C)	50.6	60.7	381	59.8±30
	R2 (37 °C)	53.0	62.7	387	69.1±73
	R3 (35 °C)	-	-	-	54.8±35
25 d*	R1 (41 °C)	48.0	56.0	333	40.1±214
	R2 (37 °C)	49.8	58.2	367	22.0±12
	R3 (33 °C)	48.6	57.4	330	25.4±12
20 d	R1 (41 °C)	52.4	57.2	339	54.4±589
	R2 (37 °C)	55.9	59.3	356	29.3±21
	R3 (33 °C)	53.1	57.7	327	24.4±24
15 d	R1 (41 °C)	53.4	57.5	341	40.8±143
	R2 (37 °C)	53.8	57.7	346	42.6±28
	R3 (33 °C)	54.3	57.3	336	54.5±36
10 d	R1 (41 °C)	47.5	53.0	330	74.5±64
	R2 (37 °C)	46.1	49.9	286	810.0±523
	R3 (33 °C)	47.8	49.9	320	551.0±678

301 \* not considered in detail, because of an insufficient start-up period and incomplete adaptation of biomass

## 302 Conclusion

303 The results of this study indicate that digesters of municipal WWTPs can be operated at highsolid concentrations as long as a minimal sludge age of 15 d is ensured and NH<sub>4</sub>-N remains 304 below 2,000 mg L<sup>-1</sup>. The findings refer to high-solid digestion of raw sludge with TSS in a 305 306 range of 6.7 to 7.8 %. High-solid digestion with 10 d SRT results in an unstable process and 307 reduced breakdown of carbonate-rich compounds. The right selection of the operational 308 temperature is important for a cost-effective anaerobic treatment. Temperatures of 41 °C did 309 not improve the reduction or the gas production nor did it lead to an unstable process by forming NH<sub>3</sub>. However, the higher energy demand for sludge heating makes a large scale 310 311 operation with 41°C unattractive. It was observed that temperatures of 33 °C led to a reduced 312 breakdown of COD compounds and decreased methane production. Thus, temperatures of 313 33 °C should not be applied to reach an efficient energy recovery. The impact of solids in the 314 feed above 8.0 % and subsequently the resulting NH<sub>4</sub>-N concentration should be examined 315 separately. Furthermore, an advanced investigation of sludge dewaterability can be helpful to 316 calculate total cost effectiveness of different treatment options.

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321

## 322 Abbreviations

323	COD	chemical oxygen demand
324	SRT	sludge retention time
325	OLR	organic loading rate
326	TSS	total suspended solids
327	VSS	volatile suspended solids
328	WWTP	wastewater treatment plant
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330 331	Figure 1:	Mean NH <sub>4</sub> -N and TKN concentration of raw sludge and NH <sub>4</sub> -N concentration of digested sludge samples
332	Table 1:	Operation adjustments and mean analytical data of used raw sludge
333 334	Table 2:	Mean analytical parameters of the reactors at temperatures of 33 to 41 °C and 10 to 25 d SRT
335 336	Table 3:	NH <sub>4</sub> -N release of the reactors at temperatures of 33 to 41 °C and SRTs of 10 to 25 d7
337 338	Table 4:	Mean organic acid concentrations, VSS and COD removal and specific methane yield at temperatures of 33 to 41 °C and SRT of 10 to 25 d9
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