Microaeration through a biomembrane for biogas desulfurization: lab-scale and pilot-scale experiences

Lucie Pokorna-Krayzelova, Jan Bartacek, Shelmith Nyawira Theuri, Camilo Andres Segura Gonzalez, Jindrich Prochazka, Eveline I. P. Volcke and Pavel Jenicek

Microaeration, a biological method to remove H₂S from biogas by oxidizing it to elemental sulfur, has been shown to be highly efficient, simple and reliable. However, dosing air directly into an anaerobic fermenter results in the dilution of biogas with nitrogen and oxygen and can cause clogging of biogas pipes by elemental sulfur. These disadvantages can be overcome by the use of a biomembrane, i.e. a membrane covered with a biofilm that separates air and biogas. Experiments with bare, wet and biofilm membranes were conducted with a commercially available PVDF LM-P2 membrane to evaluate the chemical and biological oxidation rates of H₂S. Different amounts of air were dosed through the biomembrane to determine the optimum air-to-biogas ratio, to evaluate methane losses and to evaluate biogas contamination with nitrogen and oxygen. The H₂S content decreased from 3000 ppm to less than 100 ppm within two days. The loss of methane was 3.7% of the total methane production and the specific H₂S removal rate was 32 mg m⁻² d⁻¹.

1. Introduction

During the anaerobic treatment of wastewater with high sulfate concentrations, sulfate-reducing bacteria produce a high amount of hydrogen sulfide,⁰¹² which causes major technological problems such as inhibition of anaerobic processes, corrosion of tanks, piping, engines and boilers, and emission of sulfur dioxide from biogas combustion.³ Because of these negative effects, hydrogen sulfide has to be removed from biogas.

The available methods for desulfurization are physicochemical (e.g. absorption, precipitation) or biological (e.g. biochemical oxidation of sulfide) methods.⁴⁵ Biological processes are often simpler and more cost-effective compared to physico-chemical methods.⁶ One of the options for biological H₂S removal from biogas, which has been used on a full-scale,⁷ is microaeration, i.e. the controlled dosing of a small amount of air into an anaerobic reactor.⁷⁻⁹ In microaeration, sulfide is oxidized to elemental sulfur by sulfide-oxidizing bacteria, at low oxygen concentrations. The elemental sulfur produced is insoluble and could be possibly removed from the system. Both the total concentration of sulfide in the effluent and the concentration of hydrogen sulfide in biogas can be significantly decreased with this process.¹⁰

The possible drawbacks of microaeration are the dilution of biogas with nitrogen and little or no control over the exact location where elemental sulfur is deposited. Elemental sulfur usually accumulates on the walls in the reactor headspace¹¹ or in the liquid effluent.¹² However, sulfur could also accumulate in the three-phase separators of anaerobic high-rate bioreactors or in gas pipes, which may cause serious clogging problems. These problems could be overcome by using a biomembrane, where air needed for microaeration is
delivered through the membrane and a sulfide-oxidizing biofilm needed for biological sulfide oxidation grows on the surface of the membrane. Besides better control over the location of sulfur production, dosing air over the semi-permeable membrane also decreases the contamination of biogas by nitrogen. The membrane can be placed either in the liquid phase or in the gas phase of the bioreactor. Previous lab-scale studies with a simple silicone tube used as a membrane revealed the ability to remove hydrogen sulfide from biogas in batch as well as continuous experiments.

In this paper, a commercial hollow fiber membrane was tested first on the lab-scale for hydrogen sulfide removal from biogas. Experiments with a bare membrane, wet membrane and biofilm membrane were conducted in separate batch reactors to measure the gas permeation and to detect the chemical and biological sulfide oxidation. The membrane was then placed into the headspace of a pilot-scale anaerobic digester to prove its ability to continuously remove large quantities of hydrogen sulfide during real operation. Different amounts of air were dosed through the biomembrane to determine the optimum air-to-biogas ratio, to evaluate methane losses through the biomembrane and to evaluate biogas contamination with nitrogen and oxygen.

