

Effect of NH_3 and organic loading on the inhibition of mesophilic high-solid digestion

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Abstract

BACKGROUND: The construction of small digesters will become more important for regions with demographic increase and limited areas. Savings of digester volume and a subsequently smaller footprint can be realized by the treatment of a more thickened raw sludge (high-solid digestion). However, this operation can lead to instabilities of the anaerobic process due to rising ammonium and ammonia levels. The aim of the study was to identify the limit of saving digester volume by increasing the total suspended solids (TSS) of the treated raw sludge.

RESULTS: The effects of high TSS and ammonia levels in the digested sludge were investigated in lab-scale reactors. Ammonium nitrogen ($\text{NH}_4\text{-N}$) levels were set by dosing a urea solution as well as by feeding a higher concentrated raw sludge. Chemical oxygen demand (COD) removal declined from 64 to 54% with $\text{NH}_4\text{-N}$ levels rising from 2000 to 3200 mg L^{-1} . However, the anaerobic biodegradation was not completely interrupted, which indicates the possible adaptation of anaerobic bacteria on high $\text{NH}_4\text{-N}$ levels.

CONCLUSION: From the results, it can be concluded that dewatering the raw sludge up to 7% TSS leads to an optimum benefit with maximum savings in digester volume. The benefits of the operation with high solids are a lower energy demand for heating up the raw sludge and reduced construction costs (−20%) due to smaller required digester volumes.

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Keywords: anaerobic digestion; biogas; process optimization; reactor design

INTRODUCTION

During anaerobic digestion, organic solids are converted into energy-rich methane gas and carbon dioxide. Most large wastewater treatment plants (WWTPs) use anaerobic digesters to recover energy from the sewage sludge in order to reduce their electricity demand. However, the construction of an anaerobic sludge treatment is cost-intensive. Besides all associated infrastructural measures and equipment, the investment costs mainly depend on the digester size and can make up half of the total costs. Therefore a reduced digester volume has the potential to save a substantial part of the investment costs. The digester volume is usually calculated by the hydraulic retention time (HRT) and the accruing amount of sludge. According to the literature,¹ the amount of total suspended solids (TSS) per day and population equivalent (PE) is 90 g and cannot be reduced substantially. For the digester operation, HRTs of 16–22 days (plant capacity > 100 000 PE) are state of the art² and widely applied for design.

Against the background of saving digester volume, the amount of supplied raw sludge can be reduced by increasing the solid content throughout further thickening. Hereby the TSS of the raw sludge, a mixture of sludge from preliminary clarification and waste activated sludge, mainly determines the TSS in the digester. Standard rate digesters without mixing are often operated in a range of 2–3% TSS, which corresponds to 4–6% TSS in the raw sludge. So-called ‘high-solid digestion’ is characterized by solid contents in the digester above 3%. Beside a smaller digester size,

the advantage of such a high-solid operation is an overall lower heating demand, which further reduces the operational costs of the sludge treatment.

Although there are substantial advantages of high-solid digestion, the levels of total ammonium nitrogen (TAN) and free ammonia nitrogen (FAN) should be considered carefully in order to avoid adverse process conditions. During the anaerobic process, organically bound nitrogen is released as ammonium, which is directly associated with the formation of hydrogen carbonate (HCO_3^-). Thus increasing HCO_3^- and NH_4^+ concentrations lead to increased alkalinity and higher pH values. The amount of ammonia corresponds to the ammonium concentration, pH value and temperature (ammonia-nitrogen/ammonium-nitrogen ($\text{NH}_3\text{-N}/\text{NH}_4\text{-N}$) equilibrium). It is commonly known that ammonia can cause inhibitory effects, since the hydrophobic ammonia can passively diffuse into bacteria cells and provoke proton imbalances.³

Some previous studies provide information about the impact of TAN and FAN on anaerobic biodegradation by the use of various

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organic substrates.^{4–7} Most of these studies relate to the treatment of agriculture wastes such as swine or poultry manure with nitrogen levels higher than 4 g L⁻¹.^{6,8,9} Although municipal raw sludge usually has much lower nitrogen concentrations, the treatment of a higher thickened raw sludge as well as the treatment of co-substrates can boost the nitrogen level substantially.

There is a broad range of NH₄-N levels reported that cause inhibitory effects on the anaerobic bacteria. For example, McCarty¹⁰ observed an inhibition of the anaerobic process with NH₄-N levels above 1500 mg L⁻¹ and pH values around 7.4. Duan *et al.*⁷ found a slight to significant inhibition with FAN concentration in a range of 250–800 mg L⁻¹, while the methanogenic organisms were not evidently affected with TAN levels up to 2500 mg L⁻¹. Poggi-Varaldo *et al.*⁶ reported a critical NH₄-N concentration of 2800 mg L⁻¹ for the treatment of urban wastes and biosolids. Kroeker *et al.*¹¹ investigated the anaerobic digestion of swine manure and observed that in a range of 1700–1800 mg TAN L⁻¹ a non-acclimatized biomass was inhibited, whereas an acclimatized inoculum could stand up to approx. 5000 mg TAN L⁻¹. Moreover, Van Velsen¹² and Sung and Liu¹³ stated that the acclimatization of microorganisms allows operation with TAN concentrations up to 5000 mg L⁻¹. In these studies, 'inhibited steady state' conditions were achieved with an overall lower methane production. These findings indicate that the anaerobic organisms are able to adapt to high NH₄-N concentrations, thus it is not possible to define an overall critical NH₄-N level for different bacterial communities, substrates and process conditions.

