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Residues from the dairy industry as co-substrate for the flexibilization of digester operation

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Abstract

Water resource recovery facilities (WRRF) can make an important contribution to increase the share of renewable energies in Germany. In this context, it is important to utilize unused digester capacities on WRRF. In addition, a demand-orientated biogas production could synchronize electricity demand and electricity generation and improve the overall energetic balance of the WRRF. As part of the project "Water Resource Recovery Facilities in interaction with the waste and energy industry: A German-Austrian Dialogue – COMITO," the influence of residues from the dairy industry on the digestion process was examined as well as the suitability for the flexibilization of digester gas production. Four reactors were fed with different amounts of flotation sludge from the dairy industry for several months. The difference in the feed resulted in organic loading (OLR) rates between 3.2 kg COD/(m^3 day) and 6 kg COD/(m^3 day). The reactors were fed with a daily shock load. The investigations showed that volumetric loads up to 4.4 kg COD/(m^3 day) did not lead to an accumulation of organic acids. Organic loading rate of 6 kg COD/(m^3 day) showed a significant accumulation of organic acids higher than 2,500 mg/L oHAc. Nevertheless, the reactor could be operated with a degradation rate of 71% with a corresponding biogas yield with a methane content of 71%. With increasing flotation sludge content, a higher concentration in ammonium of up to 2.000 mg/L NH_4-N could be detected in the effluent of the digester. Despite higher phosphorus concentration in the flotation sludge, the concentration of PO_4-P remained constant for all reactors fluctuating between 20 and 40 mg/L PO_4-P. Dewatering worsened significantly with increasing levels of flotation sludge.

Practitioner points

- Main purpose of the research is to flexibilize digester operation on WRRF using flotation sludges from the dairy industry.
- Flexibilization of the digester using flotation sludge is possible up to an organic load of 6 kg COD/(m^3 day).
- Higher NH_4-N concentration in the effluent of the digester must be accepted when using higher amounts of flotation sludge.
- Phosphate concentration in the effluent of the digester remained on a low level despite higher phosphorus content in the flotation sludge.
- High levels of organic acids (mainly acetic acid) can be tolerated and can be recovered within a short time after reducing the load.

Key words

co-digestion; demand-oriented biogas production; digestion; sewage sludge

INTRODUCTION

The water resource recovery facility as part of the energy transition

SINCE 2000, the share of renewable energies on total electricity production has risen up to 36% in Germany (BMWi, 2018). In January 2018, for the first time in history, the entire electricity demand was covered completely by renewable energies (Bundesnetzagentur, 2018). In order to substitute fossil energy sources as part of the energy transition entirely, the share of renewable energy must continue to increase in future.

However, the rise of renewable energies, in particular solar and wind energy, has an impact on electric grid stability. In a stable network, there is a balance between electricity generation and electricity demand. Wind and solar energy can only be regulated to a limited extent with regard to power generation. As a result, frequency fluctuations can occur leading to a network failure, in the worst case. In order to compensate the fluctuations, current surpluses and current shortages must be compensated by short-term removal or supply of electricity from the grid. Hence, technologies for the storage or flexible provision of energy as well as load management are becoming increasingly important.

Water resource recovery facilities (WRRF) are often the largest energy consumer within municipalities (MUKE, 2015). WRRF > 50,000 population equivalents (PE) produce biogas during digestion, which is converted to electricity and heat via combined heat and power plants (CHP). Thus, WRRF can make a direct contribution to the substitution of fossil fuels. Furthermore, intelligent electric load management on WRRF can contribute to the stability of the electric supply network. After all, organic substrates (sewage sludge, co-substrates etc.) can be considered as energy storage as well. Depending on the long-term stability of the substrates and storage capacities of the WRRF, they can be used for demand-oriented biogas production and power generation.

Flexibility of the biogas production

According to the quantity and quality of the inflow, power consumption in WRRF fluctuates in the course of a day. In contrast, there is a continuous digestion with a constant biogas production and correspondingly continuous power generation.

