

Biogas upgrading using a water-swollen composite polyamide membrane

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Abstract

The broad energy consumption together with the foreseen unavailability of low-cost fuels like natural gas in the near future has increased the interest for alternative fuels of high efficiency. The attention has recently shifted to the improvement of biogas quality and the generation of high purity biomethane. This work tested the suitability of a coil filter initially developed for water cleaning to be used in biogas upgrading and presents results of the performed experiments at various conditions. The filter was composed of a membrane containing at the top a hydrophilic layer made of polyamide. The layer when covered by a thin water film is transformed in a highly selective separator able to separate the water-soluble components of biogas such as CO₂ and H₂S from the non-soluble methane and produce a biomethane stream of high purity. A gas stream containing up to 96 vol. % of CH₄ was obtained under specific conditions.

Keywords: biogas, upgrading, gas mixture, membrane separation, biomethane.

Introduction

The extended mining and overuse of fossil fuels like natural gas incurred a broad resource reduction and concurrently induced negative effects on the environment. In addition to the resource abatement, the current initiation of energy crisis necessitates an adaptation of measures in the energy market that will provide self-sufficiency and energy security to the countries. In this context, the interest to partially substitute the natural gas with alternative and similarly efficient fuels is growing with biogas and biomethane being at the forefront.

In Europe, manufacture of biomethane commercially began a little earlier than ten years ago. In 2018, the use of biomethane was highly broadened with the approval of the European directive concerning the renewable energy sources (RED II). Since then, the production of biomethane has been rising and this trend is most likely to continue given the efforts for energy independency in Europe and elsewhere in the near future. According to current estimates biomethane manufacture could secure 34 billion m³ of renewable biomethane until 2030 representing about a 10% of the total gas demand in European union at the same year¹. The most important advantage of biomethane is related to the price of natural gas, which currently averages around 80 €/MWh whereas the cost of biomethane approximately at 55 €/MWh¹. Biomethane is a biofuel which produces significantly lower emissions than fossil fuels. It is indicative that biomethane produced from wastes provides up to 90% emission savings expressed in CO_{2eq} per m³ of fuel compared to the emissions coming from the use of fossil fuels².

According to the European biogas association, the biomethane producers in EU were estimated at about 729 in the year 2020³. However, the current uncertainty surrounding the availability of fossil fuels and the new legislation supporting novel energy sources are anticipated to further promote the incorporation of biomethane into the energy balance. Align with the European statistics, in Czech Republic biomethane is foreseen to substitute a 10 % of the current consumption of natural gas by 2030, which represents a total replacement of the natural gas consumed for transportation in the country¹.

A number of technologies are available for increasing the amount of methane and removing any undesired impurities from a biogas stream. Membrane separation is considered to be an inexpensive

and relatively rapid method for biogas cleaning characterized by high efficiency and low methane losses⁴. The gas resulting from such a separation contains more than 98 % of methane even without the use of additional filtration as it is frequently required in more complicated purification setups⁵.

In this article, the efficiency of an experimental setup with a single-separation step was examined using both a synthetic gas and real biogas. Separation of methane from carbon dioxide and the rest gases contained in biogas was performed on a spiral-wound membrane module. The main advantage of the method was a high separation efficiency even with the direct employment of raw biogas with its containing moisture and impurities such as hydrogen sulphide in the separation step. In this case, therefore,

it was not necessary to pretreat the biogas for the removal of moisture prior separation nor to include in the process any additional secondary desulphurisation system.

Methodology and material

Synthetic gas and biogas

A binary synthetic mixture containing 50 vol% methane and 50 vol% carbon dioxide was initially employed for the gas separation experiments. Raw and wet biogas that obtained from the 1st fermenter of an agricultural biogas plant Pustějov I (Zemspol Studénka a.s.) was used for a set of additional experiments on methane purification in order to examine the effect of the secondary components and impurities of biogas on the separation efficiency of membrane. The biogas was compressed into a galvanized tank VHG100-11 with 100 L volume and maximum storage pressure of 10 bar using an oil piston compressor (Güde 231/10/24).