2. Materials and methods

2.1. Experimental set-up: lab-scale biomembrane unit

A 120 L biomembrane unit (BMU) including a commercial membrane (specifications in Table 1) was used to simulate the headspace of an anaerobic reactor (Fig. 1). The air reservoir has been connected to the air side to decrease the ratio between the biogas and air volume. Gas permeation (the concentration of hydrogen sulfide, oxygen, nitrogen, methane and carbon dioxide) through the membrane was measured in three configurations: bare membrane (experiments with a dry membrane), wet membrane (the membrane was submerged for 30 minutes in tap water before the experiment), and biofilm membrane (the membrane was submerged for 30 minutes in mesophilic sludge for biofilm attachment before the experiment). During the biomembrane experiments, biogas was humidified by sparging through a column with saturated sodium chloride solution (359 g L⁻¹). The sodium chloride solution was acidified (pH 4) to prevent H₂S and CO₂ dissociation hence decreasing their solubility in water.

At the beginning of each experiment, the air side was flushed with fresh air from the atmosphere and the biogas side was flushed with synthetic biogas (64.1% CH₄, 35.5% CO₂, and approximately 0.2% H₂S). Both systems were sealed and recirculated at a flow rate of 13.2 L h⁻¹. Experiments were conducted under atmospheric pressure and at ambient temperature (±20 °C). Hydrogen sulfide, oxygen, nitrogen, methane and carbon dioxide were measured hourly on both sides. The transfer of methane, carbon dioxide, nitrogen and oxygen was evaluated.

Before the start of each experiment, the lab-scale BMU was tested for potential leakages and the experiment started only when the BMU was found to be perfectly tight (data not shown).

2.2. Calculations of membrane parameters in the lab-scale biomembrane unit

CH₄, CO₂, N₂, and O₂ transfer. The oxygen, nitrogen, methane and carbon dioxide transfer rates (r₁) through the membrane were calculated for the membrane. At the start of the experiment, the concentrations of oxygen and nitrogen in the biogas side were close to zero and so were the concentrations of methane and carbon dioxide in the air side. This concentration gradient resulted in the transfer of gases through the membrane until the partial pressures on both sides equalized. As the pores of the PVDF membrane were much bigger than the size of transferred molecules, the transfer was mainly carried out by convective transport and diffusion played only a minor role.

The increase in the molar amount of a gas at one side of the membrane is equal to its decrease at the other side, as well as the amount transferred through the membrane, as expressed through eqn (1):

\[
\frac{dV}{dt} = -\frac{dV}{dt} = r_1 \left( c_a - c_b \right)
\]

where cₐ and cₙ denote the gas concentrations at the biogas side and air side, respectively [mol L⁻¹]. Aₘᵢ is the membrane surface area [m²], r₁ is the transfer rate [L m⁻² h⁻¹], and Vₐ and Vₙ are the volumes of the biogas side and air side, resp. [L]. Note that the recirculation flow was much higher (4 orders of magnitude) than the flux through the membrane. By this, any limitation due to a low flow was eliminated and the concentration gradient was the only driving force for the mass transfer. This was not the case for the pilot-scale CSTR, where the biogas flow was lower and hence limited mass transport through the membrane might occur. Therefore, the transfer rates of gasses cannot be directly extrapolated to the pilot-scale CSTR experiments.

H₂S transfer. The hydrogen sulfide parameters such as the transfer rate (r₁), the chemical oxidation rate (rₗchemox), the biological oxidation rate (rₗbiox), and the half-saturation constant (Kₛ) were calculated for the membrane.

<table>
<thead>
<tr>
<th>Specification of membrane</th>
<th>Units</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Type</td>
<td></td>
<td>LM-P2⁺</td>
</tr>
<tr>
<td>Membrane material</td>
<td></td>
<td>PVDF⁶</td>
</tr>
<tr>
<td>Specific surface area</td>
<td>m²</td>
<td>20</td>
</tr>
<tr>
<td>Pore size</td>
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<td>Maximum working pressure</td>
<td>MPa</td>
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<tr>
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<td>Biogas side to air side ratio</td>
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<tr>
<td>Biogas side volume</td>
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<td>117.9</td>
</tr>
</tbody>
</table>

⁺ Model number. ⁶ Polyvinylidene fluoride.

