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Measures to prevent foam formation in the anaerobic digestion of sugar beet in biogas plants

Lucie Moeller, Andreas Zehnsdorf

The occurrence of persistent foaming is observed in many anaerobic digesters that have sugar beet as their feedstock. The formation of foam entails a significant risk of damage to biogas plants, as gas pipes can become blocked. For this reason, foaming tests have been conducted to investigate which measures lead to reductions in foam development. It was found that generally available fertilizers such as urea, ammonium nitrate and calcium cyanamide have a foam-reducing effect. However, batch fermentation tests showed inhibition of biogas production at higher concentrations of these substances, which means that they should be used with care. Calcium cyanamide was found to be very unsuitable, as this substance inhibited biogas production even at low concentrations and caused the fermentation process to come to a complete stop at higher concentrations.

Keywords
Anaerobic fermentation, biogas, foam, sugar beet, nitrogen fertilizers, urea

Sugar beet is a suitable alternative to maize and grain for the operators of biogas plants in the context of §27 para. 5 no. 1 of the 2012 German Renewable Energy Sources Act (the so-called 'maize cap'). However, there have been numerous reports of foam formation during the operation of fermenters in connection with the co-fermentation of sugar beet. A large-scale survey of 3,100 biogas plants by Lindorfer and Demmig (2016) found that sugar beet accounted for 6% of foam-forming substrates. Various effects of process operation policies on foam formation in the fermentation of sugar beet have been discussed in the literature. Suhartini et al. (2014) observed that the tendency of the fermentation material to foam increased with increasing organic loading rates under mesophile conditions. Lindorfer and Demmig (2016) stated that the risk of foam formation increases strongly for sugar beet fractions of over 20% (w/w) in the substrate mix (relative to the wet mass). However, the authors also speak of biogas plants where no foam was formed even at 30% (w/w) sugar beet in the substrate mix. Suhartini et al. (2014) showed that no foam formation occurred under thermophile conditions on a laboratory scale even with organic loading of 5 g VS/(L · d). Stoyanova et al. (2014) demonstrated that less foam was formed in a two-stage pilot-scale biogas plant than in a one-stage process with the same organic loading rate. However, the results of the experiments by Suhartini et al. (2014) and Stoyanova et al. (2014) are of little relevance for existing plants. Most of the agricultural biogas plants in Germany are one-stage and are operated in the mesophile temperature range (Eder and Schulz 2007).

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In the sources listed above, various substances have been identified as the cause of foam formation in the fermentation of sugar beet. Suhartini et al. (2014) found extracellular polymer substances in the foam that are not described in any further detail. Stoyanova et al. (2014) identified pectins that originate in the sugar beet as substances that increase the viscosity of the fermentation material. Our previous studies have shown that both pectins and sucrose can lead to foam formation in anaerobic fermentation (Moeller et al. 2015a). However, the foaming tendency of mixtures of these two substances in anaerobic fermentation is stronger than that of the individual substances. Knowledge of this fact is important, as sugar beet contains both sucrose and pectins. Moeller et al. (2015a) also described how the intensity of foam formation caused by the fermentation of sugar beet can be influenced by various substances. The addition of salts with divalent cations led to increased foam formation, while the addition of nitrogen-containing compounds such as urea and ammonium chloride reduced foaming and changed the foam structure. Additional investigations are necessary before these findings can be put into use in practice. As a result, the aim of the work described here was to identify the influence of various nitrogen fertilizers on the intensity of foam formation caused by sugar beet. Another goal was to investigate the influence of the addition of nitrogen fertilizers on biogas production in order to avoid a reduction in biogas yields due to the additives.

Materials and methods

Substrates and fermentation material

The sugar beet silage (total solids (TS): 18.9% fresh mass (FM) and volatile solids (VS): 79.4% TS) was provided by the German Biomass Research Centre (DBFZ). The digestate for the foaming experiments originated from an agricultural biogas plant close to the town of Grimma, while the digestate for the determination of the biogas potential came from an agricultural biogas plant close to Leipzig that mainly uses manure. The properties of the fermentation materials are described in Table 1.