The aim should be that the feeding takes place in correlation with the expected electricity demand, and synchronization of electricity generation and electricity demand is achieved. Precondition for the demand-oriented feeding is the tolerance of the microorganisms against large variation of organic loads. In literature, constant organic loads are described as prerequisite for a stable digester operation (Rosenwinkel, Kroiss, Dichtl, Seyfried, & Weiland, 2015). In particular for biogas plants, the possibility of a demand-orientated feed was already shown (Mauky et al., 2017; Mulat et al., 2016). Mauky et al. reported long-time process stability at high organic loading rates (OLR) of up to 6 kg TVS/(m^3 day) feeding maize silage and sugar beet (Mauky, Jacobi, Liebetrau, & Nelles, 2015). It has also been shown that longer intervals between feeding events had no influence on specific methane production but increased tolerance to organic overload and ammonium concentration (De Vrieze, Verstraete, & Boon, 2013). Nevertheless, Svensson Paruch Gaby and Linjordet (2018) compared an almost continuous feeding pattern with a once-a-day feeding pattern. The results showed higher methane yields for the continuous fed digester as well as a propionic acid inhibition for the irregularly fed digester at OLR of 21 kg COD/(m^3 day) and a hydraulic retention time of 10 days.

Co-substrates for flexibilization of biogas production

Many WRRF in Germany have free capacities in the digesters (Lensch, Schaum, & Cornel, 2016). These capacities can be used to digest organic residues with high energy content from certain industries (co-substrates) in order to increase biogas production. Co-substrates have been used in Germany for several decades. In addition to the higher organic loads, the composition of the substrates has a major effect on the anaerobic processes during the digestion in particular. Proteins, carbohydrates, and fats have a significant influence on the composition of microbial communities and on sludge characteristic (Trulli & Torretta, 2015). The substances also differ in their degradability. In particular, large amounts of readily degradable oligomers and monomers can rapidly lead to acidification of the digester due to high hydrolysis rates and a comparatively low methanogenesis for unadapted cultures. Especially in co-substrates from the food industry, detergents can be contained in the substrates, which can have a toxic impact on the microorganisms and reduce the biogas yield. High contents in proteins will have a negative impact on dewaterability (Zhang, Lu, Song, & Zhang, 2018). However, high phosphorus concentration in the digestate can result in scaling effects (struvite). Kopp (2018) also showed a negative impact of phosphorus concentration in the digestate on dewaterability (Kopp, 2018). In contrast, substrates rich on structural material like silicates, char or lignin can improve dewaterability (Zhang, Kang, et al., 2019).

For flexibilization of the digestion process, the selection of a substrate plays a decisive role. As part of the project "Water Resource Recovery Facilities in interaction with the waste and energy industry: A German-Austrian Dialogue – COMITO," financed by the European Union, the influence of flotation sludge from the dairy industry on digestion processes was analyzed as well as its potential to flexibilize gas production. The aim of this work was to investigate the suitability of floatation sludge for co-digestion by varying the amount of flotation sludge fed into the experimental reactors and to describe the influence of high organic shock loads on process stability.

Materials and Methods

Substrate

Sewage sludge (SS) was taken from a municipal WRRF with a capacity of 50,000 PE. SS was mixed in a volume ratio of primary sludge and secondary sludge of 1:1. The sewage sludge was weighed according to the daily dosage and stored in the freezer until it was fed into the digesters. Sludge samples were analyzed on the same day of collection. Flotation sludge (FS) was collected from a flotation plant for COD removal at a large dairy company producing yoghurt and mozzarella. The flotation sludge was analyzed on the same day of collection, weighed according to the daily dosage, and stored in the freezer until it was fed into the digesters. The same charge of flotation sludge was used for the entire experiments. Table 1 summarizes the analysis made for SS and FS.

Experimental setup

Semi-technical investigations were carried out in four identical digesters (D1 to D4) with a capacity of 30 liters and a working volume of 25 liters at a constant temperature of 37°C. The reactors were stirred in intervals with a velocity

Table 1. Sewage sludge and flotation sludge characterization

PARAMETER	UNIT	SEWAGE SLUDGE	FLOTATION SLUDGE
TS	%	4	7
TVS	%	74	77
COD_{tot}	mg/L	46,000	148,000
COD_{tot}/TS	mg COD/g TS	1,150	2,114
COD_{tot}/TVS	g COD/g TVS	1.55	2.75
TKN	mg/L	2,410	4,185
TKN/TS	g TKN/kg TS	60	60
P_{tot}	mg/L	985	2,384
P_{tot}/TS	g P/kg TS	25	34
Lipophilic substances	mg/L	–	27,620