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The concentration of hydrogen sulfide was high on the biogas side and close to zero on the air side. This caused a decrease in the concentration on the biogas side and an increase on the air side. The concentration decrease on the biogas side during experiments with the bare membrane and wet membrane was caused by the transfer across the membrane as well as by the chemical oxidation. The rate of transfer across the membrane and the chemical oxidation rate were modelled using a first order reaction. Considering the biomembrane, hydrogen sulfide was also used as a substrate for the bacteria growth which was modelled using a Monod-type equation.

The hydrogen sulfide concentrations on the biogas side (eqn (2)) and air (eqn (3)) side were calculated according to the following equations:

\[
V_b \frac{dc_b}{dt} = r_1 A_m (c_a - c_b) - V_b r_{chemox} (c_b) - V_b r_{bioox} \left( \frac{c_b}{c_h + K_s} \right)
\]

\[
V_a \frac{dc_a}{dt} = r_1 A_m (c_a - c_b)
\]

where \(c_b\) is the concentration of hydrogen sulfide on the biogas side \([\text{mg L}^{-1}]\), \(c_a\) is the concentration of hydrogen sulfide on the air side \([\text{mg L}^{-1}]\), \(A_m\) is the membrane surface area \([\text{m}^2]\), \(r_1\) is the hydrogen sulfide transfer rate \([\text{L m}^{-2} \text{h}^{-1}]\), \(r_{chemox}\) is the hydrogen sulfide chemical oxidation rate \([\text{h}^{-1}]\), \(r_{bioox}\) is the H2S biological oxidation rate \([\text{h}^{-1}]\), \(K_s\) is the H2S half-saturation constant \([\text{mg L}^{-1}]\), and \(dt\) is the time step \([\text{h}]\). Biological H2S oxidation was not included in eqn (3), as the biofilm grew only on the side of the membrane facing the biogas side.

Eqn (2) and (3) were solved for the parameters \(r_1\), \(r_{chemox}\), \(r_{bioox}\) and \(K_s\) by minimizing the sum of squared errors between experimental data and the data gained from the calculations, using the inbuilt Excel (Microsoft Office 2013) differential equation “solver”.

2.3. Experimental set-up: pilot-scale CSTR with a biomembrane

The applicability of the biomembrane for hydrogen sulfide removal was tested in a 250 L pilot-scale continuous stirred tank reactor (CSTR) shown in Fig. 2. The biomembrane (membrane 1 with a surface area of 10 m²) was placed into the gas space of 50 L. The reactor was inoculated with 200 L of mesophilic sludge from a municipal wastewater treatment plant and operated at 40 °C. Cheese whey \((2.75 \text{ g COD g}^{-1} \text{cheese whey, the pH value fluctuated between 2.9 and 5.2})\) was used as the feed because of its high sulfur content \((2.06\% \text{ dry weight of S})\) and good biodegradability. The organic loading rate (OLR) initially amounted to \(0.05 \text{ g COD g}^{-1} \text{VSS d}^{-1}\) and was gradually increased to \(0.30 \text{ g COD g}^{-1} \text{VSS d}^{-1}\) (Table 2). The concentration of the mesophilic sludge was kept constant the whole time. The CSTR was operated in 30 minute cycles \((30 \text{ s feeding, } 1260 \text{ s mixing, } 500 \text{ s sedimentation, and } 10 \text{ s sludge recirculation})\). The hydraulic retention time (HRT) of the CSTR was 21 days.

Microaeration was turned on after the start-up period where an OLR of \(0.30 \text{ g COD g}^{-1} \text{VSS d}^{-1}\) was reached. The amount of air dosed was expressed relative to the
stoichiometric H₂S:O₂ molar ratio of 2:1 required for the oxidation of sulfide to elemental sulfur. An excess amount of oxygen was dosed initially, and its dosing was then gradually decreased to determine the suitable amount for biogas desulfurization. In the diagrams, the dose of oxygen is shown with symbols I–VI. (Table 2): I. – 1000× stoichiometry (400 mL air per min); II. – 100× stoichiometry (40 mL air per min); III. – 10× stoichiometry (4 mL air per min); IV. – 2× stoichiometry (0.8 mL air per min); V. – 3× stoichiometry (1.2 mL air per min); and VI. – 5× stoichiometry (2.0 mL air per min).