Table 1: Properties of the initial digestate for foaming experiments and determination of biogas potential

	TS in % FM	VS in % TS	Acetat in mg/L	Propionat in mg/L	Butyrat in mg/L
Digestate for foaming experiments	7.31	78.9	176	14	< 1
Digestate for determination of biogas potential	4.54	66.3	18	< 1	< 1

FM = fresh mass, TS = total solids, VS = volatile solids

Analyses

The content of total solids (TS) and volatile solids (VS) were determined in accordance with DIN 12880 and DIN 12879. The samples of fermentation material from the experiments were passed through a sieve (mesh size 0.75 mm) for further analysis. The samples prepared in this way were then analyzed for total organic carbon (TOC) and total nitrogen (TN) concentrations using a TOC/TN measurement device (TOC-VCSH/CSN analyzer with a TN unit, Shimadzu, Japan), and for pectin content (as galacturonic acid (GA) equivalent in g_{GA}/kg), as described in Moeller et al. (2015a). The sieved sample was centrifuged (20 min, 5300 rpm and 20 °C, Avanti 30 Centrifuge, Beckman, Brea, USA) and the centrifuged sample was filtered by means of pressure filtration (SM 16 249 pressure filtration unit, Sartorius, Göttingen, Germany, nylon membrane filter: pore size 0.45 µm, Whatman, Germany). The concentrations of organic acids in the filtered sample were measured using high-performance liquid chromatography (HPLC; with an RID-10A detector, VA 300/7.8 nucleogel ion 300 OA column and 0.01 N H_2SO_4 as the eluent, Shimadzu, Japan) and ammonium-nitrogen was measured in accordance with DIN 38406 E5 (Spectroquant test kit with a measurement range of 0.01–3 mg/L NH_4-N, Merck, Germany).

Batch foaming experiments

The foaming experiments were carried out as described in Moeller et al. (2015b). The fresh digestate (Table 1) was passed through a 10 mm sieve. 40 g of sugar beet silage (corresponds to an organic loading of 13 g/(L · d)) and a defined amount of additive depending on the experiment in question were weighed out in a 1 L Schott bottle. The compounds selected as additives were primarily substances that are also used as fertilizers in agriculture. These were ammonium nitrate (Merck KGaA, Germany), ammonium sulfate (Riedel de Haën, Germany), urea (Riedel de Haën, Germany), potassium nitrate (Merck KGaA, Germany) and pearled calcium cyanamide (Beckmann & Brehm, Germany). In the first foaming experiment, 2.5 g/kg of nitrogen-containing compounds were added to each foaming test. In the second foaming experiment, 0 g/kg, 0.25 g/kg, 0.5 g/kg, 1.25 g/kg, 2.5 g/kg and 5 g/kg of urea or ammonium nitrate were used for the foaming tests. Foaming tests with 0.5 g/kg or 2.5 g/kg of sodium hydrogen carbonate and 0.5 g/kg or 2.5 g/kg of sodium carbonate were also carried out.

The mixture was made up to 500 g using fermentation material, thoroughly stirred and heated in a water bath at 38 °C. The bottle top was only half closed so as to provide pressure relief. The intensity of foam formation was evaluated after 18 hours of reaction time. Each test variant was carried out at least twice. In addition, a control test with 500 g of fermentation material with no additives was also carried out. As sugar beet does not form a clear foam layer in the fermentation material, the difference in the volume of each foaming test and the control test relative to the volume change in the foaming test with sugar beet alone was considered for evaluation purposes (Equation 1):

$$Relative\ increase\ in\ volume\ in\ \% = \frac{(h_{test} - h_{control})\ \pi\ (\frac{d}{2})^2}{(h_{SBS} - h_{control})\ \pi\ (\frac{d}{2})^2} \cdot 100 \qquad \text{(Eq. 1)}$$

h: Height of fermentation material in the test bottle at the end of the experiment (control = test bottle with digestate with no addition of substrates or additives; SBS = Foaming test with addition of 20 g/kg of sugar beet silage; test = Foaming test with addition of 20 g/kg of sugar beet silage and the substance to be tested)
d: Diameter of the test bottle (d = 8.5 cm)