Although most of the literature relates to the inhibition of FAN and TAN on the anaerobic process, some studies report positive effects on the anaerobic organisms with regard to anaerobic hydrogen production and sludge disintegration. For example, Procházka *et al.*¹⁴ revealed that NH₄-N concentrations below 500 mg L⁻¹ are responsible for lower methane yields due to a low buffer capacity. Wang *et al.*¹⁵ investigated the effects of ammonium and free ammonia on hydrogen production during dark fermentative hydrogen production, which was provoked by an alkaline pretreatment to inhibit hydrogen-consuming bacteria. The authors found an increased hydrogen production by increasing the NH₄-N level from 36 to 266 mg L⁻¹, corresponding to ammonia concentrations from 34 to 254 mg L⁻¹. Although there was an inhibition of the hydrolysis and acidogenesis by ammonia, the hydrogen consumption processes such as methanogenesis and sulfate reduction were much more affected. Moreover, Liu *et al.*¹⁶ observed an increased hydrolysis rate and methane potential after the pretreatment of raw sludge with ammonia and heat (70 °C). This combined pretreatment ensured an enhanced disintegration of waste activated sludge and provided an increased amount of biodegradable organic substances.

Another important parameter for anaerobic biodegradation is the organic loading rate (OLR), defined as the chemical oxygen demand (COD) or volatile suspended solids (VSS) load that is supplied to the reactor per day. Generally, the OLR for mesophilic single-stage digestion is in a range of 1.0–1.7 kg VSS m⁻³ day⁻¹ or 1.7–2.9 kg COD m⁻³ day⁻¹.² The treatment of raw sludge with increased TSS leads subsequently to an increased OLR and elevated ammonium levels. Since anaerobic digestion is an interaction of different organisms with different conversion rates, an increased OLR can cause imbalances in the anaerobic degradation, with the accumulation of volatile fatty acids (VFAs). High VFA concentrations can subsequently decrease the pH value

and inhibit the methanogenic archaea. A pH value in a range from 6.8 to 7.8 is prerequisite for a stable anaerobic process without the occurrence of considerable inhibition.¹⁷ The pH value can be stabilized by enhanced NH₄-N levels and increased buffer capacity.

Information about the OLR and NH₄-N on anaerobic digestion is very limited. Fujishima *et al.*¹⁸ investigated the anaerobic digestion of raw sludge with TSS contents of 3–11%. VSS removal ranged between 45.6 and 33.8% with NH₄-N levels from 710 to 3100 mg L⁻¹. The highest methane production of 330 mL g⁻¹ VSS was found with 7.1% TSS in the raw sludge. Although that paper provides extensive information on the effect of NH₄-N on the biodegradation and methane yield, the adjusted HRT was only 14 days and thus considerably lower compared with the recommended range for municipal digester operation. Jahn *et al.*¹⁹ observed an increased organic acid accumulation with HRTs below 15 days, indicating that this parameter should be controlled carefully to ensure stable process conditions. Highest removal was reported with HRTs between 20 and 25 days, which is also considered for the design of municipal digesters. In another study from Kapp,²¹ it was concluded that the anaerobic process is not impaired with up to 9% TSS in the digested sludge, corresponding to 13% TSS in the raw sludge. Lay *et al.*²² found that increasing the TSS from 4 to 10% leads to a declining methanogenic activity (from 100 to 53%). The correlation between the increased TSS and NH₄-N concentrations is essential for high-solid digestion and indicates the challenging character to forecast the effects of different operational strategies and substrates.

Since the results of previous studies vary widely owing to different settings (pH value, temperature) and organic substrates,⁵ the focus of the present study was to investigate the impact of increased NH₄-N levels and OLRs on the anaerobic biodegradation of municipal raw sludge, especially on the methane production and removal performance. For the experimental procedure, the common settings for municipal digester operation in Austria were considered with an HRT of 25 days and a mesophilic temperature of 37 °C. This is relevant in order to ensure the transfer of the results to full-scale plant operation, since previous studies often relate to different settings or substrates. Defined NH₄-N levels were set to lab-scale reactors by adding a urea solution as well as by feeding a more thickened raw sludge to simulate increased OLRs and high-solid digestion. A further purpose of the present study was to identify the optimal OLR for mesophilic high-solid digestion. Particular attention was paid to the COD and VSS removal in order to estimate the potential in saving digester volume without an inhibition of the anaerobic community.

MATERIALS AND METHODS

Experimental setup and procedure

The experimental setup consisted of three identical reactors (3 L) placed in a thermostatic bath at 37 °C. The reactors were fed with a mixture of primary sludge and waste activated sludge (hereafter referred to as raw sludge) taken from a static thickener of a two-stage activated sludge plant. The reactors were stirred continuously to ensure sufficient mixing. Raw sludge was supplied once a day on 5 days a week to adjust an HRT of 25 days. The amount of raw sludge added per day was 168 g. The biogas volume was measured by a drum gas analyzer (Bochum Langendreer 11066, Ritter, Bochum, Germany) and recorded daily before feeding. Since the room temperature was constantly in a range of 22–24 °C, the gas volume

Table 1. Mean analytical data and standard deviations of supplied raw sludge during phases I–IV

Parameter	Unit	Phase I	Phase II	Phase III	Phase IV
Chemical oxygen demand (COD)	g L ⁻¹	61.8 ± 10.9	58.9 ± 14.1	61.3 ± 9.1	75.7 ± 3.8
Total suspended solids (TSS)	%	4.4 ± 0.9	4.0 ± 1.0	4.3 ± 0.6	5.2 ± 0.4
Volatile suspended solids (VSS)	%	3.5 ± 0.7	2.9 ± 0.6	3.3 ± 0.4	4.1 ± 0.3
Total Kjeldahl nitrogen (TKN)	g L ⁻¹	2.2 ± 0.3	2.2	2.5	–
COD/VSS ratio	–	1.79	1.92	1.84	1.87