Hydrogen sulfide, nitrogen, oxygen, methane, and carbon dioxide were measured in the biogas and air effluent, while the total and dissolved COD, organic acids, sulfide, pH and ammonia were measured in the CSTR influent and effluent.

2.4. Analytical methods

The hydrogen sulfide concentration in the gas for the lab-scale BMU was monitored using an online electrochemical gas sensor (Membrapor H₂S sensor type H₂S/S-10000-S). The hydrogen sulfide concentration in the gas for the pilot-scale CSTR with a biomembrane unit was measured using an RAE H₂S gas detection tube. Gases (CH₄, CO₂, N₂, and O₂) were measured using a GC Shimadzu 2014 equipped with a thermal conductivity detector (CH₄, CO₂, air) and using a GC 8000TOP (Fisons Instruments, USA) equipped with a heat conductivity detector HWD 800 (O₂, N₂, CH₄). Analyses of COD, pH, VFA, solids, ammonia and sulfide were performed according to the Standard Methods (American Public Health Association, 1997). The sulfur composition of the sludge was assessed using an Elemental Vario EL III (Elementar Analysensysteme GmbH, Germany) and by X-ray fluorescence analysis using an ARL 9400 XP sequential WD-XRF spectrometer (THERMO ARL, Switzerland).

3. Results

3.1. Lab-scale biomembrane unit

The concentration of hydrogen sulfide in the biogas side and air side is shown in Fig. 3. The experiment with the dry membrane showed not only the transfer of H₂S from the biogas...
side to air side, but also the potential chemical sulfide oxidation (Table 3). The chemical sulfide oxidation accounted for 0.002 L h$^{-1}$ for the dry membrane, while for the experiment with the wet membrane the chemical sulfide oxidation was 0.013 L h$^{-1}$. However, it could also be caused by the solubility of hydrogen sulfide in water. The last configuration with the biomembrane showed also biological sulfide oxidation in addition to the other mentioned parameters with a ratio of approximately 20% for chemical and 80% for biological sulfide oxidation. The complete results of kinetic parameters are shown in Table 3.

The transfer of methane, carbon dioxide, oxygen and nitrogen (Fig. 4) between the biogas side and air side depended on their permeability through the membrane as well as on their solubility in water. While for the experiments with the bare membrane the transfer of all gases was comparable (approx. 0.103 m$^3$ m$^{-2}$ h$^{-1}$) as shown in Table 3, the wet membrane and biomembrane experiments showed differences which can be caused by the mentioned solubility in water. For the wet membrane and biomembrane experiments, nitrogen (0.115 ± 0.005 m$^3$ m$^{-2}$ h$^{-1}$) and carbon dioxide (0.130 ± 0.027 m$^3$ m$^{-2}$ h$^{-1}$) had the highest transfer rates, respectively.

3.2. Pilot-scale CSTR with a biomembrane

3.2.1. COD removal efficiency. The CSTR was operated for over 250 days. Microaeration was turned on at day 83; following an 82 day start-up period. The COD removal efficiency (Fig. 5a) was 93% on average (start-up excluded) and was not affected by microaeration. The pH value of the feedstock fluctuated between 2.9 and 5.2. Despite the high acidity of the feedstock, the effluent pH was 7.3 ± 0.4 on average.

3.2.2. Efficiency of hydrogen sulfide removal. The hydrogen sulfide concentration (Fig. 5b) increased up to 3000 ppm during the start-up period (periods A–D). Upon the start of microaeration (period I), the concentration of hydrogen sulfide decreased within one day from 3000 ppm to less than 100 ppm. The rate of H$_2$S oxidation was approximately 320 mg d$^{-1}$. After 40 days, microaeration was turned off and the H$_2$S concentration in biogas increased to 4000 ppm. This was repeated in periods II and III (100× and 10× stoichiometry, respectively). The results were identical to period I: the H$_2$S concentration decreased within one day from 4000 ppm to less than 100 ppm (the H$_2$S oxidation rate in period II was approx. 430 mg d$^{-1}$). In periods IV and V (2× and 3× stoichiometry, resp.) the amount of oxygen was not sufficient to completely remove H$_2$S from biogas resulting in approximately 500 ppm of H$_2$S left in biogas for both periods. During period VI (5× stoichiometry) enough oxygen to remove H$_2$S from biogas was supplied (concentration below 100 ppm) with minimum biogas contamination with O$_2$ and N$_2$.

