

Research Article

Experimental Investigation of Biogas Production from Wastewater Sludge

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Abstract

The paper has information about experimental investigation of biogas production from wastewater sludge. The effect of incubation temperature, volatile suspended solids contents, and pressure on the amount of the biogas produced was studied in 2L plastic flasks. The experiments were repeated in 25L vessel using the above conditions showed that the CH₄ content increased with time reaching a maximum value in 16 days then decreased. No H₂S gas was detected indicating that biogas produced from wastewater sludge is environmental friendly source of energy. The results show the optimum conditions under which digesters should be operated.

Keywords: Biogas; Methane; Sludge; Wastewater treatment; Anaerobic digestion; Volatile suspended solids

Introduction

Anaerobic Digestion (AD) of organic waste has been recognized as a cost-effective and environmental friendly process to convert organic solid waste into biogas which can be used to produce heat, electricity and fuel [1]. In this digestion process the biodegradable organic wastes are converted to simple molecules by anaerobic bacteria producing a combustible biogas containing about 50–75% methane and 25–50% carbon dioxide [2]. Several feedstock have been used to produce biogas using anaerobic digestion, such as biomass waste [3,4], algae [5,6], food waste [7,8], municipal solid waste [9-16]. Waste Activated Sludge (WAS) is a highly putrescible residue generated from wastewater treatment plants. Activated sludge contains abundant organic matters, bacterial pathogens, nutrients, and high water content. Therefore sludge handling is a big problem in itself from environmental and economic point of view since it accounts for 50% of the operating costs of wastewater treatment plants [17]. Therefore, anaerobic digestion is considered to be an interesting option for WAS treatment as it can allow sludge stabilization to reduce odors and pathogens, sludge decrement, and biogas recovery [18].

Elango et al. [19] investigated the production of biogas from domestic sewage in Chennai metropolitan city using anaerobic digestion process at 26 and 36°C for 25 days. Biogas generation was enhanced by the addition of domestic sewage to MSW. The maximum biogas production (0.36 m³/kg VS added-day), the maximum reduction of Total Solids (TS) (87.6%), VS (88.1%) and Chemical Oxygen Demand (COD) (89.3%) occurred at a feeding rate of 2.9 kg of VS/m³-day. Kalloum et al. [20] studied biogas production from the wastewater treatment plant in Adrar city, Algeria. The diluted sludge with a content of 16 g/l of Total Solids (TS) was fermented under anaerobic conditions during 33 days. The biogas produced was 280.31 Nml with a yield of 30 Nml of biogas/mg of COD removed. The biogas production increased for 26 days then started to decrease. During the first 5 days the pH dropped from 7.0 to 6.3 due to formation of

Volatile Fatty Acids (VFA). After this period pH increases to 6.9 and remains unchanged.

Several attempts have been done to enhance biogas production. Feng et al. [21] enhanced the anaerobic digestion of wasted activated sludge, from municipal wastewater treatment plant in Dalian, China, by the addition of Zero Valent Iron (ZVI) as a reducing material. The production of VFAs was enhanced by 37.3% with ZVI during the hydrolysis and the acidification steps. After the digestion for 20 days, the methane productivity at ZVI of 20 g/L increased by 43.5%, and the sludge reduction ratio increased by 12.2%. Wang et al. [22] enhanced the anaerobic digestion of wasted activated sludge, from wastewater treatment plant in Brisbane, Australia, by 26% using combined Free Nitrous Acid (FNA) and heat pre-treatment. Dębowski et al. [6] studied the effect of Magneto-Active Filling (MAF) on the effectiveness of methane fermentation of dairy wastewaters. It has been found that MAF incorporation into the technological system significantly improved effectiveness of biogas production, increased methane concentration and lowered content of hydrogen sulfide in gaseous metabolites of fermentative bacteria. A significant increase was also observed in the effectiveness of COD removal from dairy wastewaters.

The objective of this study is to investigate the potential production of biogas from the sludge produced at the wastewater treatment plants and to investigate the effect of various operating parameters such as pH, VSS concentration and reactor pressure and temperature on the biogas production rate.

Materials and Methods

Experiment procedure

Two sets of experiments were performed, the first set was performed in a 25L vessel, and the second set was performed in 2L plastic bottles both to mimic batch bioreactors. Details of these two sets of experiments are discussed below:

Table 1: Characteristics of the sludge produced by the wastewater treatment plant.