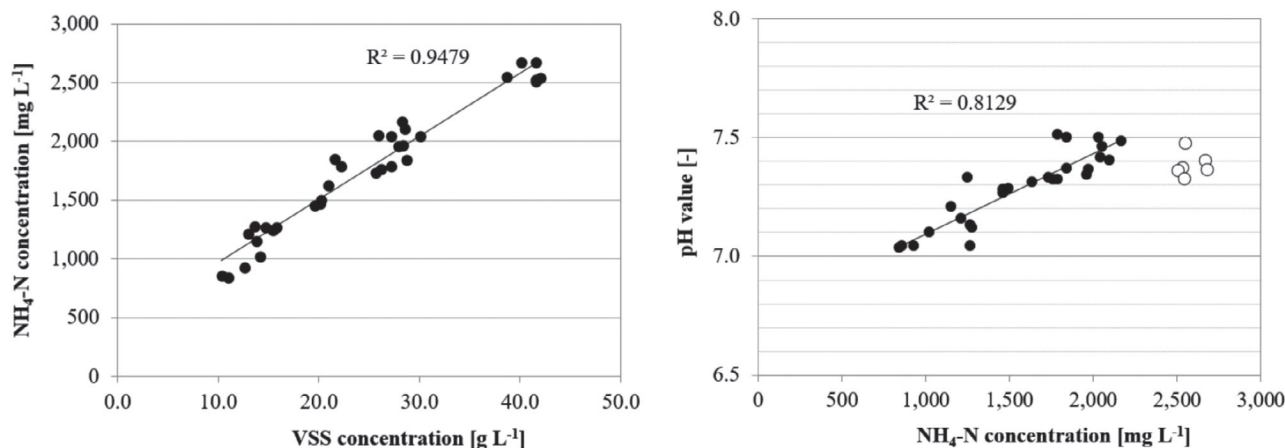


Figure 1. VSS to NH₄-N concentrations (left) and NH₄-N concentrations to pH values (right) in digested sludge for R3 (HRT = 25 days, *t* = 37 °C) (full circles, low organic acids; empty circles, increased organic acids).

was only normalized for calculating the COD balance. The experiments were conducted according to Jahn *et al.*¹⁹ and VDI (2016).²⁰

Raw sludge and digested sludge samples were analyzed for COD, TSS, VSS, total Kjeldahl nitrogen (TKN) and NH₄-N. NH₃-N concentrations were calculated according to Hobiger.²³ COD analysis comprised the total COD (solid and liquid fractions). Temperature and pH values were controlled daily by a WTW SenTix20 pH meter. Organic acids (formic, butyric, valeric, acetic, propionic and lactic acids) were measured by an isocratic high-performance liquid chromatographic method including the separation and quantitative analysis of organic acids. The detected organic acids were calculated as acetic acid equivalent, whereby only the liquid fraction of the samples was considered. A GFM400 gas analyzer (Gas Data LTD., Coventry, UK) was used to determine the methane content in the biogas. COD mass balances were calculated in order to control the analyzed samples and methane yields.

Experimental procedure

The laboratory trials were divided into four experimental phases with different NH₄-N concentrations and OLRs. During phases I–III, reactors 1 and 2 (R1 and R2) were dosed with a urea solution (150 g N L⁻¹) to set defined NH₄-N concentrations. Reactor 3 (R3) served as reference reactor without a urea dosage. During phase IV, different NH₄-N concentrations were set by the use of individual thickened raw sludge to examine the effect of the OLR in combination with increased NH₄-N levels. A laboratory centrifuge (Sigma 3-16L) was applied for the raw sludge thickening.

Table 1 summarizes the mean analytical data and standard deviations of the raw sludge during phases I–IV. Raw sludge was directly taken from a municipal WWTP. The composition of the raw sludge showed seasonal fluctuations in a range of 2.9–4.1% VSS

and 5.9–7.6% COD. Moreover, the COD/VSS ratio ranged from 1.79 up to 1.92 and the TKN was about 2.2–2.5 g L⁻¹. COD/VSS ratios were calculated to document changes in the sludge composition for the different experimental phases. The reason for a slightly fluctuating composition is that the raw sludge comprised also primary sludge, which is mainly affected by rain events and temperature fluctuations.

RESULTS AND DISCUSSION

NH₄-N release

Figure 1 (left) shows the VSS and NH₄-N concentrations in the digested sludge for R3 (reference without urea dosage). A linear correlation between the two parameters was found with a coefficient of determination of 0.95. Data evaluated under low organic acid concentrations are expressed as full circles; results belonging to higher organic acids are shown as empty circles. A correlation between the VSS load and the released ammonia can be expected if the biodegradation of organic compounds is stable. Moreover, higher NH₄-N concentrations lead to increased pH values (Fig. 1, right). During the anaerobic process, the NH₄-N concentrations are directly associated with the occurrence of HCO₃⁻, which in turn affects the alkalinity of the digested sludge.²⁰ However, decreased pH values can appear due to higher organic acids consuming the alkalinity (empty circles). Especially methanogenic archaea are sensitive to pH shifts and prefer pH values in a range of 7.0–7.5. Moreover, higher NH₄-N levels can increase the pH value, which in turn affects the NH₄-N/NH₃-N balance in the direction of NH₃. The presented data illustrate the link between the TSS/VSS in the supplied raw sludge and the NH₄-N/pH values, which are key factors concerning a stable as well as economic anaerobic process.

