

# Membrane separation to improve degradation of road side grass by rumen enhanced solid incubation

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**Abstract** Membrane separation proved to be an excellent means to maintain high residence time of microorganisms in an anaerobic hydrolysis reactor, and relatively low concentration of hydrolysis products. The microbial biocommunity typical for the rumen environment could be maintained, and the reactor efficiency of the reactor improved. Less than 4 days were required to reach almost complete hydrolysis of the grass fed into the reactor. To avoid blocking of the membrane unit, a backwash system is necessary. The membranes needed to be backwashed every 20 min with 4 bar gas-pressure for 10 s. After this treatment the initial permeability was regained. The plant was operated with a flux of  $12 \text{ ml h}^{-1} \text{ cm}^{-2}$  on average. The transmembrane pressure was in the range of 0.8–0.9 bar. 90% of the dissolved fatty acids permeated through the membrane.

**Keywords** Anaerobic digestion; hydrolysis; lignocellulosic waste; membrane; rumen microbiology

## Introduction

Anaerobic digestion is gaining importance as a process to treat and recover energy from organic matter rich in lignocellulosic content. Anaerobic digestion is a multi-step process in which complex organic matter present in the organic material is hydrolysed into soluble compounds under fully or partially fermentative conditions. Acidogens and acetogenic bacteria convert these intermediates into volatile fatty acids, hydrogen, carbon dioxide and other low molecular weight compounds. In wastes containing high lignin and cellulose contents like grass, the lignocellulosic portion constitutes the major fraction of substrates convertible into methane gas. The biotechnological methods currently available have a major limitation as they demand long reaction times for hydrolysis and subsequently large reactor volume. In nature, in contrast, hydrolytic processes are performed in a much shorter period of time. Ruminant organisms such as cows, sheep or goats are especially effective in metabolizing structured organic material. Hypothetically, it can be assumed that a significant gain in process efficiency can be achieved by translating the hydrolytic processes performed in the rumen into a technological concept. To achieve a retention time as short as in the rumen it is indispensable to minimize washout of microorganisms from the hydrolysis reactor. In this study a membrane system is used to exclusively extract the liquid fraction of the reactor containing the dissolved organic acids. Three different membrane types were investigated in cross-flow mode, microfiltration membranes of Teflon and ceramic material, and ion exchange membranes typically used for dialysis. The dialysis membrane was applied to achieve selective transport of volatile fatty acid (VFA) from the fermentation liquid. VFA permeated well through the membrane but only at a very low pH (about 2). At pH values above 6.0, required to achieve reasonable VFA production in the hydrolysis reactor, the flux was unsatisfactory, however.

To be able to keep the pH in the range optimal for hydrolysing bacteria, to uncouple hydraulic and solid retention time, and to keep VFA from accumulating in the reactor we switched to microfiltration membranes for solid-liquid separation.

Several attempts have been made by other researchers (Engler and Wiesner, 2000; Lee

*et al.*, 2001; Kang *et al.*, 2002; Schwille *et al.*, 2002) to investigate the performance of membranes for anaerobic wastewater management applications. Results from these studies showed that solid materials accumulated very quickly at the surface of the membrane and within the pores. Lee *et al.* (2001) have shown that membrane performance was enhanced when brewery-wastewater was pre-filtered through a 63  $\mu\text{m}$  mesh before application to the membrane system. Frequent backwashing every 15 min was still necessary, nevertheless. A combination of membrane rotation and backwashing has been proposed by several authors to keep the flux at a reasonable range. Schwille *et al.* (2002) found that rotating cylindrical filtration displays significantly reduced plugging of filter pores and build-up of a cake layer. The dominant mechanism to reduce pore plugging was the rotational shear.

Based on these observations we decided to apply rotating microfiltration membranes for the removal of dissolved VFA from a biological hydrolysis reactor fed with grass as digestible solid material. The reactor was inoculated with rumen microbiology adapted to grass as normal feed. In the following, the results of preliminary experiments obtained with this reactor setup are presented and discussed.