Experimental setup for biogas upgrade

The laboratory setup was made of stainless steel (AISI 304, AISI 316 L, AISI 316 Ti) tubes with outer and inner diameter of 6 and 3 mm, respectively and Swagelok-type valves and fittings. A schematic diagram of the apparatus is shown in Figure 1. For many purification methods, the water contained in biogas is required to be removed before separation to avoid potential complications⁶. The advantage of the current setup is that allows the gas separation without the removal of water and gas impurities like hydrogen sulfide.

The inlet gas passing through a system of controlling valves and pipes was introduced in the filter tube from the bottom where a tank containing 1.2 L of water heated by an electric resistance heater at 90 °C was located. The pressure of the gas which was also the pressure applied to the module was adjusted between 0.19 – 3.25 bar by a regulator. The saturated in water vapour gas was transferred upwards to the filter module located immediately above the water container. A vacuum pump WELCH ILMVAC Rotary Vane Pump P 6 Z - 101 Chemvac (97 L/min; 2×10^{-3} mbar) assisted the separation by creating vacuum at the centre of the membrane. The vacuum pump was chemically resistant and contained four stages (two membrane modules, two piston oil modules). A spiral-wound membrane filter DUPONT DOW FILMTEC™ XLE-2521 industrially used for desalination of brackish waters was utilised for biogas upgrade. Total area of the membrane was 1 m². The filter consisted of two bags each made of two membranes with the reverse sides glued together at the edges. Both bags were rolled into a cylinder (approximate dimensions 440 x 50 mm) around a plastic tube with an internal diameter of 12 mm containing small holes to allow for the passage of the gases penetrating the membrane. The stream formed at this side of membrane is referred to as the permeate. The gas that passed the module on the axial direction without penetrating the membrane is the biomethane stream (referred to as the retentate) which in an ideal case would be almost exclusively composed of CH₄. The flows of both permeate and retentate were adjusted using needle valves and in all cases were set around similar values.

According to the operating principle of the membrane the saturated with water vapour inlet gas approached the one end of the module and passed the layers of the coiled membrane. Due to the low

temperature of the filter the vapours were condensed on the hydrophilic polyamide layer of membrane and eventually its surface was covered by water. The bottom part of the module was exposed to an operating temperature of 75 °C due to the existence of the heated water tank, however the temperature at the upper part was constantly lower between 25 and 40 °C securing vapour condensation. Water was recycled between the membrane and the tank following a continuous vaporization and condensation process, whereas the water losses through the filter were minimal. In principle, the separation was caused by the different solubility of gases in water ^{7,8}. CO₂, H₂S, NH₃ and other soluble components were expected to be dissolved in the condensed water, while methane only marginally. The pressure gradient caused by the overpressure of the biogas at the inlet and the vacuum from the pump connected at the outlet of the axially located perforated pipe (permeate side) were the driving forces assisting the gas separation. The CO₂, H₂S, NH₃ and other soluble components including H₂ were expected to enter the gas phase and be drained by the operation of the pump.

According to the experimental procedure inlet pressure was set to a desirable value and the system was let for sufficient time to attain a steady state. The composition of permeate and retentate were measured after the partial pressures of the two main gases were stabilized, i.e. the difference between two consequent measurements was less than 2%. The time required to obtain a steady state in each test depended on the applied pressure and flow conditions. Samples of biogas, retentate and permeate were collected in 0.5 L bags made of fluorinated plastic. Before sampling each bag was evacuated with a pump KNF 6 L/min. The gas composition was determined immediately after filling the bag using a portable biogas analyser GEOTECH Biogas 5000. The accuracy of the measurements was further validated using a gas chromatograph (BACS BRK MAG GC-TCD). The flow and total volume of the retentate and permeate were determined by using two Ritter TG 05/PVC/PVC drum-type gas meters (1 – 60 L/h).