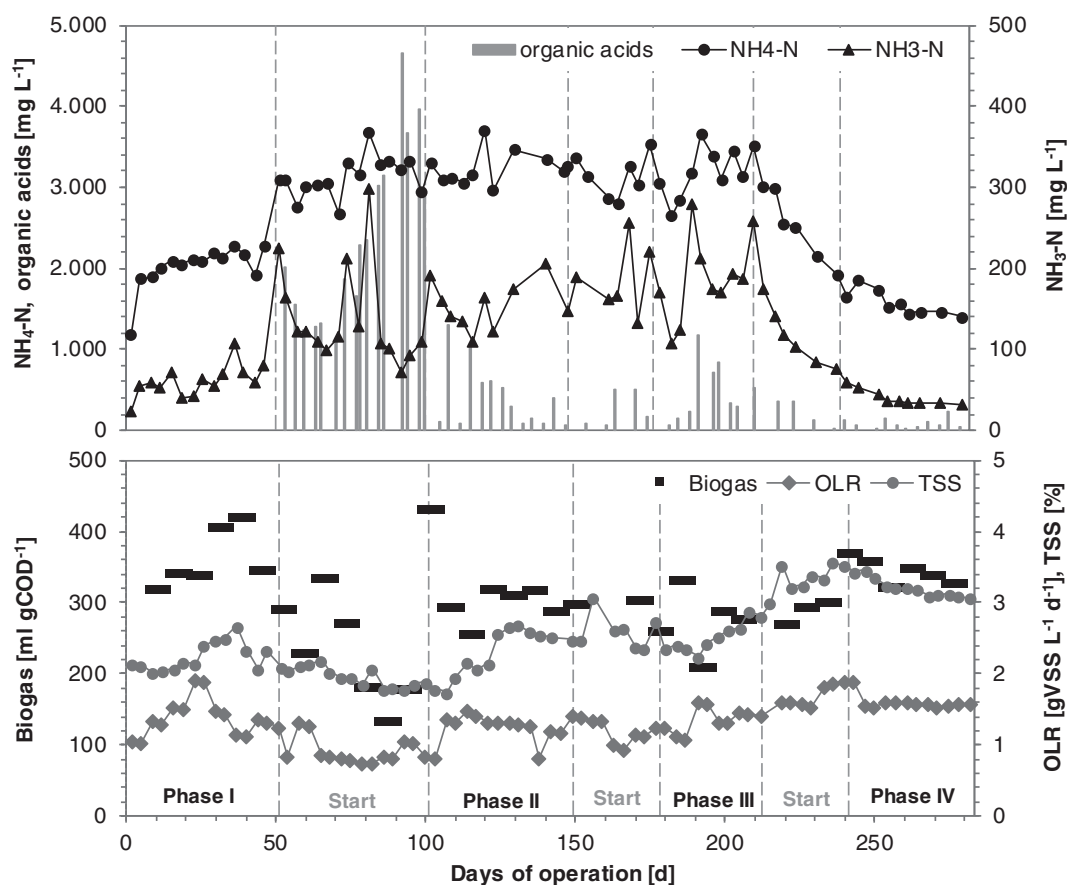


Figure 2. OLR, TSS, NH₄-N, NH₃-N, organic acids and biogas production of R1 (HRT = 25 days, $t = 37\text{ }^{\circ}\text{C}$).

Reactor performance

Reactor 1

Figure 2 shows the parameters analysed for R1 during phases I–IV. The mean TSS during the tests was 2.3% at OLRs in a range of 1.4–1.7 g VSS L⁻¹ day⁻¹. During phases I–III, a urea solution of 150 g N L⁻¹ was given stepwise to the reactor to adjust and hold a selected NH₄-N level. During the first phase, the mean NH₄-N concentration was 2018 mg L⁻¹ with an average NH₃-N concentration of 60.4 mg L⁻¹ (pH value of 7.39). Biogas production was between 340 and 420 mL g⁻¹ COD (related to the supplied COD). An increased urea dosage with the beginning of phase II led to an abrupt appearance of approx. 300 mg NH₃-N L⁻¹. In consequence, an accumulation of organic acids up to 4665 mg L⁻¹ was noticed. Biogas production decreased temporarily to 130 mL g⁻¹ COD but recovered within two weeks to 300 mL g⁻¹ COD. This period was regarded as the start-up phase and excluded from the evaluation. In the following phases II and III, mean NH₄-N concentrations of approx. 3250 and 3200 mg L⁻¹ respectively were measured. Considering the mean pH values of 7.62 and 7.71, the NH₃-N concentrations were between 153 and 183 mg L⁻¹. For phase IV, the dosage of urea was stopped and thickened raw sludge was supplied to the reactor at a mean OLR of 1.7 g VSS L⁻¹ day⁻¹. In consequence, the NH₄-N level decreased to 1485 mg L⁻¹ and the pH value to 7.33. The average solid concentration in the digested sludge was 3.2%. Compared with the previous phases, NH₃-N decreased below 50 mg L⁻¹, resulting in negligible organic acid concentrations. Under these conditions, the biogas production was stable at approx. 330 mL g⁻¹ COD.