## Methodology

### Feed material

Experiments were conducted with road-side grass as feed material. The grass was cut into small pieces (1–2 cm in length) by means of a grass cutter, and water was added to generate a slurry. Rumen content from cattle was collected from a slaughterhouse and used as inoculum.

### Membrane

The selected Dia-FILTROPLAST polyethylene membrane (USF Schumacher, Germany) was 200 mm long with 70 mm outer-diameter (Figure 1), and exhibited a pore size of around 1  $\mu\text{m}$ . The fine layer was at the outer surface of the membrane. During the experiments the membrane was not rotated.

### Experimental set-up

A vacuum pump (AEG, Germany) was used to suck the liquid from the reactor through the membrane. The utilized pump delivers a constant suction of 0.8–0.9 bar. The reactor was

**Table 1** Characteristics of the grass slurry used as feed

COD	g/kg	1,132.0
TS	g/kg	400.0
TOC	%	46.14
TR	g/kg	0.24
OTS	%	91.20
Hemicelluloses	%	14.00
Cellulose	%	28.30
Lignin	%	5.40
Ash	%	0.46



**Figure 1** Polyethylene membrane

placed inside a water bath to control the slurry temperature. Sodium hydroxide (Merck, Germany) was used to control the pH of the slurry. Thus, flux data could be obtained at different slurry temperatures and pH values. All joints were airtight.

## Results and discussion

### Comparison of flux values for different liquids

The membrane transfer capacity was investigated by applying water, VFAs acids dissolved in water, and a model slurry. The results are summarized in Figure 2.

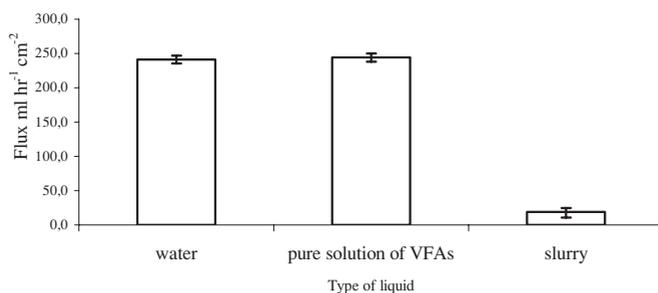
It is certainly not surprising that there is no significant difference ( $P = 0.05\%$ ) between flux values for water and the VFAs pure solution. However, flux of slurry is significantly ( $P = 0.05\%$ ) less than that of the VFAs pure solution or water. This may be evidence that the solids of the slurry accumulate very quickly at the surface and in the pores of the membrane.

### Transfer of VFAs and solids

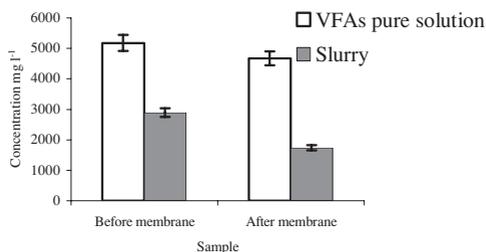
Concentrations of the transferred VFAs through the polyethylene membrane are shown in Figure 3.

Evidently, more than 90% of the VFAs were transferred through the membrane within the time frame provided. The high percentage of the transferred VFAs through the membrane encouraged further investigations on the use of the selected membrane to transfer the VFA solution from the hydrolysis reactor into a second-stage reactor operated for methane gas production.

The effect of different temperature with respect to the flux was higher at 32°C or 37°C slurry temperature than at 26°C or 21°C during the first 50 min of the laboratory trial. The higher flux value for the 37°C slurry may be due to the decrease in its viscosity with temperature. The flux values for slurries of 32°C and 37°C did not differ significantly during the first 40 min of operation. Nonetheless, the flux values of 37°C were unexpectedly lower than those at 32°C, particularly after 2 hr of operation. The reason for this unexpected behaviour is not clear.