Batch fermentation tests

The batch fermentation tests were carried out in accordance with the VDI 4630 guideline. The fermentation material (Table 1) was first passed through a 5 mm sieve and then allowed to ferment for a week at 37 °C in an incubator. 240 g of fermentation material was mixed with 5 g of sugar beet silage and a corresponding amount of additives for the batch fermentation tests. Each test variant was carried out three times. The tests were incubated in a water bath at 37 °C and the biogas produced was collected in glass columns with acidified saturated saline solution. The bottles were shaken briefly once each day. Biogas formation was read off each day and the methane concentration in each test was measured twice a week using gas chromatography (Agilent GC 6850 WLD, Agilent Technologies, USA). The biogas volume was adjusted to standard conditions (273.15 K, 1.01325×10^5 Pa).

Results and discussion

The aim of the first foaming experiment was to investigate the influence of the addition of nitrogen-containing compounds on the intensity of foam formation during the fermentation of sugar beet. It was found that all nitrogen fertilizers used had the effect of reducing foam formation (Figure 1). The strongest effect was shown by calcium cyanamide, which almost completely suppressed foam formation. The weakest effect was demonstrated by ammonium sulfate, which reduced foam formation by 42%, followed by potassium nitrate, which caused a reduction in foam formation of 57% relative to the foaming test with sugar beet silage.

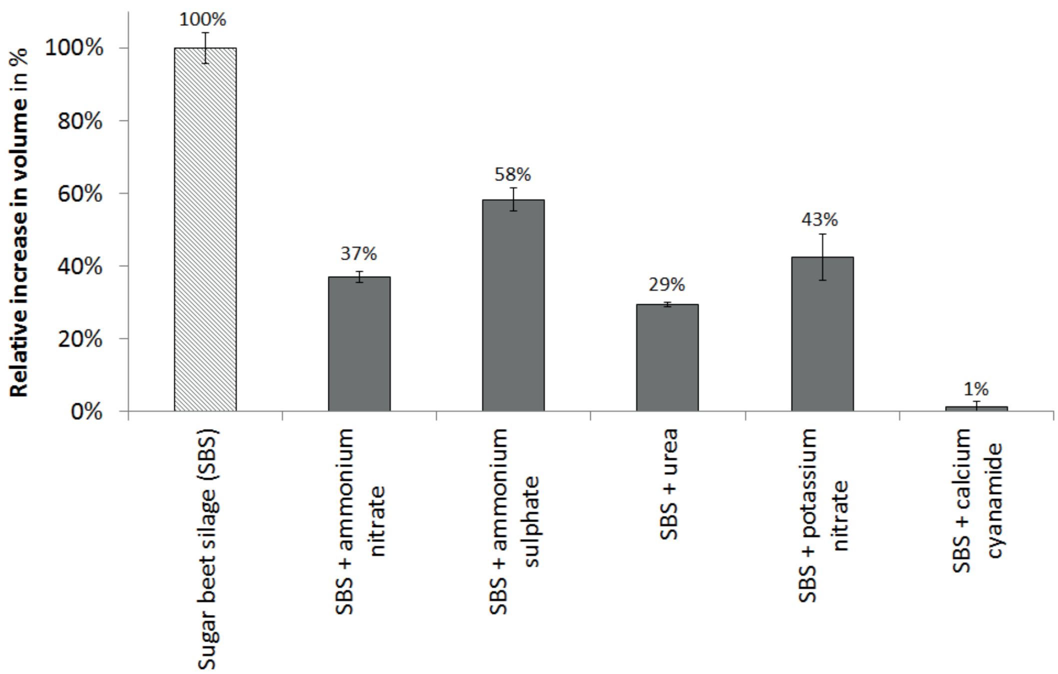

Figure 1: Relative increase in volume in foaming tests with the addition of 2.5 g/kg of nitrogen-containing compounds