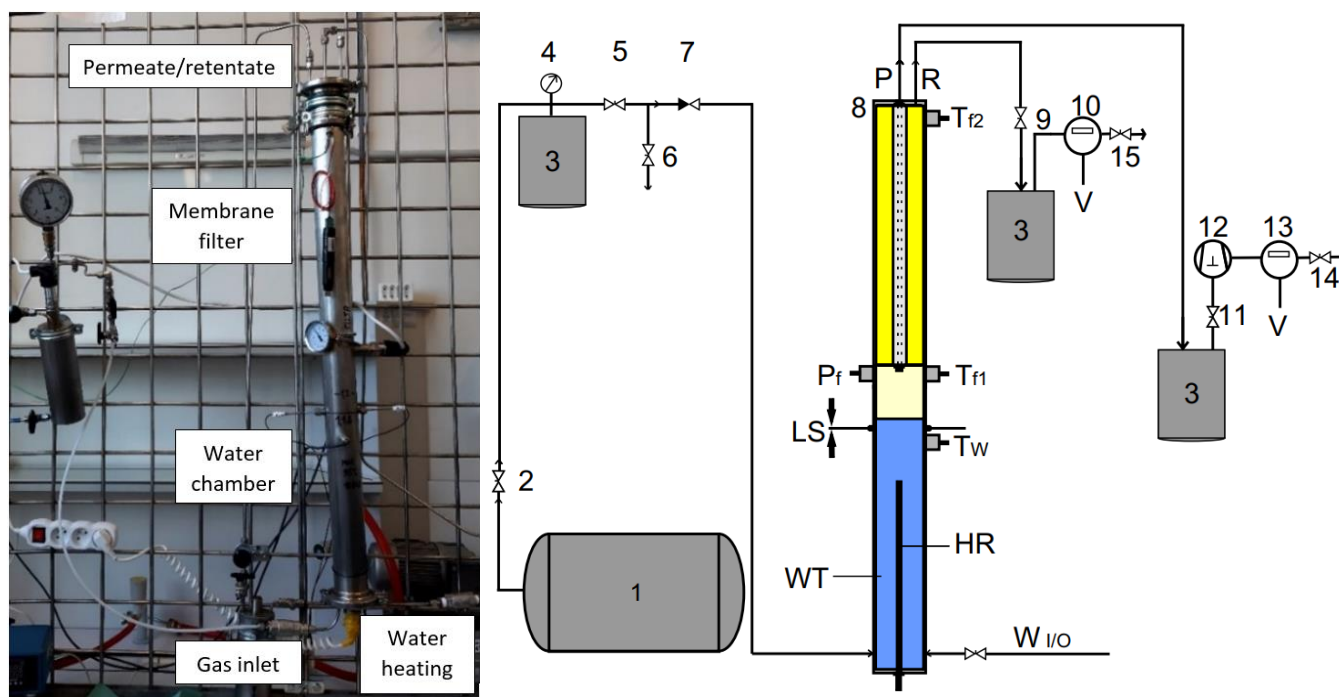


Figure 1: Single-filter setup (a) image of the system, (b) detailed schematic diagram: (1) Gas holder; (2) Pressure control valve; (3) Condensate collector; (4) Manometer; (5) Security valve (inlet blocking); (6) Sampling valve; (7) Backflow prevention valve; (8) Filter vessel; (9) Retentate flow control valve; (10) Retentate flow meter; (11) Permeate flow control valve; (12) Vacuum pump; (13) Permeate flow meter; (14) Permeate sampling valve; (15) Retentate sampling valve; (P) Permeate; (R) Retentate; (LS) Level sensor; (W_{I/O}) Water input/output; (T_w) Water temperature; (T_{f1}) Temperature at low-end of filter; (T_{f2}) Temperature at upper-end of filter; (P_f) Pressure at low-end of the filter; (HR) Heating resistance; (V) Vent; (WT) Water tank

Display and acquisition of data were automatically performed by a custom-developed software based on LOXONE. The software assisted also maintaining the set gas inlet pressure by controlling a switch valve and the set water-tank temperature by switching between on and off an electric resistance heater.