Reactor 2

Figure 3 shows the analytical data for R2. Similarly to R1, the operation included the dosage of urea to set desired NH₄-N concentrations. During the first phase, the mean NH₄-N concentration was 1693 mg L⁻¹ with an average NH₃-N level of 42 mg L⁻¹ (pH value of 7.3). Biogas production was similar to R1 at 330–415 mL g⁻¹ COD. A slightly increased pH value of 7.6 was observed during phases II and III, which was attributed to the elevated NH₄-N levels of approx. 2750 and 2800 mg L⁻¹ respectively. Biogas production decreased to about 240 mL g⁻¹ COD during the start-up of phase II, which was comparable to the behavior of R1. Organic acids were determined in concentrations up to 2480 mg L⁻¹. According to the NH₄-N concentrations and pH values, the NH₃-N concentrations were calculated as 113–191 mg L⁻¹. During the start-up of phase IV, the OLR was changed from 1.3 to 2.8 g VSS L⁻¹ day⁻¹ by feeding a higher concentrated raw sludge. Consequently, the TSS climbed from 2.3 to 5.9% during this start-up. NH₄-N stabilized at approx. 2620 mg L⁻¹, which led to about 71 mg NH₃-N L⁻¹ at a pH value of 7.37. Organic acids increased continuously up to 3870 mg L⁻¹, confirming an unbalanced biodegradation. However, the biogas production declined to approx. 200 mL g⁻¹ COD, which underlines an inhibited steady state removal.

Reactor 3

R3 was used as reference reactor without urea dosage (Fig. 4). During the first and second phases, the OLR was between 1.4

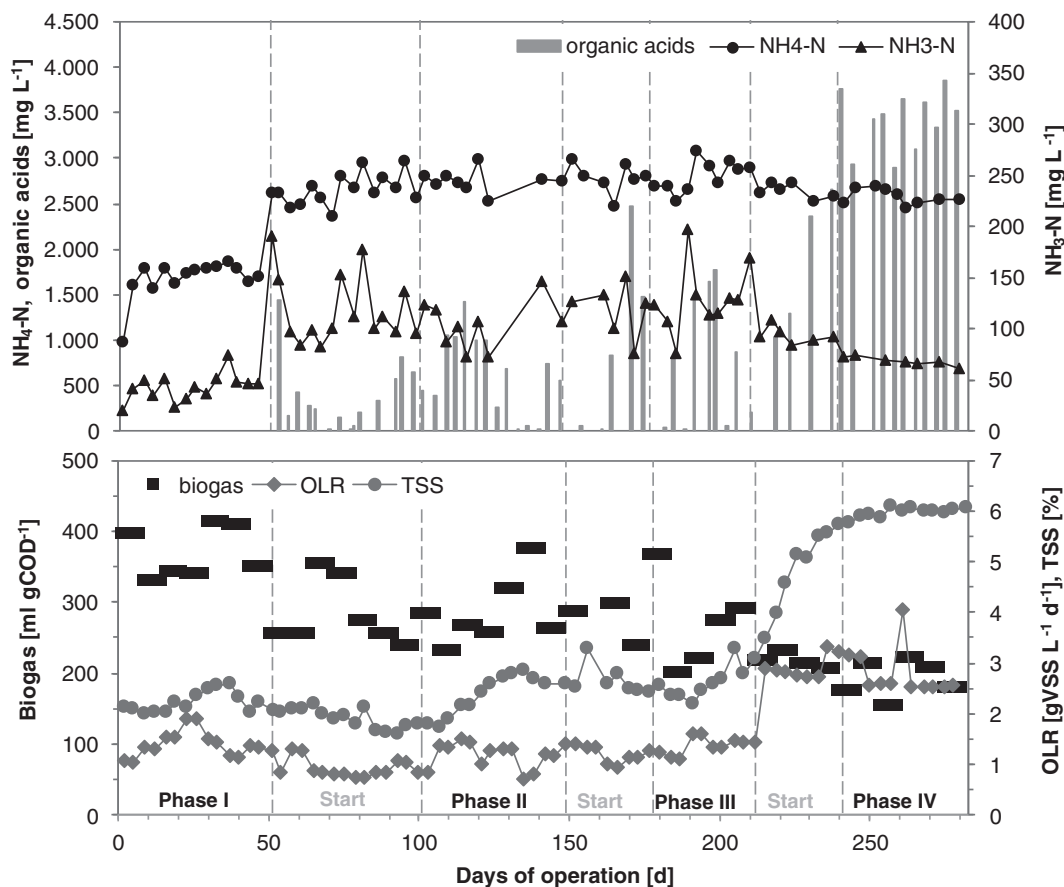


Figure 3. OLR, TSS, $\text{NH}_4\text{-N}$, $\text{NH}_3\text{-N}$, organic acids and biogas production of R2 (HRT = 25 days, $t = 37^\circ\text{C}$).

and $1.2\text{ g VSS L}^{-1}\text{ day}^{-1}$ with TSS in the digested sludge in a range of 2.2–2.8%. A clear correlation between the TSS and $\text{NH}_4\text{-N}$ was found for R3 as explained beforehand. $\text{NH}_3\text{-N}$ concentrations were low at $17.9\text{--}37.2\text{ mg L}^{-1}$. Organic acids were mostly below 30 mg L^{-1} . From phase III on, the OLR was increased to about $2.2\text{ g VSS L}^{-1}\text{ day}^{-1}$ by feeding a higher concentrated raw sludge. As a result, the mean TSS in the reactor was 4.4% with $2042\text{ mg NH}_4\text{-N L}^{-1}$. According to a pH value of 7.45, $\text{NH}_3\text{-N}$ was calculated as approx. 66.4 mg L^{-1} . The increased OLR at the beginning of phase III led to a temporary organic acid accumulation up to 3680 mg L^{-1} . In phase IV, the average OLR was $2.2\text{ g VSS L}^{-1}\text{ day}^{-1}$ with a mean TSS concentration of 4.4%. Organic acids were below 100 mg L^{-1} , indicating a stable anaerobic process.

Impact of OLR and $\text{NH}_4\text{-N}$

Table 2 summarizes the results of the anaerobic reactors for the four experimental phases with the different $\text{NH}_4\text{-N}$ concentrations and OLRs. Deviations in the mass balances were in a small range and below 10%.