**Figure 2** Average fluxes of different types of liquids through the 1 µm polyethylene membrane (error bar = 1 standard deviation (SD),  $N = 3$ ), suction = 0.9 bar



**Figure 3** Transfer of VFAs through the 1 µm polyethylene membrane (error bar = 1 SD,  $N = 3$ )

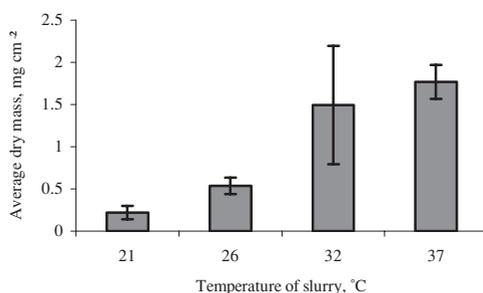
Investigation of the cake which had been built up after about four hours of operation suggests a strong temperature effect (Figure 4). There is, however, no significant difference ( $P = 0.05$ ) between the mass of organic material accumulated on membrane surface for slurries of 32°C and 37°C. It appears that microbial growth at the membrane surface and subsequent biofilm formation were the main reason for the observed phenomenon.

The desirable pH range for the solution in the hydrolysis reactor is between 6 and 6.5 (Ince *et al.*, 2000; Lee *et al.*, 2001). A row of experiments showed, that there are no significant differences among the flux values for the studied pH range.

*Effect of backwashing and cleaning the membrane surface on flux values.* The flux values of the hydrolyzed slurry through the utilized membrane continued to decline with time. This is probably due to the build up of biofilm on the membrane surface and the clogging of its pores with solids.

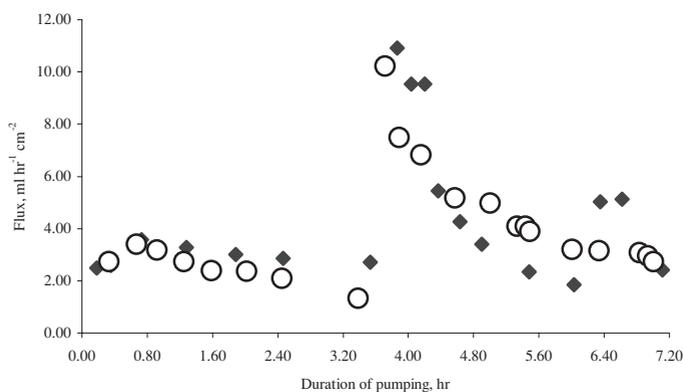
Therefore, the membrane was backwashed after about 4 hr of operation with gas (air) at 4 bar in one set of experiments. In another set of experiments the surface of the membrane was wiped with a sponge after 4 hr of operation to examine the effect of mechanical cleaning of the membrane surface on flux values without gas backwashing. Results of the two sets of experiments are summarized in Figure 5.

Figure 5 shows that the flux values increased when the membrane was gas backwashed or when the accumulated biofilm was wiped from the membrane surface with a sponge. The flux value increased from 1.8 ml hr<sup>-1</sup> cm<sup>-2</sup> to about 10 ml hr<sup>-1</sup> cm<sup>-2</sup> on average when the



**Figure 4** Accumulated biomass on one cm<sup>-2</sup> of the polyethylene membrane for slurries of different temperatures after about 4 hr of operation. Average slurry pH = 6.4 (error bar = 95% significant interval,  $N = 3$ )

◆ cleaning the membrane surface with a sponge ○ air back washing



**Figure 5** Effect of backwashing the membrane with 4 bar gas and wiping its surface with a sponge on flux values. Suction = 0.9 bar, average pH = 5.2, average slurry temperature = 34°C. Mass of cake on membrane surface after 3.5 hr (membrane kept in solution overnight) was about 1.7 ± 0.2 mg cm<sup>-2</sup> and 1.97 ± 0.3 mg cm<sup>-2</sup> after 7.1 hr.