Parameter	Concentration
PO ₄ -P	22.2 mg L ⁻¹
NH ₄ -N	2.2 mg L ⁻¹
COD	262 mg L ⁻¹
pH	7.5
DO	0.8 mg L ⁻¹
TDS	790 mg L ⁻¹
TSS	2.60wt%
alkalinity	526 mg L ⁻¹
Cd	0.13 mg L ⁻¹
Ni	1.44 mg L ⁻¹
Mn	0.084 mg L ⁻¹
Fe	1.74 mg L ⁻¹
Pb	1.38 mg L ⁻¹
CU	0.05 mg L ⁻¹
Zn	0.418 mg L ⁻¹

Set 1: The second part of the experiments was carried out in 2L plastic bottles filled by 1 L sludge. The purpose of this part is to study the effect of sludge concentration, temperature, and pressure on biogas production. Conditions at which maximum biogas production obtained were used for set 2 experiment carried out in a 25L reactor. The bottles were hooked with a hollow pipe sealed with rubber tube which can be closed to prevent gas escape from the bottles. When sampling is needed, a syringe is inserted through the rubber tube which is then opened allowing the gas to flow into the syringe. The withdrawn gas sample was then sent to the gas analyzer. The sludge as received contains 6% VSS. To study the effect of sludge concentration on biogas produced, the sludge was diluted with distilled water to obtain sludge of Volatile Suspended Solids (VSS) concentrations of 1%, 2%, and 4%. The effect of pressure was investigated by preparing 3 groups of bottles. In the first group the gas in the bottles was analyzed and then it was released till the pressure in the bottles reaches atmospheric pressure. In the second group, the bottles were left without any interference till the end of the incubation period (9 weeks) when a gas sample was taken for analysis. In the third group, a sample from the gas was taken for analysis and the bottles were immediately sealed keeping the bottles pressurized. The reason for the third group is to determine biogas production with time under pressure and compare its results to that of group 1 conducted under atmospheric pressure condition. The effect of temperature on methane generation was studied by carrying out the same tests under constant temperature of 35°C and 25°C. Three bottles for each test were prepared to assure the reproducibility of the results. The data presented is the average of the three readings. The initial weight of the bottles and their weight after sampling were recorded. Knowing the difference in the weight and the concentration of CH₄ gas enables calculating its mass (see results and discussion below, section 3.1).

Set 2: The anaerobic digestion was carried out in a 25L vessel containing a heating element and a thermostat to control the temperature of the vessel at 35°C. The vessel contains a pressure

gauge, a gas sampling point at the top of the vessel and a drainage valve at the bottom for liquid sample withdrawal. The vessel is filled with 23L sludge and gas samples were taken periodically every 2 days for 44 days for analysis. The pressure was read directly from the pressure gauge. The gas samples were withdrawn by a syringe and analyzed for its CH₄, CO₂, and H₂S contents. Every time a gas sample was withdrawn, the pressure was released until it reaches atmospheric pressure. Knowing the pressure of the vessel, the temperature, and methane gas content its amount can be calculated assuming that the gas is ideal (see results and discussion section, section 3.2).

Sludge characterization

The Volatile Suspended Solids (VSS) and the Total Suspended Solids (TSS) of the sludge were determined following the standard methods for the examination of water and wastewater [23]. The pH was measured using portable pH meter (pH 3310 IDS, WTW, Germany). The Chemical Oxygen Demand (COD), the PO₄-P, and the NH₄-N were determined using photometer photo lab (photoLabS12, WTW, Germany). Heavy metals including Fe, Mn, Zn, and Cu as well as other metals such as Na and K were measured using atomic absorption spectrophotometer (Varian SpectraAA-10 and SpectraAA-20, Australia). The alkalinity was determined using the potentiometric method. The sludge characteristics are presented in Table 1.