For the evaluation, the results of the experimental phases were divided with regard to the applied OLRs resulting from the feed with different concentrated raw sludge. In group I (black squares), the tests with OLR between $1.2\text{ and }1.4\text{ g VSS L}^{-1}\text{ day}^{-1}$ were summarized corresponding to the recommended OLR for mesophilic digester operation.² During these tests, TSS in the raw sludge was between 4.0 and 4.4%. In the second group (gray circles), the experimental phases with OLR in the range of $1.7\text{--}2.2\text{ g VSS L}^{-1}\text{ day}^{-1}$ were summarized and thus above the usually

applied OLR for mesophilic digesters. Hereby the raw sludge samples were thickened to 5.2–7.0%. Moreover, the OLR of R3 during phase IV was increased to $2.8\text{ g VSS L}^{-1}\text{ day}^{-1}$ (third group, crosses), which was attributed to the feed of raw sludge with 8.9% TSS.

Figure 5 shows the COD removal (left) and the specific methane production (right) for the different groups. For all groups, a decreased COD removal and methane yield was observed with rising $\text{NH}_4\text{-N}$ levels. For the OLR in the range of $1.2\text{--}1.4\text{ g VSS L}^{-1}\text{ day}^{-1}$ (group I), the COD removal declined from 64% at $2000\text{ mg NH}_4\text{-N L}^{-1}$ to approx. 54% above $3200\text{ mg NH}_4\text{-N L}^{-1}$. A comparable trend with an overall lower COD removal and methane production was observed for the tests with OLRs above $1.7\text{ g VSS L}^{-1}\text{ day}^{-1}$ (group II). In the range of $1480\text{--}2040\text{ mg NH}_4\text{-N L}^{-1}$, the COD removal was only 55–58%. With a further enhanced OLR of $2.8\text{ g VSS L}^{-1}\text{ day}^{-1}$, a reduced COD removal of 36.1% was detected, while the average $\text{NH}_4\text{-N}$ concentration was 2580 mg L^{-1} . During this test, the methane production was significantly lower at approx. $140\text{ mL g}^{-1}\text{ COD}$.

Overall, the results reveal a decreased removal and methane production with $\text{NH}_4\text{-N}$ concentrations above 2500 mg L^{-1} . In addition, it was found that OLRs above the recommended range for municipal WWTPs lead to an overall reduced biodegradation and methane production. Nevertheless, the experiments indicate that even at a high OLR of $2.8\text{ g VSS L}^{-1}\text{ day}^{-1}$ the anaerobic degradation still works, although with a reduced removal and methane yield. The results of the tests with an increased OLR are in line with Duan *et al.*,⁷ who found a reduced VSS removal of 33.3% with an OLR of $3.0\text{ g VSS L}^{-1}\text{ day}^{-1}$ (TSS 10%). Furthermore, the authors

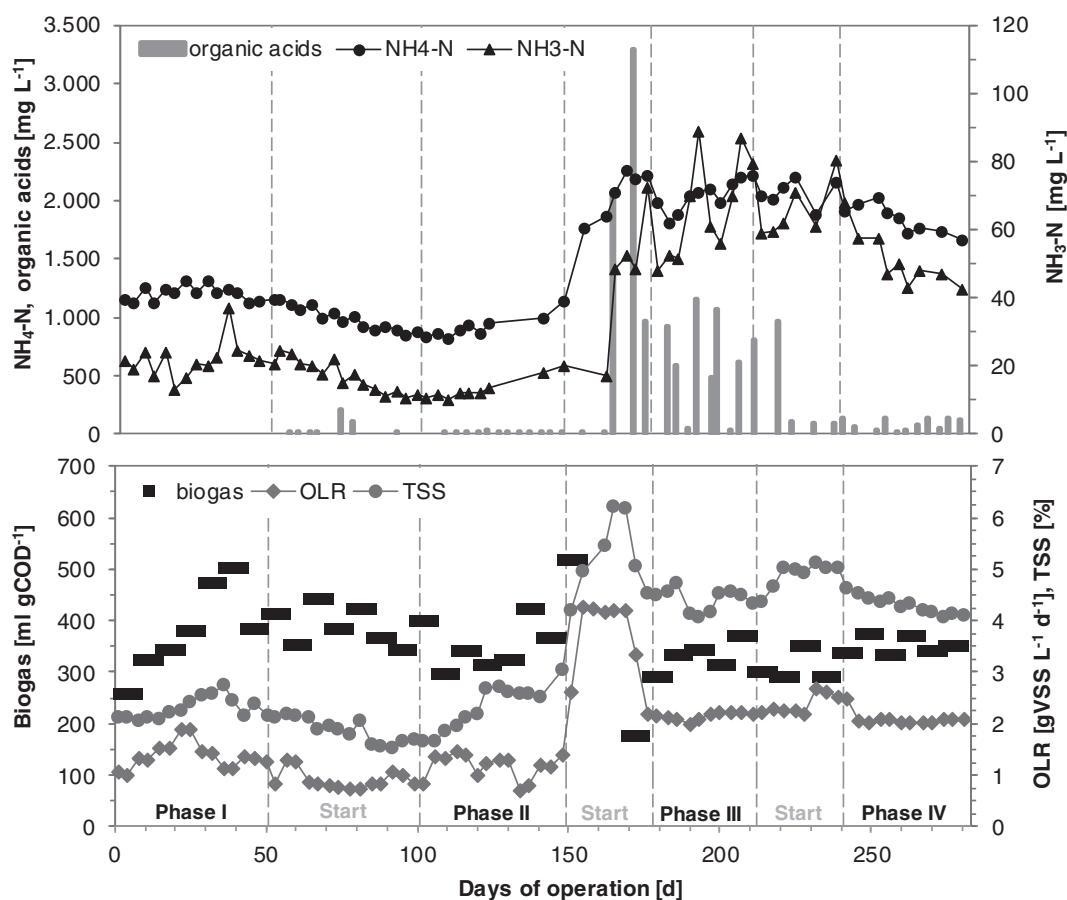


Figure 4. OLR, TSS, NH₄-N, NH₃-N, organic acids and biogas production of R3 (HRT = 25 days, $t = 37^{\circ}\text{C}$).