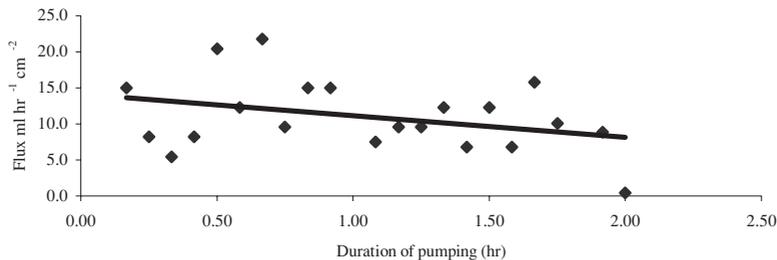
membrane was backwashed with gas, and from  $3 \text{ ml hr}^{-1} \text{ cm}^{-2}$  to  $10 \text{ ml hr}^{-1} \text{ cm}^{-2}$  on average when the biofilm was removed with a sponge. The differences are obviously marginal. Thus, it must be assumed that the decrease in flux was mainly caused by cake or biofilm build up, but not that much by clogging of pores. Subsequently, we assume that abrasive forces induced by rotation of the membrane within the slurry may help keeping cake and biofilm build up under control.

The membrane was backwashed every 15 min for 25 s in another set of experiments. Figure 6 shows the effect of this gas backwashing mode on flux values.

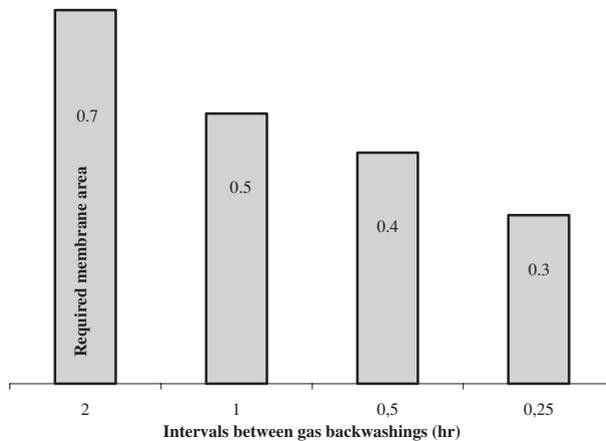
The data indicate that most flux values were above  $5 \text{ ml hr}^{-1} \text{ cm}^{-2}$  and the average flux value was about  $12 \text{ ml hr}^{-1} \text{ cm}^{-2}$ . The latter is significantly higher than the average flux value  $5 \text{ ml hr}^{-1} \text{ cm}^{-2}$  when the membrane was not backwashed while operating.

*Calculation of the required surface area of membrane to establish a certain value of flux.* The obtained experimental data were used to develop a simple excel computer model to predict the required area of membrane for transferring a certain value of flux. Figure 7 shows required surface areas of membrane for different backwashing modes.

The required surface area of membrane decreases as the operational duration between the successive gas backwashes decreases. Therefore, it should be possible to optimize operational and backwashing modes for efficient liquid transfer and minimum energy consumption.



**Figure 6** Effect of backwashing the membrane with 4 bar gas every 15 min for 25 s on flux values. Suction = 0.9 bar, average pH = 6.4, average slurry temperature = 32°C. Mass of cake on surface after 2 hr of operation was about  $0.1 \text{ mg cm}^{-2}$



**Figure 7** Calculated required surface area of the  $1 \mu\text{m}$  polyethylene membrane (70 mm outer diameter) to transfer  $100 \text{ l day}^{-1}$  of hydrolyzed fluid from the slurry reactor. Suction = 0.9 bar, average slurry pH = 6.4, average slurry temperature = 35°C

## Conclusions

The polyethylene membrane applied throughout the experiments has the capacity to efficiently transfer the liquid phase from the hydrolysis reactor into the methane production reactor.

The average flux through the membrane was about  $5 \text{ ml hr}^{-1} \text{ cm}^{-2}$  for the grass slurry without backwashing with gas. The average flux, however, was about  $12 \text{ ml hr}^{-1} \text{ cm}^{-2}$  when the membrane was periodically backwashed with gas.

The flux values increased after the cake or biofilm was wiped off the membrane by means of a sponge. This indicates that the naturally occurring solids in the slurry may continuously scrub off the accumulated cake or biofilm on the membrane surface if the membrane is rotated in the reactor.

Data from the simple model developed for this study showed that the required surface area of membrane to remove the liquid fraction from the hydrolysis reactor decreases as the operational duration between the successive gas backwashes decreases.

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