Gas sampling

The biogas samples were analyzed using a portable gas analyzer (EAGLE, RKI Instruments, Inc. USA) that determines the CH₄, CO₂, and H₂S content of the biogas. The analyzer includes PID (Photo Ionization Detector) capability. Conversion factors for a variety of

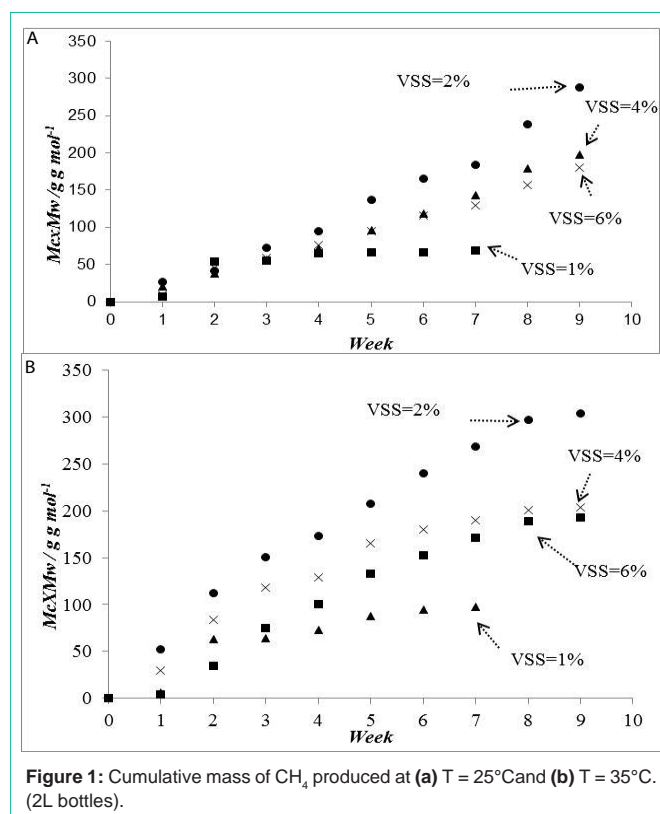
**Figure 1:** Cumulative mass of CH₄ produced at (a) T = 25°C and (b) T = 35°C. (2L bottles).

Table 2: Measured weight loss and CH₄ concentration (CCH₄) from gas samples for group #1 (2L bottles) (The initial VSS weight is 6 g).

Week	1%VSS		2% VSS		4% VSS		6%VSS									
	25°C	35°C	25°C	35°C	25°C	35°C	25°C	35°C								
	Weight loss/week (g week ⁻¹)	Concentration (mg L ⁻¹)	Weight loss/week (g week ⁻¹)	Concentration (mg L ⁻¹)	Weight loss/week (g week ⁻¹)	Concentration (mg L ⁻¹)	Weight loss/week (g week ⁻¹)	Concentration (mg L ⁻¹)								
1	0.85	350	0.50	450	0.60	1800	0.90	2300	0.85	1000	0.80	1450	0.50	740	0.40	400
2	1.10	1750	1.75	1300	0.40	1550	0.60	3900	0.55	1300	0.75	2850	1.20	1250	0.90	1350
3	0.15	190	0.25	35	0.50	2500	0.70	2200	0.50	1450	0.60	2250	0.40	1300	0.70	2250
4	0.40	1050	0.60	630	0.35	2550	0.35	2500	0.45	1400	0.35	1200	0.45	1550	0.55	1850
5	0.13	150	0.55	1050	0.65	2700	0.60	2250	0.60	1650	0.85	1700	0.50	1550	0.80	1600
6	0.17	200	0.50	500	0.50	2300	0.65	2000	0.45	2000	0.55	1100	0.50	1700	0.55	1420
7	0.25	340	0.30	450	0.25	3100	0.60	1850	0.45	2300	0.40	940	0.40	1450	0.55	1300
8					0.60	3700	0.60	1900	0.60	2450	0.50	850	0.54	2000	0.60	1150
9					0.65	3100	0.18	1500	0.35	2100	0.19	670	0.51	1900	0.17	900

common gases are programmed into the PID sensor and LEL/PPM catalytic sensor.

Results and Discussion

Results of set 1 (plastic bottles)

Group # 1: Table 2 shows measured concentration of CH₄ (C_{CH₄}) in the biogas produced for the various VSS% tested. The mass of CH₄ produced (M_{CH₄}) was calculated from the following equation assuming that the gas is ideal

$$M_{CH_4} = \frac{mRTC_{CH_4}}{1000P_aM_w} \quad (1)$$

Where: m is the mass in grams of the measured biogas released from the bottles determined by weighing the bottles before and after sampling, M_w is the molar mass of the biogas, P_a is the atmospheric pressure, and R is the international gas constant. The factor 1000 appears in the equation is a conversion factor to get the mass in grams.