Knowledge of how biogas production is influenced by these additives is necessary for the use of nitrogen fertilizers for reducing foam formation in biogas plants. For this reason, the biogas potential of sugar beet in combination with the three most effective additives of calcium cyanamide, urea and ammonium nitrate was determined (Table 2). The results show that the addition of small amounts of urea and ammonium nitrate of 0.25 g/kg to the experimental tests had no negative influence on biogas formation. In the case of calcium cyanamide, even a low mass fraction of 0.25 g/kg resulted in a biogas yield that was reduced by 23% relative to sugar beet silage with no additives. However, biogas production was inhibited with increasing mass fraction for all additives. The strongest inhibition was caused by calcium cyanamide, for which biogas was no longer formed above an amount of 1.25 g/kg. In the case of 2.5 g/kg of calcium cyanamide, it was not possible to take a sample of the gas phase, as the gas tubes were blocked with liquid as a result of the vacuum in the fermentation bottle. When the ammonium nitrate amount was increased to 1.25 g/kg, inhibition of biogas production was stronger than in the case of urea. In the case of urea, it was also observed that the methane fraction of the biogas increased with increasing urea content. The final pH value of the fermentation material also showed a slight tendency to increase with increasing dosage for urea and ammonium nitrate.

Table 2: Biogas potenital, methane content in biogas and final pH value in tests with 12.5 g/kg of sugar beet silage (SBS) in combination with various mass fractions of the nitrogen-containing compounds urea (UR), ammonium nitrate (AN) and calcium cyanamide (CC)

Test	Biogas yield	Methane concentration in biogas at end of experiment	pH value at end of experiment
	in L/kg VS	in % CH_4	-
Urea (UR):			
SBS	735 ± 43	59 ± 2.0	7.21 ± 0.00
SBS + 0.25 g/kg UR	728 ± 21	59 ± 1.9	7.23 ± 0.01
SBS + 1.25 g/kg UR	571 ± 15	60 ± 3.0	7.34 ± 0.01
SBS + 2.5 g/kg UR	349 ± 78	65 ± 0.1	7.53 ± 0.01
Ammonium nitrate (AN):			
SBS	677 ± 16	59 ± 0.4	7.28 ± 0.06
SBS + 0.25 g/kg AN	688 ± 36	58 ± 1.3	7.25 ± 0.01
SBS + 1.25 g/kg AN	494 ± 2.6	61 ± 1.1	7.33 ± 0.02
SBS + 2.5 g/kg AN	431 ± 1.7	59 ± 1.0	7.48 ± 0.02
Calcium cyanamide (CC):			
SBS	752 ± 0.0	58 ± 0.0	7.17 ± 0.01
SBS + 0.25 g/kg CC	577 ± 34	59 ± 2.8	7.18 ± 0.02
SBS + 1.25 g/kg CC	0	21 ± 1.6	6.93 ± 0.01
SBS + 2.5 g/kg CC	0	Could not be determined	7.14 ± 0.05

The methane potential of sugar beet silage was between 405 and 436 L CH_4/kg VS and was thus within the range of data already published. Gissén et al. (2014) determined a methane potential of 419 m^3 CH_4/t VS for sugar beet. The Bavarian State Research Center for Agriculture calculated that the theoretical biogas potential of fresh sugar beet should be around 696 L/kg VS (LfL 2016). At an average of 721 L/kg VS, the potential gas productivities determined here are above this theoretical value.