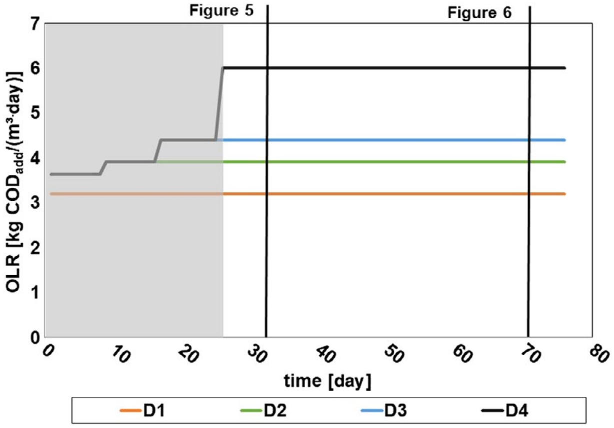

Figure 1. Organic loading rate in D1 to D4 during the experiment.

of 20 rpm. The feed of D2 to D4 consisted of municipal sewage sludge and flotation sludge from the dairy industry. D1 was fed only with sewage sludge and acted as a blank reactor. At the beginning of the experiment, all reactors were filled with the same inoculum from a full scale digester and fed with the same sewage sludge for two months to ensure the comparability of the four reactors. After verifying the comparability, the amount of flotation sludge was steadily increased in reactor D2 to D4 until reaching the target value. After reaching the target values, organic load was kept constant for another 55 days in each reactor. Figure 1 shows the increase of OLR expressed in kg COD/(m^3 day) and kg TVS/(m^3 day) for D1 to D4 during the experiment. The operation parameters are summarized in Table 2.

At the end of the start-up phase, total volatile solids (TVS) load varied between 52 g TVS/day in D1 and 71 g TVS/day in D4. The feeding was done intermittently with high shock loads resulting in OLR of up to 6 kg COD/(m^3 day) in D4. Because of higher total solid content (TS) in the flotation sludge, the TS of the feed increased from D1 to D4. OLR was 3.2 kg COD/(m^3 day) in D1 and 6 kg COD/(m^3 day) in D4. The ratio between COD from flotation sludge (COD_{FS}) and COD from sewage sludge (COD_{ss}) was between 0.4 in D2 and 1.9 in D4. The digesters were fed over a period of 5 months constantly keeping a hydraulic retention time (HRT) of 20 days for all reactors. Feeding took place once a day on 5 days per week.

Analytical methods

Both SS and FS were analyzed for TS, TVS, COD, TKN, and P. Additionally, lipophilic substances were measured in FS. Digested sludge from D1 to D4 was monitored continuously during the experiment. TS and TVS were analyzed gravimetrically by drying at 105°C and subsequent burning at 550°C. Analysis was taking place on a daily basis. PO_4-P and NH_4-N were analyzed weekly using a continuous flow analyzer (CFA; Bran+Luebbe Auto Analyzer III. Norderstedt, Germany)

Table 2. Averaged operation parameters for D1 to D4

PARAMETER	UNIT	D1	D2	D3	D4
Temperature	°C	37			
Feed	kg/day	1.75			
HRT	Day	20			
COD_{FS}/COD_{SS}	–	–	0.4	0.75	1.89
TS_{in}	%	4.0	4.3	4.5	5.3
TVS_{in}	%	76.9	76.4	76.6	76.8
COD/TVS	g COD/g TVS	1.55	1.74	1.86	2.15
Load COD	g COD/day	80.0	98.0	110.0	150.0
Load TVS	g TVS/day	51.8	56.5	58.4	70.6
OLR	kg TVS/(m^3 day)	2.07	2.26	2.34	2.83
OLR	kg COD/(m^3 day)	3.20	3.92	4.40	6.00

after filtrating the sample with a 0.45-μm syringe filter. COD_{tot} was analyzed once a week using cell test (Spectroquant, Merck, Darmstadt, Germany). The organic acids were measured at least once a week using gas chromatography (Agilent Technologies 6890N; capillary column Agilent J&W HP-FFAP, Santa Clara, California) after filtrating the samples with a 0.45-μm syringe filter. Gas quantity was recorded via online gas counter (Ritter TG 0.5, Bochum, Germany), and the values were normalized to standard temperature and pressure conditions. Gas quality (CH_4, CO_2) was measured once a week using μGC (Agilent Technologies 490 Micro GC, Santa Clara, California). All analyses were conducted according to the analytical methods specified by the German Institute for Standardization (DIN), which are in accordance with the APHA Standard methods. For the COD analysis, the recommendations of Schaum Rühl Lutze and Kopf (2016) were complied. Table 3 shows the analysis and intervals of the measurements during the experiment.