The cumulative mass of CH₄ produced, presented as M_c = Σ(M_{CH₄} × M_w) because the exact value of M_w is not known since complete analysis of the biogas was not possible, at 25°C and at 35°C for the various VSS contents examined is shown in Figure 1-a and Figure 1-b respectively. It should be noted that the initial mass of VSS present

in the bottles is 6 g. It can be seen that the biogas production was higher at VSS content of 2% compared to other VSS contents studied. As expected higher incubation temperature yield higher amount of methane gas. To illustrate this, the data for 2VSS% at 35°C and 25°C is plotted in Figure 2. The above results are in agreement with those found by other researchers [24-28].

Group # 2: In this group of experiments, no gas samples were withdrawn and analyzed till the end of the experiment (9 weeks). The

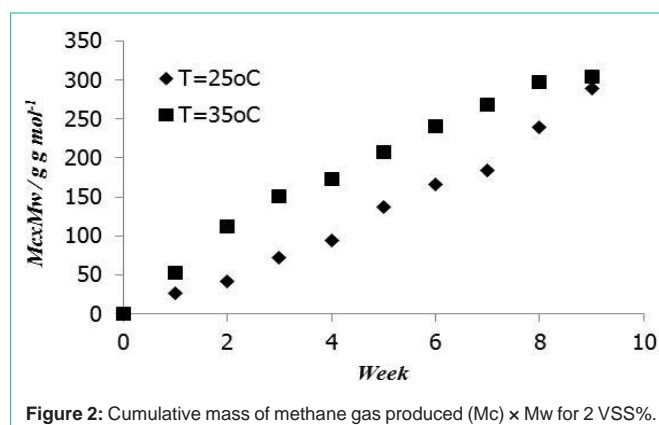


Figure 2: Cumulative mass of methane gas produced (Mc) × Mw for 2 VSS%.

Table 3: Measured CH₄ concentration (C_{CH₄}), % weight loss after 9 weeks at 25°C and 35°C for group #2 (2L bottles) (The initial VSS weight is 6 g).

% VSS content	25°C		35°C	
	CH ₄ concentration (mg L ⁻¹)	% Weight Loss	CH ₄ Concentration (mg L ⁻¹)	% Weight Loss
1%	336	4.2	450	10.8
2%	840	81.7	1820	72.5
4%	800	80.0	1480	67.5
6%	828	79.2	1125	70.8

Table 4: Comparison between weight loss of the sample in group #1 (atmospheric pressure) and group #2 (under pressure) after 9 weeks. (The initial VSS weight is 6 g).

%VSS content	group #1		group #2	
	25°C	35°C	25°C	35°C
1	-	-	4.2	10.8
2	75.0	86.3	81.7	72.5
4	80.0	83.2	80.0	67.5
6	83.3	87.0	79.2	70.8

results for the experiments performed at 25°C and at 35°C are shown in Table 3. As can be seen the concentration of CH₄ at 35°C is higher than that at 25°C similar to that observed for group #1. However the percent mass loss is larger at 25°C compared to that at 35°C. This can be explained as follows: at 35°C more biogas and hence more CO₂ is produced initially compared to that at 25°C. Since the gas was not released from the bottle, the produced CO₂ gas dissolves in the water producing HCO₃⁻ reducing the pH and that in turn reduces the activity of the bacteria. Since the reduction in the pH value is faster at 35°C compared to that at 25°C the weight loss at 25°C is larger. This result recommends that bioreactors should operate at atmospheric pressure and the produced biogas should be removed as soon as it forms. To confirm the above result, weight loss for group #2 where the bottles were under pressure to those of group #1 where the pressure in the bottles was atmospheric is presented in Table 4. The results confirm the findings mentioned above that releasing the pressure or withdrawing the gas as it forms gives better gas production.

Group # 3: In this set of experiments, gas samples were withdrawn for analysis and the bottles were then immediately closed keeping the

Table 5: A comparison between the cumulative mass × M_v(Mc) obtained at 25°C and 35°C for group #1 (atmospheric pressure) and group #3 (under pressure). (The initial VSS weight is 6 g).