Table 2. Results of anaerobic tests with and without urea dosage and variable TSS concentrations

Phase	Reactor	OLR (g VSS L ⁻¹ day ⁻¹)	NH ₄ -N (mg L ⁻¹)	VSS removal (%)	COD removal (%)	CH ₄ yield (mL g ⁻¹ COD)
Phase I (49 days)	R1	1.4	2018	59.0	63.8	223
	R2	1.4	1693	60.3	64.2	225
	R3	1.4	1207	58.2	63.2	220
Phase II (49 days)	R1	1.2	3253	46.5	60.1	210
	R2	1.2	2752	44.7	57.1	200
	R3	1.2	900	48.1	60.7	212
Phase III (35 days)	R1	1.3	3197	47.3	54.0	189
	R2	1.3	2797	46.9	53.2	186
	R3	2.2	2042	49.2	49.2	193
Phase IV (46 days)	R1	1.7	1485	52.6	56.3	192
	R2	2.8	2579	36.1	39.1	137
	R3	2.2	1850	54.8	59.4	208

determined a clear decreased VSS removal of 20–25% above an OLR of 4.0 g VSS L⁻¹ day⁻¹. From an economic point of view, the results confirm the limit in increasing the OLR as approx. 2.2 g VSS L⁻¹ day⁻¹.

Additionally, the mean COD/VSS ratios of the digested sludge samples were calculated and found within a range of 1.59–1.66. However, a clear increased COD/VSS ratio of 1.76 was found for R3 (phase IV, OLR of 2.8 g VSS L⁻¹ day⁻¹), which was probably attributable to an overall disturbed conversion and acid accumulation. From the COD/VSS ratios, it becomes apparent

that the COD removal was more affected than the VSS removal at high OLRs. The differences in VSS and COD removal can be explained by the fact that organic compounds (VSS) are only converted into organic acids (dissolved COD) without a complete COD removal. Fujishima *et al.*¹⁸ reported a decreased carbohydrate removal efficiency in the tests with increased NH₄-N and explained the results by a decreased number of glucose-consuming acidogenic bacteria. Moreover, the authors state that the declined carbohydrate removal was attributable to an inhibition of ammonia on the glycolytic pathway. However,

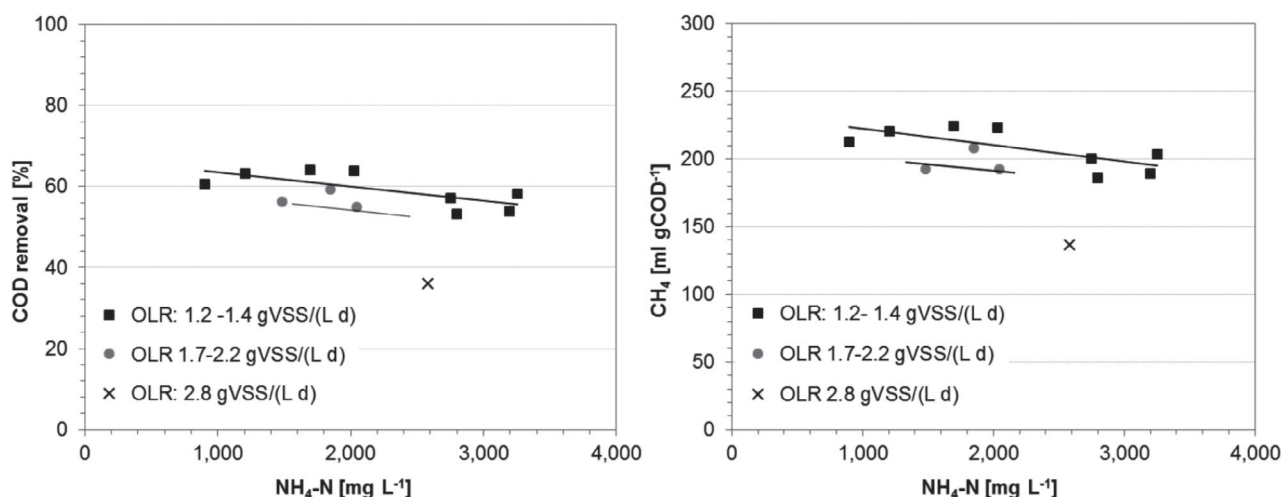


Figure 5. COD removal (left) and methane yields (right) at different $\text{NH}_4\text{-N}$ concentrations for experiments with different OLRs.