In order to investigate the influence of the flotation sludge on dewaterability of the digested sludge, capillary suction tests (CST) were carried out using a Triton—W.P.R.L. Type 92/1. The test was conducted according to the APHA Standard methods.

RESULTS AND DISCUSSION

Process performance

Conventionally, digestions are mainly balanced and dimensioned via TVS as well as the HRT. This is especially applicable if substrates comparable to sewage sludge regarding the composition are used, in particular based on the COD/TVS ratio. As soon as co-substrates deviate from this, as in the present case (cf. Table 1), this type of balancing is no longer applicable. In this case, a balance based on the COD should be preferred. In Figure 2, a COD balance for D1 to D4 between day 32 and 69 is demonstrated. The balance shows the measured and calculated COD loads for the feed, the effluent, and the degraded COD. The degraded COD (COD-deg) was determined by the difference between the COD measured in the feed (COD-input) and the COD-output (COD measured in the effluent of the digester). To cross check the results, the biogas content was also converted into a COD load (COD-biogas) using the conversion factor of 0.35 m^3 CH_4/kg COD (Tchobanoglous, 2003). Ideally, COD-deg and COD-biogas should be identical. In the present mass balance, there is a difference of <20% between these two values, indicating a high conformity. As seen in Figure 2, degradation rate varied between 51% in D1 and 76% in D4. Based on the COD calculation, an inhibition of anaerobic degradation is not apparent for neither reactor despite the high OLR of up to 6 kg COD/(m^3 day). According to DWA (2014), a OLR of 1.8–2.6 kg COD/(m^3 day) is recommended for WRRF of the size of 50,000–100,000 PE. With an increase of the flotation sludge fraction, the degree of degradation increased from 51% in D1 to 76% in D4.

Table 3. Overview of analyzed parameters

	UNIT	PARAMETER	INTERVAL
Input	%	TS, TVS	Day of sampling
	mg/L	COD_{tot}	Day of sampling
	mg/L	P_{tot}, TKN	Day of sampling
	mg/L	org. acid	Day of sampling
Output	%	TS, TVS	Daily
	mg/L	COD_{tot}	Weekly
	mg/L	PO_4-P, NH_4-N	Weekly
	mg/L	org. acid	Weekly/daily
Gas	%	CH_4, CO_2, H_2S	Weekly

Figure 3 shows the mean values of digester gas production for D1 to D4. The shaded area marks the start-up phase. During this phase, the OLR was steadily increased to the target levels. It can be seen that an increase in COD load due to higher amount of flotation sludge in the feeding results in higher gas production. Specific gas production was 269 Nl/kg TVS (208 NL/kg COD) in D1 and 540 Nl/kg TVS (255 Nl/kg COD) in D4.

Figure 4 shows the methane concentration during the experiment. In D1, a methane concentration of 63% in average was observed for the entire experiment. After the OLR has been increased by 0.7 kg COD/(m^3 day) by feeding flotation sludge, methane concentration increased to 66% in D2 to D4 (COD_{FS}/COD_{SS} = 0.4). After a further increase in the amount of flotation sludge in D3 and D4, a methane concentration of 68% at a COD_{FS}/COD_{SS} ratio of 0.75 and 71% at a ratio 1.94 could be observed, respectively. The increasing methane concentration of D1 to D4 can be related to the higher COD/TOC ratio of the flotation sludge compared to the sewage sludge which were 4.0 and 3.0, respectively.

Measurements showed that due to a high proportion of lipophilic substances, the energetic potential of flotation sludge is with 2.75 g COD/g TVS high in comparison with sewage sludge (1.55 g COD/g TVS) (cf. Table 1). Concurrently, both nitrogen and phosphorus content referred to the TS are more or less similar to sewage sludge.