Nitrate is an electron acceptor in energy metabolism and thus does not act as competition for microbiological methane synthesis (Zumft 1997). However, Sheng et al. (2013) observed that the addition of less than 0.75 g/L of nitrate nitrogen leads to no inhibition of methane production. The addition of 0.5 g/L NO_3-N even led to an increase in the methane yield of 11.8% relative to the test with no addition of NO_3-N. In the case of the results presented in Table 2, it was not possible to confirm this observation as the difference between the biogas yield of SBS and of SBS with the addition of 0.25 g/kg of ammonium nitrate was only marginal. The use of ammonium nitrate-urea solution (AUS with 28% nitrogen) for foam reduction in the fermentation of sugar beet silage in a full-scale plant has been documented in Moeller et al. (2016). No negative influence on the amount of electricity produced each day was observed that would suggest a negative effect on biogas production.

It was observed from the experiments for the determination of the potential gas productivities that calcium cyanamide is not suitable for use as a source of nitrogen in biogas plants. The results showed that the reduction in foam formation in this case was a consequence of strong inhibition of biogas production. It was also shown that biogas production is already inhibited for the fractions of urea and ammonium nitrate (2.5 g/kg) that were used in the foaming experiment (Figure 1). In order to rule out the possibility that the disturbance in biogas production was the reason for the reduction in foam formation in this case too, a foaming experiment with various dosages of urea was carried out. It was demonstrated here that even low amounts of urea had a foam-reducing effect (Figure 2).

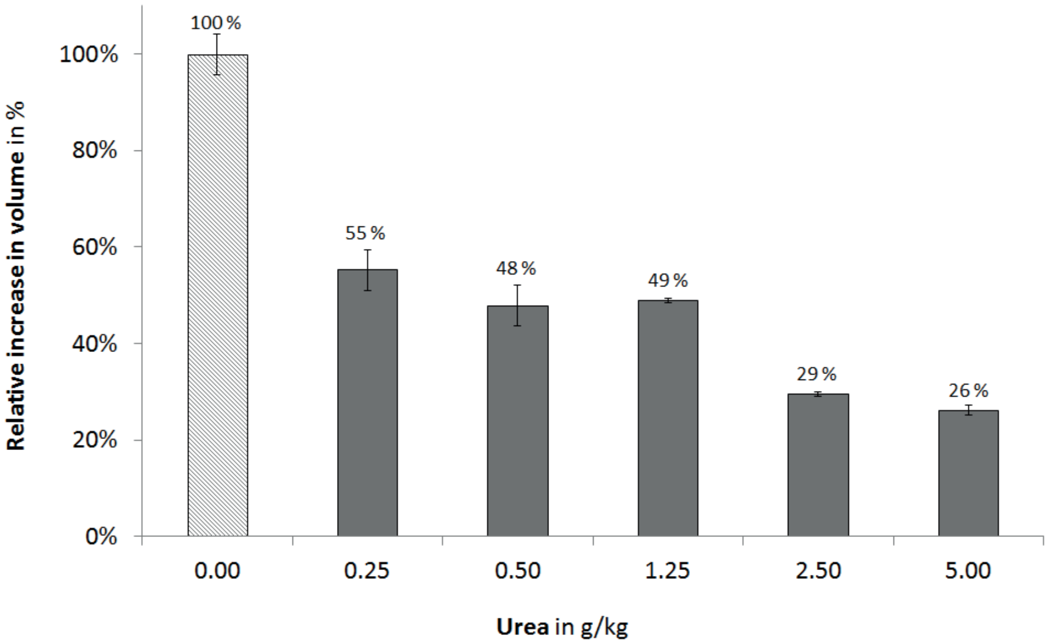

Figure 2: Relative increase in volume in foaming tests with 20 g/kg of SBS and 0 to 5 g/kg of urea

The addition of 0.25 g/kg of urea to foaming tests (corresponding to a urea-to-SBS ratio of 1 : 12 in terms of TS) led to a reduction in the relative increase in volume of 42% compared to SBS without the addition of urea. Further increases in the urea fractions improved the foam-reducing effect only a little at first; however, the foam reduction became more pronounced above 2.5 g/kg of urea (corresponding to a TS ratio of urea to SBS of 1 : 1.2). This is a result of the onset of inhibition of biogas production, as described for the TS ratio of urea to SBS of 1 : 1.5 for 0.25 g/kg of urea in the experimental test in the measurement of the potential fermentation productivity, where less SBS was added (Table 2).