3.2.3. Biogas and air composition. The composition of biogas and air leaving the pilot-scale CSTR is shown in Fig. 6 and Table 4. In periods C and D where no oxygen was dosed, the amount of methane and carbon dioxide was 57 and 36%, respectively. Even though there was no microaeration at this time, some oxygen and nitrogen were observed in biogas during these two periods (approx. 2 and 5%, resp.). This is probably caused by the contamination of the samples by air. The composition of gasses in the biogas side for the periods III–VI was almost the same (around 52:41:1:6 for CH$_4$:CO$_2$:O$_2$:N$_2$). Periods I and II (with strong air overdose) had higher amounts of oxygen and nitrogen. It was only in period I that the methane-to-CO$_2$ ratio was higher.

The decreasing amount of air blown into the reactor resulted in decreasing amounts of oxygen and nitrogen and increasing amounts of methane and carbon dioxide in the effluent air. Methane losses also decreased with the decreasing amount of air (Table 4). For period I the methane losses were 9.5 liters per day while during period VI, the losses decreased to 0.4 L d$^{-1}$. Specific H$_2$S removal was almost equal for all periods (approx. 34 mg m$^{-2}$ d$^{-1}$).

4. Discussion

4.1. Pilot-scale CSTR with a biomembrane

The pilot-scale CSTR with a biomembrane was capable of removing up to 99% of H$_2$S from biogas without affecting the production of methane. When a sufficient amount of oxygen...
A few lab-scale studies comparable to this research have been published so far. Valdes and Camiloti\textsuperscript{16} placed a silicone membrane into a continuous anaerobic fixed bed reactor through which pure oxygen was dosed oxidizing sulfide to elemental sulfur. The sulfide removal efficiency reached 96%; however, elemental sulfur was not the only end-product. Sulfate and thiosulfate accounted for 27.9% and 25.5%, respectively. In the set-up of Camiloti and Oliveira,\textsuperscript{15} wastewater recirculated between a CSTR and an external silicone membrane module. Oxygen was dosed only through the membrane wall and it (chemically and biochemically) oxidized sulfide to elemental sulfur (and also to sulfate) proving the applicability of the membrane for desulfurization. Different pH and applied velocity values were tested and the total dissolved sulfide removal efficiency was 89.7–97.9% (the amounts of elemental sulfur and sulfate varied a lot in the different set-ups). In these studies, the membrane, through which air was dosed, was in direct contact with the liquid reactor content (i.e. sludge), so that sulfide was removed in the liquid phase. However, this can cause problems with the decrease in the membrane transfer capacity contributing extra resistance to the oxygen transfer because of biomass and elemental sulfur settling.\textsuperscript{16} Moreover, as the hydrogen sulfide concentration in biogas is controlled indirectly through sulfide concentration in liquid, almost all sulfide has to be removed in order to achieve sufficiently low hydrogen sulfide concentration in biogas. In our study, the biomembrane served as a barrier between air and biogas with a thin biofilm layer on the membrane surface at the biogas side for H$_2$S removal. Therefore, the hydrogen sulfide content in biogas was controlled directly.

### 4.2. Lab-scale biomembrane unit

The transfer of gasses through the membrane depends on the biomembrane properties. While for the bare membrane the transport of all gasses was comparably fast, for the wet membrane and biomembrane, the transport rates differed between various gasses. Note that the biogas flow in the pilot-scale CSTR was lower and hence limited mass transport through the membrane might occur. Therefore, the transfer rates of gasses from the lab-scale experiment cannot be directly extrapolated to the pilot-scale CSTR experiment.

The water layer on the membrane caused the decrease of the hydrogen sulfide and carbon dioxide transfer rates.
Fig. 4 Composition of gasses in the air side and biogas side: A – CH$_4$ in the air side; B – CH$_4$ in the biogas side; C – CO$_2$ in the air side; D – CO$_2$ in the biogas side; E – O$_2$ in the air side; F – O$_2$ in the biogas side; G – N$_2$ in the air side; H – N$_2$ in the biogas side.
Hydrogen sulfide and carbon dioxide are highly soluble in water (3.85 and 1.69 mg g\(^{-1}\) H\(_2\)O, respectively). This contrasts with the solubilities of methane, oxygen and nitrogen (0.023, 0.043 and 0.019 mg g\(^{-1}\) H\(_2\)O, respectively).\(^{21}\) The decrease of methane, oxygen and nitrogen transport rates was only negligible.