Results and discussion

The results on membrane separation including the flow rate and the composition of inlet and retentate streams are shown in Table 1. The partial pressure of methane in the retentate and the recovery efficiency were used for the characterization of the separation performance of the filter. The recovery efficiency was expressed as⁹:

$$n = \frac{Q_{ret} \cdot p_{CH_4}^{ret}}{Q_{feed} \cdot p_{CH_4}^{feed}}$$

where n is the recovery efficiency, Q_{ret} is the flow rate of the retentate, Q_{feed} is the flow rate of the feed gas, and $p_{CH_4}^{ret}$ and $p_{CH_4}^{feed}$ are the partial pressures of methane in the retentate and feed gas, respectively.

Tests were conducted at relatively low pressures ranging between 0.19 – 3.25 bar. For the synthetic gas mixture, a high amount of methane in retentate (95.6 – 96.6 vol.%) was observed at the low pressures (0.19 – 0.68 bar) and low flow rates of retentate of 0.315 and 0.425 L·min⁻¹ tested. This suggests that the proportion of methane was inversely proportional to the separation pressure and the flow rate of retentate. However, this was not the case when the real biogas sample was used as inlet. In this instance, an increase of pressure from 0.5 to 0.9 bar and a concurrent increase of retentate flow from 0.156 to 0.250 L·min⁻¹ resulted in an increment of methane partial pressure in retentate from 93.8 to 94.2 vol.%. Nevertheless, further enhancement of pressure and flow rate did not accordingly increase the methane in retentate, Table 1. For the synthetic gas mixture, pressures above 3 bar and flow rates of retentate higher than 1.7 L·min⁻¹ made the separation inefficient limiting the methane proportion in retentate at levels lower than 94 vol.%. On the other hand, very low pressures, even lower than 0.19 (results not shown here) provoked problems in system operation because the retentate flow was diminished at a rather low level that could not have practical application whereas much of the methane was lost in the permeate stream.

The recovery efficiency increased with the increment of pressure for the tests with the real biogas sample, Table 1. A similar trend was also observed for the tests performed with the binary gas mixture of 50 vol.% CO₂ - 50 vol.% CH₄. In this case, the recovery efficiency increased from 47.9% at the pressure of 0.19 bar and retentate flow of 0.315 L·min⁻¹ to 75.1% at the pressure of 3.25 bar and retentate flow of 2.043 L·min⁻¹. Nevertheless, the recovery efficiency was not in all cases proportional to the pressure. For example, an efficiency of 75.1 % observed at the pressure of 3.25 bar and a higher one (76.4%) at the lower separation pressure of 3.18 bar. This was probably attributable to the different flow rates of retentate. In principle, the recovery efficiency is favoured by a low flow rate of retentate¹⁰. In our case, the flow rate was high (2.043 L·min⁻¹) at the pressure of 3.25 bar and lower (1.717 L·min⁻¹) at the pressure of 3.18 bar which likely explains the reduction of the recovery efficiency. However, the interrelation of the two parameters was valid only for pressures higher than 3 bars and was not recurred in any other circumstance where the recovery efficiency was not proportional to the pressure. Consequently, the flow rate of retentate played a substantial role in the recovery of methane at pressures higher than 3 bar, however in all other cases the pressure was the principal parameter defining the recovery efficiency.

Table 1: Gas separation results using synthetic gas and biogas. Experimental errors (\pm) appear in parentheses