In Figures 5 and 6, the gas production after a feeding event at 0 hr is plotted. Figure 5 shows the course for day 32, while Figure 6 shows the course for day 69 (cf. Figure 1). Within minutes, an increase in production can be observed for both

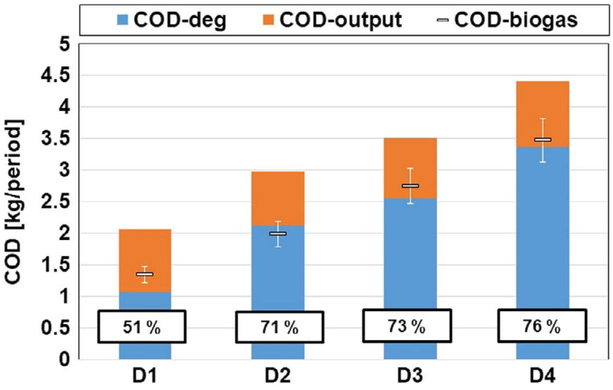

Figure 2. COD balance and degradation rate in D1 to D4 between days 25 and 60. COD-output being the COD load measured in the effluent. COD-deg being the difference between the COD load in the influent and the effluent. COD-biogas being a COD-equivalent calculated from the produced methane gas.

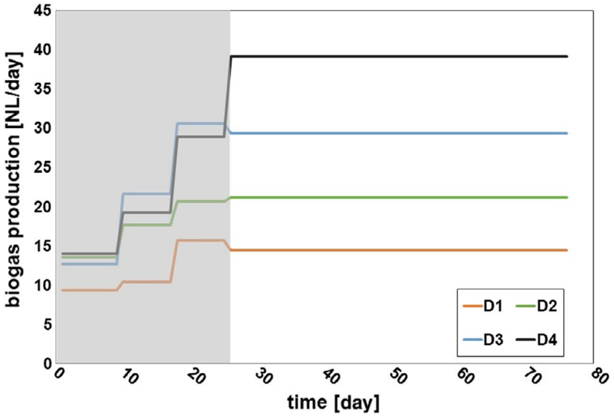

Figure 3. Averaged gas production in D1 to D4 during the experiments.

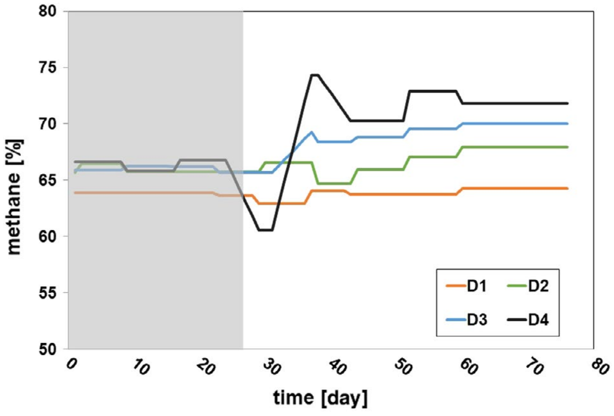

Figure 4. Averaged methane concentration in the gas phase of D1 to D4.

periods. The maximum production is reached within 2.75 hr for day 69 and 2.0 hr for day 32. Due to higher COD concentration in SS at day 32, higher biogas production is observed for D1, D2, and D3 in Figure 5 compared to Figure 6. For D1, D2, and D3, similar patterns can be observed especially in Figure 6. The course can be divided into 5 phases. In the first phase, a drop in biogas production is observed, which should be due to pressure equalization during feeding. In the second phase, there is a linear increase within one hour. In the third phase, a linear increase with a lower slope compared to phase 2 is observed reaching a maximum at 2.75 hr. After a short drop in biogas production in phase 4 being deeper in Figure 5, a sigmoid function can be observed in phase 4. The last phase is characterized by a slight linear decline in biogas production. In Figure 5, the similarities between D1, D2, and D3 are less clear. In Figure 4, D4 has a minor gas production compared to D3 which indicates inhibition that could be due to the high organic acid or the ammonium concentration.