The transport rate of gasses in the biomembrane (membrane covered with the biofilm) decreased for all gases except for carbon dioxide. The biofilm acted as a barrier and limited the transfer. The very low transport rate of methane and the two times higher transport rate of carbon dioxide could be explained by methane aerobic oxidation to carbon dioxide with the help of methanotrophs.\(^{22}\) However, there was not enough biofilm for bacterial analyses. In the pilot scale experiment, this was not observed.

During the biomembrane experiment (membrane covered with the biofilm), 117.3 mg H\(_2\)S h\(^{-1}\) was removed from the biogas with the combination of chemical and biological sulfide oxidation. However, approximately 20% of the membrane was covered with the biofilm, resulting in a lower H\(_2\)S removal rate.

Chemical and biological sulfide oxidation accounted for 0.54 g d\(^{-1}\) and 2.27 g d\(^{-1}\), respectively. By using the biomembrane the biological sulfide oxidation was approximately 4.2 times faster compared to chemical sulfide oxidation, while Pokorna-Krayzelova and Vejmelková\(^{23}\) observed the biological sulfide oxidation being 2.5 times faster in a batch reactor under microaerobic conditions. According to Buisman and Uspeert,\(^{24}\) at a lower sulfide concentration (around 10 mg L\(^{-1}\)) the biological sulfide activity was 75 times faster than chemical sulfide oxidation, and according to Janssen and Sleyster,\(^{10}\) the chemical sulfide oxidation became important when the biological activity of sulfide-oxidizing bacteria was limited. According to our results, this was not the case. Chemical sulfide oxidation occurred in all three experiments (bare, wet, and biofilm membranes) independent of the biological activity. The chemical sulfide oxidation was higher for the wet membrane and biofilm membrane (0.013 and 0.016 L h\(^{-1}\), resp.) compared to the dry membrane (0.002 L h\(^{-1}\)). This could be explained by the creation of polysulfide formed from a reaction between elemental sulfur (from the
microaerobic oxidation of hydrogen sulfide) and an aqueous solution of hydrogen sulfide (dissolved in the wet/biofilm layer). In natural waters, sulfide can be oxidized to sulfur which then combines with the remaining sulfide to form polysulfide. However, it was not possible to collect samples on the membrane surface for polysulfide determination.

5. Conclusions

The efficiency of the pilot-scale CSTR with a biomembrane for H₂S removal from biogas was tested:

- Both chemical and biological sulfide oxidation were observed in the lab-scale biomembrane. Biological sulfide oxidation was four times faster.

![Fig. 6](image)

**Fig. 6** The gas composition during the experimental period, A – biogas composition, B – air composition. The OLR increased during periods A–D (Table 2) and the air/H₂S ratio varied during periods I–VI (Table 2).

<table>
<thead>
<tr>
<th>Biogas composition [%]</th>
<th>Air composition [%]</th>
<th>Specific H₂S removal [g m⁻² d⁻¹]</th>
<th>CH₄ losses [L d⁻¹]</th>
<th>CH₄ production [L d⁻¹]</th>
</tr>
</thead>
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<tr>
<td>CH₄</td>
<td>CO₂</td>
<td>O₂</td>
<td>N₂</td>
<td>CH₄</td>
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<tr>
<td>C–D</td>
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<tr>
<td>II.</td>
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<tr>
<td>VI.</td>
<td>51.6</td>
<td>41.6</td>
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</table>
The amount of air needed for complete H$_2$S removal from biogas with no oxygen and nitrogen leftovers in biogas was 5 times higher than the stoichiometric amount based on the sulfur content in the feed.

- The methane loss through the biomembrane was 3.7%.
- The maximum specific H$_2$S removal was 34 mg m$^{-2}$ d$^{-1}$.

It was limited by the rate of hydrogen sulfide production inside the reactor.

**Conflicts of interest**

There are no conflicts to declare.

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