Week	Group #1				Group #3			
	2%		6%		2%		6%	
	∑ Mc 25°C	∑ Mc 35°C	∑ Mc 25°C	∑ Mc 35°C	∑ Mc 25°C	∑ Mc 35°C	∑ Mc 25°C	∑ Mc 35°C
1	10.0	15.0	8.33	6.66	20.8	10.8	12.5	11.7
2	16.7	25.0	28.3	21.7	30.8	20	23.3	20.8
3	25.0	36.7	35.0	33.3	44.2	30	35	31.7
4	30.8	42.5	42.5	42.5	55	43.3	49.2	44.2
5	41.7	52.5	50.8	55.8	75	65	65	65
6	50.0	63.3	59.2	65.0	85.8	76.7	75.8	79.2
7	54.2	73.3	65.8	74.2	-	-	-	-
8	64.2	83.3	74.8	84.2	-	-	-	-
9	75.0	86.3	83.3	87.0	-	-	-	-

contents of the bottles under pressure. The results are presented in Table 5 where it can be seen that the conversion at 25°C is higher compared to that at 35°C confirming the results obtained for group # 2. The results also show that conversion at VSS content of 2% is larger than those at VSS content of 6% as was observed in the previous sets of experiments. Comparing the results of group # 3 where the bottle contents were under pressure to those of group #1 where the contents were under atmospheric pressure is also shown in Table 5. It can be seen that group #3 gives lower biogas conversion compared to those of group #1 which confirms the results obtained previously from group #2.

Results of set2 (25L vessel)

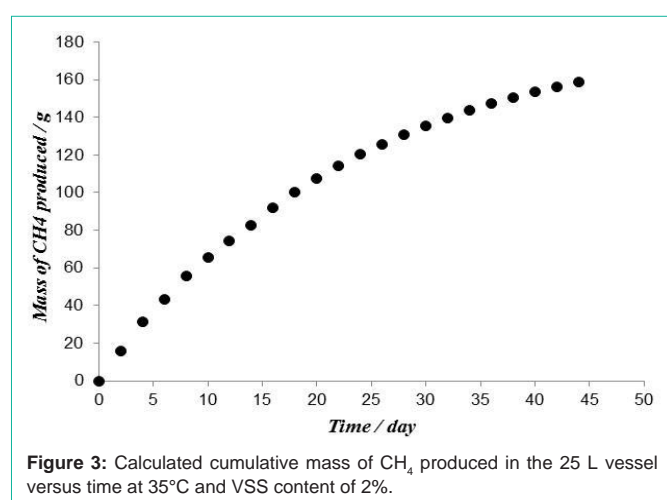
The measured CH₄ concentration (C_{CH₄}), CO₂ volume percent, and the measured gauge pressure of the vessel (P_g), are shown in Table 6. The pressure and the concentration allow calculating the mass of CH₄ produced (M_{CH₄}) assuming that the gas is ideal

$$M_{CH_4} = \frac{C_{CH_4}}{1000} \left(\frac{P_v}{P_a} \right) \left(\frac{T_a}{T_b} \right) V_v \quad (2)$$

Where: the subscript “a” and “v” indicate the ambient and the vessel conditions respectively, V_v is the volume of the space in the vessel above the liquid which is equal to 2L, P is the absolute pressure, and T is the absolute temperature. The factor 1000 appears in the equation is a conversion factor to get the mass in grams. Figure 3 shows the cumulative mass of CH₄ gas produced versus time. As can be seen the rate is fast at the beginning then decreases and eventually becomes zero. The variation of pH and CH₄ concentration are plotted in Figure 4. It can be seen that the biodegradation process was fast since the amount of CH₄ reaches 6750 ppm within 2 days. The CH₄ content of the biogas continues to increase and reaches its maximum value within 16 days then starts to decline slowly. At this time the bacteria enters the decay phase, when the rate of its death exceeds that of its reproduction [22]. The pH values in Figure 4 drops from 7.5 to 7.1 within the first 8 days and then increases again to 7.5. The reason for this is that Volatile Fatty Acids (VFA) is produced by the acidogenic bacteria causing the pH to drop. At the same time methanogenic bacteria produces CH₄ gas from the produced VFA causing the pH to rise. The experimental results indicate that within the first 8 days the rate of VFA production is faster than its consumption to produce CH₄ gas which causes the pH to drop. However after 8 days the rate of CH₄ production exceeds that of VFA resulting in an increase in the pH value till the two rates become equal causing the pH value to remain constant [20]. Moreover the reaction of CO₂, which is soluble in water, with the hydroxide ions results in the formation of HCO₃⁻, which tends to restore the neutrality of the process pH. The above results are in accordance with those of [20]. Table 6 also shows that pressure values are high at the beginning then it drops gradually. This

Table 6: Measured CH₄ concentration (C_{CH₄}), gauge pressure (P_g), and CO₂ volume (%) in the 25 L vessel. The VSS content is 2wt%. H₂S concentration = 0.