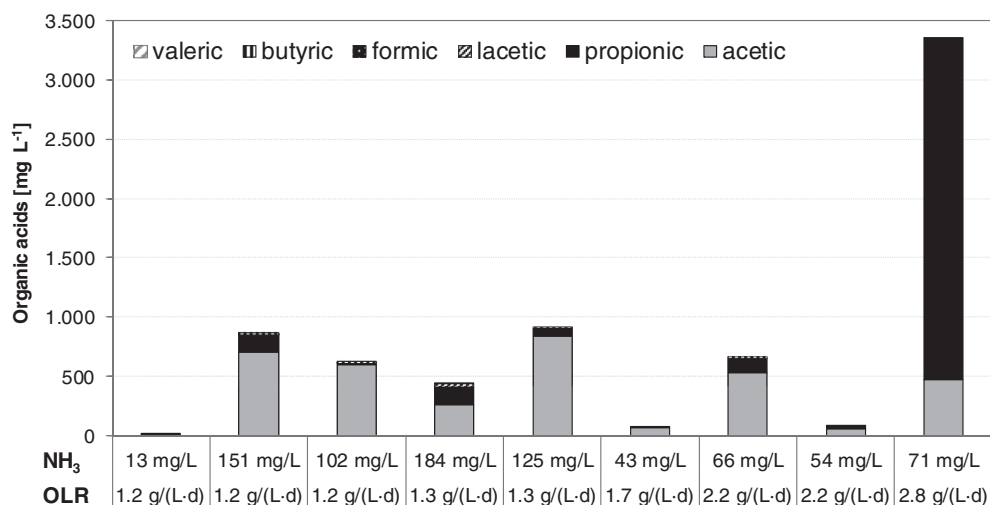


Figure 6. Mean organic acid concentrations at different OLRs (COD) (runs II–IV).

the ammonia concentrations were not significantly increased during the tests with high OLRs, which indicates that the low COD removal was mainly attributable to the higher organic acid accumulation.

Figure 6 shows the distributions of the organic acids during the anaerobic tests for runs II–IV, whereby especially propionic and acetic acids were detected. Lactic, formic, butyric and valeric acids were analyzed in concentrations below 20 mg L^{-1} . Unfortunately, there were no organic acids sampled during the first run. Considering the organic acids during the tests with OLRs in a range between 1.2 and $1.3 \text{ g VSS L}^{-1} \text{ day}^{-1}$, the percentage of acetic acid was 63–100% ($7\text{--}838 \text{ mg L}^{-1}$). Similar distributions were found for OLR in a range of $1.7\text{--}2.2 \text{ g VSS L}^{-1} \text{ day}^{-1}$, with approx. 68–83% acetic acid. The results confirm that the formation of acetic acid caused by an inhibition of the acetoclastic methanogens was mainly attributable to higher $\text{NH}_4\text{-N}$ and $\text{NH}_3\text{-N}$ concentrations, since the OLR was only slightly above the recommended range for mesophilic digester operation. This observation is in line with recent studies which report that ammonia concentrations of $130\text{--}330 \text{ mg NH}_3\text{-N L}^{-1}$ inhibit especially the acetoclastic methanogenic archaea in their activity.²⁴ Furthermore, the methanogenic pathway over acetate is more

effected since the acetoclastic methanogenic archaea have a lower growth rate than the hydrogenotrophic methanogenic archaea.

In contrast to these findings, with an OLR of $2.8 \text{ g VSS L}^{-1} \text{ day}^{-1}$, the propionic acid percentage rose to 86% (2890 mg L^{-1}). Similar findings were reported by Fujishima *et al.*,¹⁸ whereby acetic acid concentrations were mainly on the same level while propionic acid concentrations increased with higher ammonia levels. The authors claimed that increased ammonia levels are mainly responsible for the propionic acid accumulation. However, in their tests, they also applied higher OLRs, which would fit to our findings that the propionic acid accumulation is caused by a delayed conversion of long-chain fatty acids. Acetogenic bacteria, especially propionic acid-degrading bacteria, have a lower growth rate and metabolic rate compared with hydrogenotrophic and acetoclastic methanogenic archaea.²⁴ For this reason, the conversion of propionic acid is delayed at higher OLRs, leading to an acid accumulation in the digested sludge (Fig. 6).

The benefits of the operation with high solids are a lower energy demand for heating up the raw sludge and a reduction of the construction costs through a smaller required digester volume. In summary, a further thickening of raw sludge from 4 to 7%

TSS is regarded as optimum in order to save digester volume and ensure high removal performance. The operation with raw sludge TSS in a range of 6.8–7.0% resulted in TSS of approx. 4.4% in the digested sludge with NH₄-N concentrations in a range of 1850–2040 mg L⁻¹. With this operation, the NH₃-N and organic acids were less, indicating a stable anaerobic digestion. Digester operation with 7% TSS in the raw sludge instead of 4% would allow a reduced volume of 42%. Under the assumption that about 45% of the construction costs arise for the construction of the digester and 55% of the costs are for the technical equipment (pipes, heat exchangers, CHP), the total construction costs can be reduced by approx. 20%.

Finally, it should be mentioned that the flow characteristics of the sludge are affected by the operation with increased OLRs and enhanced TSS. Füreder *et al.*²⁵ investigated the impact of raw sludge TSS on the rheological parameters and found that increasing the raw sludge TSS from 6.0 to 8.0% leads to twice the shear stress and thrice the friction loss. These findings confirm that the high-solid conditions can have major effects on pipe dimensioning and pumping and should be considered carefully for the infrastructure of anaerobic digesters.

CONCLUSIONS

In this study, it was found that high-solid digestion is a possible and efficient strategy to optimize the digester volume and to reduce the construction costs substantially. Stable removal and methane production were found with NH₄-N levels below 2100 mg L⁻¹. Limitations occur with an NH₄-N level of 3200 mg L⁻¹ and ammonia concentrations above 150 mg L⁻¹. However, the anaerobic process was not completely interrupted owing to high alkalinity and adaptation. Further limitations appeared with an OLR of 2.8 g VSS L⁻¹ day⁻¹ (raw sludge TSS 8.9%), as seen in a declined removal and accumulation of propionic acid. Raw sludge thickening to 7% instead of 4% TSS allows the reduction of the construction costs by 20%. An operation with OLR above 2.2 g VSS L⁻¹ day⁻¹ should not be exceeded in order to ensure a stable mesophilic conversion without the formation of organic acids.

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