Inlet								Retentate								Recovery efficiency
Pressure	Flow	Composition						Flow	Composition							
		CH ₄	CO ₂	O ₂	N ₂	H ₂	H ₂ S		CH ₄	CO ₂	O ₂	N ₂	H ₂	H ₂ S		
Bar	L·min ⁻¹	vol%				ppmv		L·min ⁻¹	vol%				ppmv		%	
Synthetic mixture																
0.19 (0.001)	0.980 (0.005)	50.0 (0.250)	50.0 (0.250)	0.0	0.0	0	0	0.315 (0.002)	96.6 (0.483)	3.4 (0.017)	0.0	0.0	0	0	47.9	
0.68 (0.003)	1.102 (0.006)	50.0 (0.250)	50.0 (0.250)	0.0	0.0	0	0	0.425 (0.002)	95.6 (0.478)	4.4 (0.022)	0.0	0.0	0	0	58.1	
1.16 (0.006)	1.586 (0.008)	50.0 (0.250)	50.0 (0.250)	0.0	0.0	0	0	0.677 (0.003)	94.9 (0.475)	5.1 (0.026)	0.0	0.0	0	0	64.7	
1.69 (0.008)	2.586 (0.013)	50.0 (0.250)	50.0 (0.250)	0.0	0.0	0	0	1.200 (0.006)	94.5 (0.473)	5.5 (0.028)	0.0	0.0	0	0	71.0	
2.15 (0.011)	3.205 (0.016)	50.0 (0.250)	50.0 (0.250)	0.0	0.0	0	0	1.533 (0.008)	94.4 (0.472)	5.6 (0.028)	0.0	0.0	0	0	73.4	
2.25 (0.011)	2.529 (0.013)	50.0 (0.250)	50.0 (0.250)	0.0	0.0	0	0	1.173 (0.006)	94.2 (0.471)	5.8 (0.029)	0.0	0.0	0	0	70.7	
2.69 (0.013)	3.556 (0.018)	50.0 (0.250)	50.0 (0.250)	0.0	0.0	0	0	1.725 (0.009)	94.1 (0.471)	5.9 (0.030)	0.0	0.0	0	0	74.4	
2.73 (0.014)	2.843 (0.014)	50.0 (0.250)	50.0 (0.250)	0.0	0.0	0	0	1.325 (0.007)	94.1 (0.471)	5.9 (0.030)	0.0	0.0	0	0	71.0	
3.18 (0.016)	3.455 (0.017)	50.0 (0.250)	50.0 (0.250)	0.0	0.0	0	0	1.717 (0.009)	93.9 (0.470)	6.1 (0.031)	0.0	0.0	0	0	76.4	
3.25 (0.016)	4.169 (0.021)	50.0 (0.250)	50.0 (0.250)	0.0	0.0	0	0	2.043 (0.010)	93.8 (0.469)	6.2 (0.031)	0.0	0.0	0	0	75.1	
Raw biogas																
0.50 (0.003)	0.561 (0.003)	52.5 (0.263)	42.8 (0.214)	0.9 (0.001)	3.1	600 (1.200)	55 (0.110)	0.156 (0.001)	93.8 (0.469)	1.3 (0.007)	0.9 (0.001)	3.4	5 (0.010)	5 (0.010)	37.8	
0.90 (0.005)	0.823 (0.003)	52.5 (0.263)	42.8 (0.214)	0.9 (0.001)	3.1	600 (1.200)	55 (0.110)	0.250 (0.001)	94.2 (0.471)	1.4 (0.007)	0.8 (0.001)	3.0	10 (0.020)	10 (0.020)	41.7	
1.33 (0.007)	0.871 (0.004)	52.5 (0.263)	42.8 (0.214)	0.9 (0.001)	3.1	600 (1.200)	55 (0.110)	0.330 (0.002)	93.1 (0.466)	1.5 (0.008)	1.0 (0.001)	3.8	75 (0.150)	10 (0.020)	52.8	