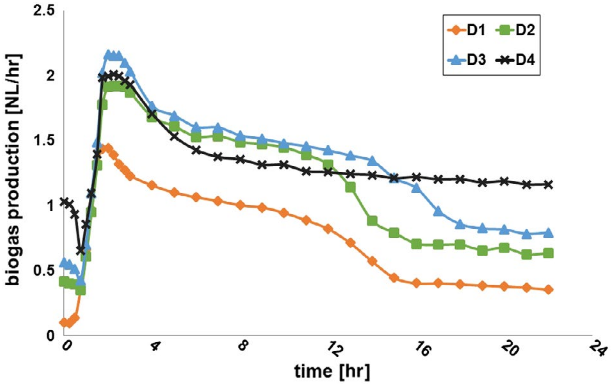

Figure 5. Biogas production after shock load (at time 0) at day 32.

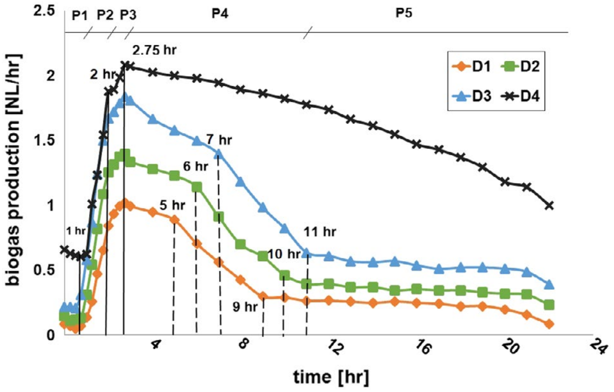

Figure 6. Biogas production after shock load at day 69.

Organic acid concentration was <50 mg/L oHAc in D1 to D3 during the experiment. After increasing the organic load in D4 for 40 g COD/day compared to D3, organic acid accumulation was observed. The organic acid concentration reached a peak of >2,500 mg/L oHAc. Approximately 90% of the organic acid was contributed to acetic acid while 4% were attributed to propionic acid. These findings are in contrast to the observations of Svensson Paruch Gaby and Linjordet (2018). In these experiments, propionic acid was the main constituent of organic acid. The differences could be due to the difference in substrate used. Afterward, a steady decline in organic acid could be observed. Forty-five days after increasing the OLR to 6 kg COD/(m^3 day) in D4, a steady state was noticed. Organic acid concentration was comparable to the other reactor from then on.

Effect of co-substrate characteristic on the digestion process

Figure 7a shows the mass balance of TKN in D1 to D4 within 35 days. TKN-SS-in is the TKN load introduced into the digester via the SS, while TKN-FS-in is the TKN load introduced by the flotation sludge from 100 g TKN/period to above 140 g TKN/period. An increase in nitrogen load can be observed with increasing fraction of flotation sludge. With increasing nitrogen input, an increased ammonium load in the effluent of the digester can also be ascertained. However, it becomes clear that the level of ammonium load is not correlated with the COD degradation level, which is also due to the different TS values in the feed and the effluent. The N concentration in the digested sludge remains almost constant having a concentration of

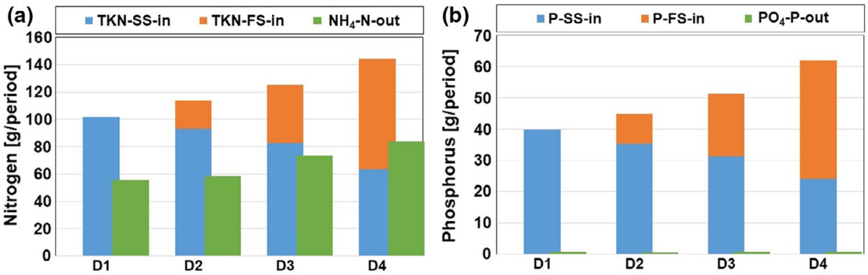

Figure 7. Balance of nitrogen load (a) and phosphorous load (b) between days 25 and 60. TKN/P-SS-in represents the respective loads introduced to the digesters via sewage sludge while TKN/P-FS-in are the loads introduced to the digester through flotation sludge. NH4-N/PO4-P being the respective loads in the effluent.

50 mg TKN/g TS in D1, D3, and D4, while in D2, a concentration of 60 mg TKN/g TS is observed. Despite a high ammonium concentration of up to 2,000 mg/L NH_4-N in D4, inhibition of methanation was not observed in the long term considering the high methane content of >70%. Nevertheless, a significant increase of ammonium concentration from 800–1,200 mg/L NH_4-N in D1 to 1,500–2,000 mg/L NH_4-N in D4 has a major impact on the biological step of WRRF and has to be taken into account when using flotation sludge.