The effect of urea on the anaerobic digestion of SBS was investigated in more detail by analysing fermentation products and foams from the foaming tests with 1) 2.5 g/kg of urea, 2) 20 g/kg of SBS and 3) 2.5 g/kg of urea with 20 g/kg of SBS. Firstly, the appearance of the foaming tests was evaluated. The surface of the foam in the foaming tests with SBS with no addition of urea had an inhomogeneous structure, was bright in color and had large gas bubbles with irregular shapes. The foam formed after the addition of urea to the SBS looked completely different. The surface of the foam was smooth and dark, and the gas bubbles were smaller and more homogeneously distributed. The lower part of the foam layer was bright in color with a structure similar to that of the foaming test with SBS without urea.

The analysis results showed that urea caused the pH value to increase by 0.6 relative to the reference test (Table 3). This difference can also be seen for the foaming tests with SBS: the difference in the pH values of the fermentation products was 0.5 and that of the foams was 0.6. The addition of urea also led to a significant shift in the TOC/TN ratio. SBS caused an increase of 1.4 in the TOC/TN ratio in the fermentation product relative to the reference test, while nitrogen-rich urea caused a decrease of 1.39 in the TOC/TN ratio of the fermentation product compared to the reference test. Urea in combination with SBS reduced the TOC/TN ratio of the fermentation product by 0.9 relative to the reference test. The addition of urea also caused an increase in the concentration of ammonium-nitrogen. This effect could explain the inhibition of biogas production at higher urea concentrations, as presented in Table 2.

Table 3: Results of analysis of the fermentation products and foams from foaming experiments with 20 g/kg of sugar beet silage and 2.5 g/kg of urea in various combinations

	pH	**TOC in g/L**	**TN in g/L**	**TOC/TN**	**NH_4-N in g/L**
Reference (fermentation product)	8.4	75	23	3.27	0.79
Urea (fermentation product, *no foam*)	9.0	196	104	1.88	1.58
Sugar beet silage (SBS)					
Fermentation product	7.9	203	43	4.72	0.83
Foam	8.2	207	44	4.69	0.82
SBS + urea					
Fermentation product	8.4	174	72	2.36	1.80
Foam	8.8	192	82	2.30	1.79

Based on the analysis results, two mechanisms for the effect of urea can be assumed: a pH-increasing effect and a correction of the C/N ratio. These assumptions will be discussed here.

Lindorfer and Demmig (2016) stated that the effect of urea is based on a buffering of the pH value in the fermenter by means of binding of the volatile organic acids that are formed. The authors proposed that effect of urea was due to the increasing of the alkalinity of the contents of the fermenter. This occurs as a result of the enzymatic transformation of urea to ammonium carbonate. Indeed, a significantly higher ammonium-nitrogen concentration was measured in the foaming test with urea with no SBS relative to the reference test (Table 3). The pH value also increased markedly in this test. This tendency was also observed in the case of ammonium nitrate (Table 2).

A check performed involving a foaming test with 0.5 g/kg of ammonium carbonate and 20 g/kg of SBS showed a relative increase in volume of 46%. Thus the effect of 0.5 g/kg of ammonium carbonate on foam formation with sugar beet was similar to the effect of urea (Figure 2). The pH value in the foaming test with 0.5 g/kg of ammonium carbonate increased by 0.3 relative to the foaming test with SBS. An increase in ammonium carbonate to 2.5 g/kg led to a further increase in the pH value of 0.3 with a marginal reduction of foam formation (relative increase in volume: 42%).