*parts per million of volume

The tests performed with the real biogas mixture showed that although the proportion of H₂S in the raw biogas was relatively low (55 ppmv) compared to the typical values of raw agro-biogas (average 1000 ppmv)¹¹, it was further reduced in the retentate stream to values lower than 10 ppmv, Table 1. Hence, it seems that H₂S followed a route similar to that of the CO₂ passing through the water film formed at the membrane surface and egressing the module as a component of the permeate. This provided a purer retentate stream containing only traces of H₂S. In addition to the separation of the two main gases, therefore, the filter allowed for a further purification and desulphurization of methane. It is interesting to note that those small shares of H₂S were almost constant (5 – 10 ppmv) regardless of the pressure (0.50 – 1.33 bar) and the retentate flow rate (0.156 – 0.330 L·min⁻¹). This indicates that H₂S presented a great potential to easily penetrate the membrane at the pressure and flow conditions examined. This is not surprising, however, given that the H₂S is characterized by a higher solubility in water even than the CO₂¹². In addition, the H₂ identified in the raw biogas at a concentration of 600 ppmv was significantly separated from the methane presenting a share of only 75 ppmv in the retentate at the highest pressure (1.33 bar) and flow rate (0.330 L·min⁻¹). In this case, however, both inlet pressure and retentate flow appeared to have an effect on hydrogen separation since increment of those parameters resulted in an increased residual hydrogen partial pressure in retentate.

The flow rates of the inlet gas as well as of the permeate and retentate streams are considered to be vital for the determination of the efficiency of a membrane filter. In our case the flow rates were defined by the pressure, i.e. any shift of pressure was accompanied by a corresponding change in the flow rates of all system streams including inlet gas, permeate and retentate. The dependence of the flows on pressure is presented in Figure 2. In all cases, flow rate gradually increased with increasing inlet pressure. For the inlet gas stream the increment seems to be linear at low pressures from 0.19 – 1.16. Above this range, the increase was also linear however was more intensive as expressed by a slightly higher slope of the

trendline in Figure 2, whereas the flow rate values showed a greater dispersion around the linear trend. The flow rate of the permeate and retentate enhanced more proportionally to the pressure.