The concentration in the effluent of the digester was fluctuating between 20 and 40 mg/L PO_4-P for D1 to D4. Although the daily load of phosphorus increased from D1 and D4, no significant difference could be observed in the effluents of D1 to D4. Figure 7b shows the total loads of phosphorus in the feed and in the effluent between days 25 and 60. Measurements of the effluents showed that the molar ratio of Fe:P, Ca:P as well as Mg:P was on a high level for each reactor, so that incorporation of phosphate in (hydroxyl-)apatite, struvite or the adsorption on iron or aluminum could be assumed as described by Bratby (2018) especially if the increase in pH from D1 (7.0) to D4 (7.7) is considered. Fe:P ratio decreased from D1 to D4 from 12:1 to 9:1. The molar ratio of Mg:P remained constant between 9:1 and 10:1, while Ca:P increased from 38:1 in D1 to 65:1 in D4.

Due to the high ability of phosphorus uptake by the digested sludge, the risk of phosphorus accumulation in the process water and exceeding permitted discharge values while using flotation sludge is low. During the experiment, PO_4-P concentration in TS increased from 33 mg P/g TS in D1 to 47 mg P/g TS in D4. According to Kopp & Benisch, (2018), an increase of PO_4-P concentration increases the water binding. Nevertheless, there are plenty of parameters that can have an effect on dewaterability as high protein or exopolymeric substances (Li & Yang, 2007). In order to estimate the influence of flotation sludge on dewaterability, capillary suction tests (CST) were conducted. Figure 8 shows the results of the investigations. A significant decrease in dewaterability as a function of the amount of flotation sludge can be observed. It can be assumed that high protein concentrations in the flotation sludge could decrease the dewaterability as well as higher P concentration in the digested sludge. Furthermore, it must be pointed out that the CST test may not be a sufficient accurate indicator for dewaterability when examining substrates with high fat or protein contents with hydrophobic character. This has to be validated in further examinations and has to be compared with other methods.

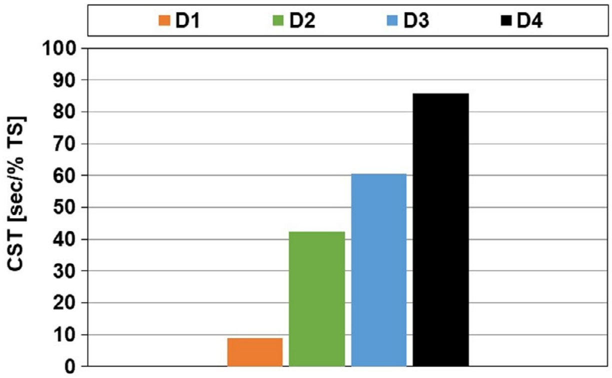

Figure 8. Capillary suction tests for D1 to D4.

Conclusion and Outlook

Due to their high energy content, flotation sludge is very well suited to make the operation of digesters more flexible. The adaptation time is short and shock load feeding is possible without serious decline in biogas production and quality. However, a balance between nutrient supply and turnover rate was reached after 73 days. So a certain degree of sensitivity is needed during that phase to avoid digester failure. However, with the addition of high amounts of flotation sludge, a rise in NH_4-N concentration in the output can be observed, and advanced nitrogen elimination from process water should be discussed, when systematic flotation sludge feeding is intended. Furthermore, it can be summarized:

- Flexibilization of the digestion reactors by flotation sludge is possible up to an organic load of 6 kg COD/(m^3 day) or even more for adapted sludge.
- High levels of organic acids (mainly acetic acid) can be tolerated and can be recovered within a short time after reducing the load.
- Higher NH_4-N concentration in the effluent of the digester must be accepted when using higher amounts of flotation sludge. However, the concentration in the effluent does not increase in the same proportion as the concentration of nitrogen in the feed.

- Phosphate concentration in the effluent of the digester remained on a low level despite higher phosphorus content in the flotation sludge.

Further studies comparing continuous feeding and shock load feeding should be undertaken, especially to compare specific methane gas yield at different feeding patterns, as well as the effects on the dewaterability and the release of ammonium. In addition, further investigations will be carried out in order to investigate whether overloading can be predicted on the basis of characteristic biogas production courses.

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