Lindorfer and Demmig (2016) also identified sodium carbonate and sodium hydrogen carbonate as buffering substances that are used alongside urea in biogas plants. Our own investigations showed that the addition of 2.5 g/kg of Na_2CO_3 led to a significant increase in the pH value of 0.7. In addition, the relative increase in volume was just 47%. The foam bubbles were significantly smaller and more regular than in the case of the foaming experiment with SBS without additives. The effect of sodium hydrogen carbonate was not as strong as that of sodium carbonate. For the same relative amount of additive, the increase in the pH value was just 0.3 and the relative increase in volume was 72%.

The second assumption was that urea increases the nitrogen content in the fermentation material and thus improves the C/N ratio of the substrate mix in the case of carbon-rich substrates. The important role played by the C/N ratio of the substrate mix in anaerobic fermentation is well established. An optimal ratio of carbon to nitrogen of between 30 : 1 and 10 : 1 has been quoted (Eder and Schulz 2007). If the ratio is too high, there will be a lack of nitrogen as the decisive building block in the production of enzymes. These are essential for hydrolysis of macromolecules such as proteins and carbohydrates. This causes an increase in the amount of these polymer substances that stabilize the biogas bubbles that are produced. As a result, foam is created or else the fermenter contents become bloated. Sugar beet is a carbon-rich substrate. Eluates of cut sugar beet had a C/N ratio of up to 31 : 1 (Moeller et al. 2015a), and – in the case of finely grated sugar beet – even of up to 48 : 1. Sugar beet contains 76.8% water, 14% sucrose and 5.5% pulp (FAO/EBRD 1999). The pulp is not soluble in water and contains 21.5–23% pectins (uronic acids) and 7–8% protein (Michel et al. 1988), which have been linked with foam formation in anaerobic fermentation (Ganidi et al. 2009, Wang et al. 2012). Sucrose and pectins are carbohydrates that contain no nitrogen. Analyses of the fermentation products in foaming experiments have shown that the use of SBS as a substrate resulted in an increase in pectin content from 0.118 g_{GA}/kg in the reference test to 0.263 g_{GA}/kg. However, if the sugar beet was mixed with urea, the amount at the end of the experiment was 0.207 g_{GA}/kg. This shows that the presence of urea promotes the decomposing activity of the polysaccharide pectin in the present case.

If the C/N ratio is too low, inhibition of the bacteria due to an excessive ammonia content results. These conditions lead to disrupted biogas production. Before nitrogen-containing substances are added, the pre-existing ammonium-nitrogen content in the digestate must be investigated in order to avoid possible ammonia inhibition. There is little agreement in the literature regarding optimal ammonium-nitrogen concentrations. Eder and Schulz (2007) note that the inhibition of bacteria by ammonia is dependent both on temperature and on pH value. For example, an ammonium-nitrogen concentration of 3 g/L at 38 °C and a pH of 7 may not be critical; on the other hand, even 1 g/L of NH_4-N in the digestate at the same temperature and a pH of 8 can lead to the onset of ammonia inhibition. However, the biocenosis can adapt to high ammonium-nitrogen contents. Stable anaerobic processes at up to 6 g/L of NH_4-N have been described (Hansen et al. 1998).

Conclusions

Foam formation in the fermentation of sugar beet can be reduced in an effective manner using urea. This substance led to a reduction in the increase in volume of the fermentation mass of 45% in foaming tests with the addition of just 0.25 g/kg of urea. Based on the batch experiments carried out, the use of calcium cyanamide in biogas plants is explicitly discouraged, as biogas production in batch fermentation experiments came to a stop at a calcium cyanamide mass fraction of just 1.25 g/kg.

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Authors

Dr.-Ing. Lucie Moeller is researcher and **Prof. Dr.-Ing. Andreas Zehnsdorf**, is senior researcher and Head of the Bioprocess Technology working group at the Centre for Environmental Biotechnology (UBZ) at the Helmholtz Centre for Environmental Research – UFZ, Permoserstr. 15, 04318 Leipzig, e-mail: lucie.moeller@ufz.de.

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