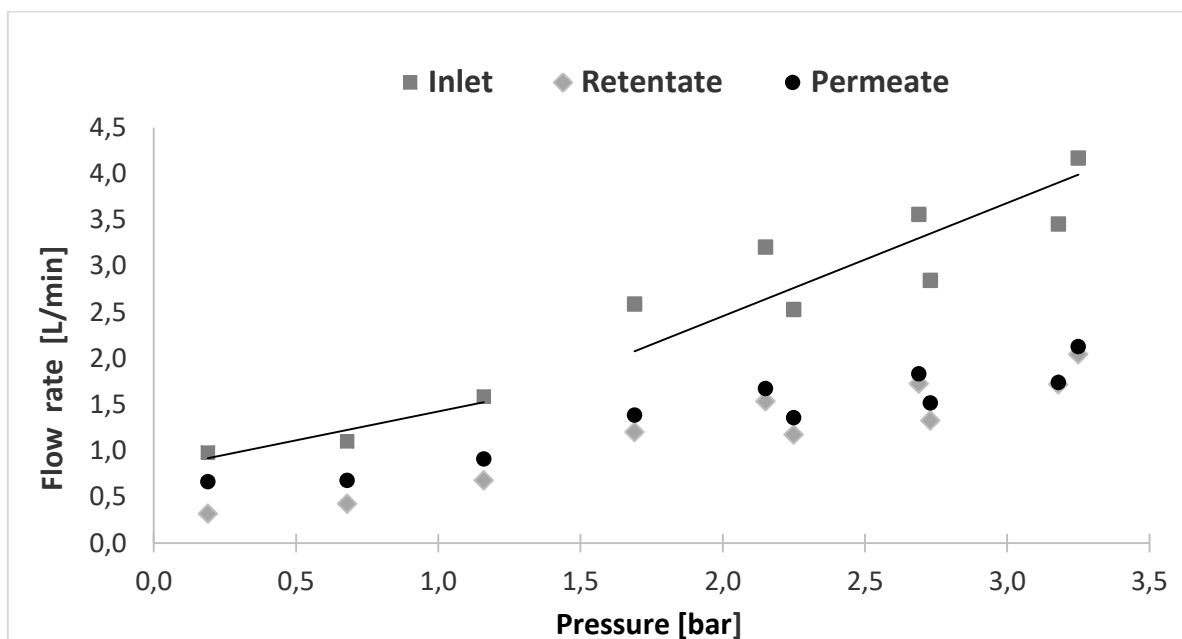


Figure 2: Dependence of flow rates on pressure

The effect of the total pressure on the CO₂ and CH₄ partial pressures in the permeate stream for the synthetic mixture is shown in Figure 3. It is evident that as the input pressure increased the permeate enriched in CO₂. In particular, the concentration of CO₂ increased from 65.3 vol.% at the lowest inlet pressure of 0.19 bar to 80.1 and 79.2 vol.% at the pressures of 3.18 and 3.25 bar, respectively. This increase suggests that the separation of CO₂ occurred on membrane was favoured by the high pressures. It is interesting to mention that at the pressure of 3.18 bar the maximum concentration of CO₂ of 80.1 vol.% was attained when the inlet concentration of CO₂ was at 50 vol.% (synthetic gas), which constitutes a significant increase. The high recovery of CO₂ was comparable to the results of previous studies on gas separation⁹ reporting that an 83.1 vol.% of CO₂ concentration in the permeate stream can be accomplished starting from a 40 vol.% of CO₂ in the feed gas of with the use of a hollow-fiber membrane.

The increase in CO₂ partial pressure was followed by a decrease in the CH₄ proportion in the permeate stream (Figure 3). The CH₄ found in permeate was considered as loss in the separation process given that a number of moles of CH₄ in the permeate passed through the membrane layers together with the water-soluble gases and were separated by the main CH₄ quantity recovered in the retentate stream. The proportion of CH₄ in the permeate was at 34.7 vol.% at the lowest pressure of 0.19 bar whereas at the highest pressure of 3.25 bar it dropped at 20.8 vol.%. At the same time the flow rates of all streams were increased with the total pressure (Figure 2). Membrane surface potentially covered with a greater amount of water with the increment of inlet flow occurred at higher pressures and this might constitute reason for the drop of CH₄ concentration in the permeate. In essence, excess of water on the surface of module presumably incurred an increased thickness of the water film surrounding the membrane, which in turn substantially altered the methane diffusion resistance limiting the passage of the gas to the permeate stream. It can be concluded, therefore, that an increase in pressure which first provoked an enhancement of all flow rates eventually resulted in a reduction of the CH₄ losses in the permeate and therefore in a higher efficiency of the overall membrane system.