Day	C _{CH₄} (mg L ⁻¹)	P _g (bar)	M _{CH₄} (g)	CO ₂ (vol%)	Day	C _{CH₄} (mg L ⁻¹)	P _g (bar)	M _{CH₄} (g)	CO ₂ (vol%)
2	6750	1.20	15.7	3.18	24	7600	0.40	5.9	5
4	8000	1.00	15.5	4.36	26	7370	0.39	5.6	5
6	8950	0.71	12.3	4.74	28	7150	0.37	5.1	5
8	8900	0.70	12.1	5	30	6780	0.35	4.6	5
10	8750	0.60	10.2	5	32	6560	0.33	4.2	5
12	8900	0.50	8.6	5	34	6420	0.31	3.9	5
14	9750	0.48	8.5	5	36	6250	0.30	3.6	5
16	9350	0.51	9.2	5	38	5860	0.29	3.3	5
18	8910	0.48	8.3	5	40	5700	0.27	3.0	5
20	8500	0.45	7.4	5	42	5450	0.26	2.7	5
22	8050	0.43	6.7	5	44	5250	0.25	2.5	5

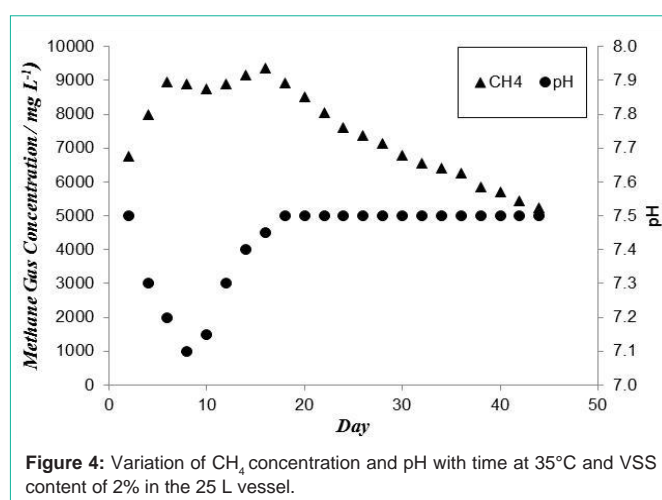


confirms that biogas production rate is fast at the beginning then drops gradually. The amount of CO₂ also increases with time until it reaches the maximum detection limit of the instrument (5% Vol.). The analysis shows that no H₂S was detected in the biogas produced (or the concentration is below the detection limit of the instrument). This may be attributed to the low sulfate concentration [25] since the rate of SRB growth is much faster than that of Methane Producing Bacteria (MPB) [26]. The undetected H₂S concentration under such fermentation conditions indicates that sludge is an environmentally friendly source of energy.

Conclusion

The experimental investigation performed in this research showed that the sludge produced from the wastewater treatment plant is an environmental friendly potential source of biogas. The following conclusions can be withdrawn from the results obtained in this study:

1. Amount of biogas produced from sludge and its CH₄ content depends on the Volatile Suspended Solids (VSS) concentration. The highest amount of biogas production was achieved at 2% VSS content.
2. Biogas production and methane concentration at 35°C was higher than that at 25°C.



3. Bioreactor pressure negatively affects biogas production and its methane content. It is recommended to withdraw the produced biogas as soon as it forms.

Acknowledgment

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Nomenclature

C _{CH₄}	[mgL ⁻¹]	Concentration of methane gas
M _c	[g]	Cumulative mass of methane gas produced
M _w	[g gmol ⁻¹]	Molar mass of the biogas
P	[Pa]	Absolute pressure
R	[N.mkmol ⁻¹ K ⁻¹]	International gas constant
T	[K]	Absolute temperature

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