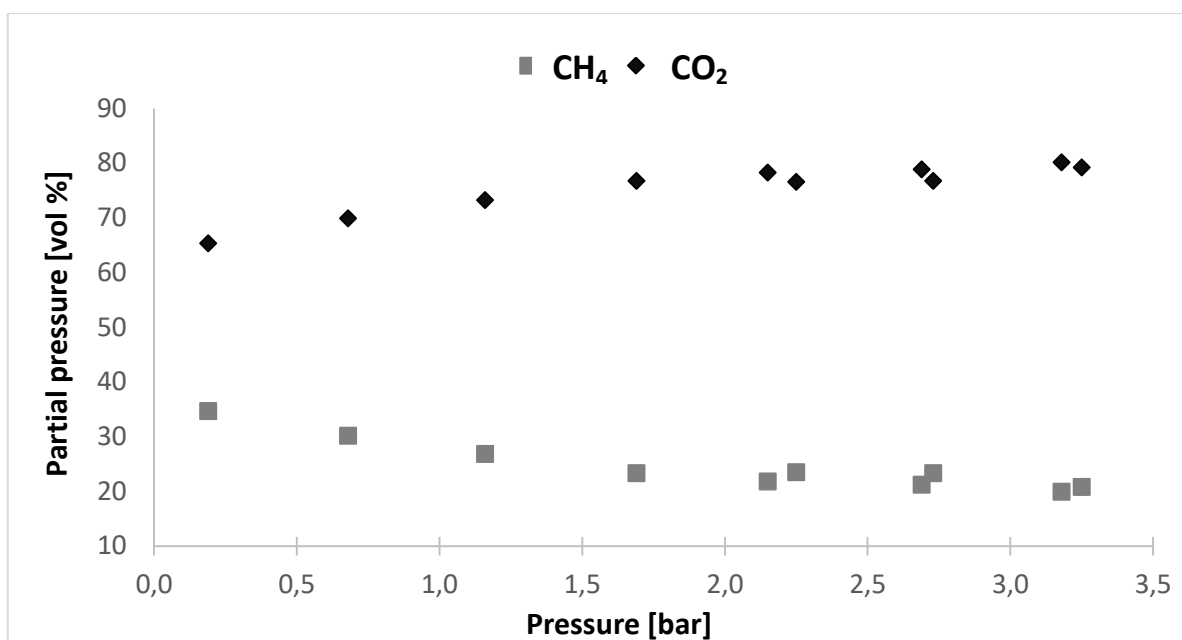


Figure 3: Effect of pressure on the CO₂ and CH₄ content of permeate

Despite of the high recovery and the great proportions of methane in the retentate that obtained under some of the applied conditions in many cases a significant amount of the gas passed through the membrane and lost in the permeate stream. The separation efficiency of the filter is dependent on many operational parameters such as the inlet gas pressure, capacity of the vacuum pump, water enrichment temperature, filter temperature and retentate flow rate. All of these parameters can be optimized in such a way that the methane proportion and recovery efficiency will remain high and at the same time the methane losses will be minimized. Another option for the efficient recovery of those quantities could be the implementation of a more complex setup where a second filtration module would be connected at the permeate side of the filter. A purity in methane higher than even 98 vol.% could be attainable in this case and the biomethane quality would potentially cover the requirements of the International and Czech Republic norms (TPG 902 02) defining its use in the natural gas network.

Conclusions

A laboratory setup for upgrading biogas to biomethane using a water condensate covered spiral-wound membrane was tested. The goal was to obtain an outlet stream containing more than 95 vol.% of methane and minimal concentration of hydrogen sulfide while minimising the methane losses. High purity biomethane was obtained at relatively low inlet pressures, whereas a relatively wide range of pressures allows for production of biomethane of such a high quality that can be directly used in the natural gas grid. Research efforts will be directed to experiments utilising more filtration modules in order to further improve the quality of biomethane and minimize the methane losses.

Acknowledgments

This work was supported by the Ministry of Education, Youth and Sports of the Czech Republic under the projects “COOPERATION” [No. CZ.02.1.01/0.0/0.0/17_049/0008419], “Large Research Infrastructure ENREGAT” [No. LM2018098], and the Doctoral grant competition VSB – Technical University of Ostrava [No. CZ.02.2.69/0.0/0.0/19_073/0016945], project “Study of photochemical, photocatalytic and membrane processes with application in environmental technologies” [DGS/TEAM/2020-004].

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Čištění bioplynu pomocí vodou nabobtnalé kompozitní polyamidové membrány

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Souhrn

Rozsáhlá spotřeba energie spolu s předpokládanou nedostupností relativně levných paliv, jako je zemní plyn, zvýšily zájem o alternativní paliva s vysokou účinností. Pozornost se v poslední době přesunula na zlepšování kvality bioplynu a výrobu vysoce čistého biomethanu. Tato práce testovala vhodnost spirálově vinutého filtru, původně vyvinutého pro čištění vody, pro použití při zušlechťování bioplynu a prezentuje výsledky experimentů provedených za různých podmínek. Filtr byl složen z membrány obsahující v horní části hydrofilní vrstvu tvořenou polyamidem. Vrstva pokrytá tenkým vodním filmem je transformována ve vysoce selektivní separátor schopný oddělit ve vodě rozpustné složky bioplynu, jako je CO₂ a H₂S, od nerozpustného methanu a vytvořit proud biomethanu vysoké čistoty. Za specifických podmínek byl získán proud plynu obsahující až 96 obj. % CH₄.

Klíčová slova: bioplyn, čištění, upgrading, membránová separace, biomethan.