Scientific Research Publishing

Energy and Power Engineering, 2023, 15, 229-240
https://www.scirp.org/journal/epe
ISSN Online: 1947-3818
ISSN Print: 1949-243X

Kinetics and Process Studies of the Potential for Transformation of Biogas to Biomethane and Liquefaction using Cryogenic Liquid for Domestic Applications

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How to cite this paper: Ogembo, B., Njogu, P. and Ochieng, F. (2023) Kinetics and Process Studies of the Potential for Transformation of Biogas to Biomethane and Liquefaction using Cryogenic Liquid for Domestic Applications. *Energy and Power Engineering*, **15**, 229-240.
https://doi.org/10.4236/epe.2023.156011

Received: May 16, 2023
Accepted: June 27, 2023
Published: June 30, 2023

Abstract

The present work dealt with the generation, purifying and liquefaction of biomethane to improve energy density using local materials for domestic applications. Cow dung was sourced at JKUAT dairy farm and experiments were conducted at JKUAT Bioenergy laboratory using biogas generated in laboratory scale 1 m^3 bioreactors. Experiments were done in triplicates and repeated under different conditions to get the optimal conditions. The results showed that enhanced cow dung substrate displayed an improved fermentation process with increased biogas yields. Purified biogas optimized methane content from 56% ± 0.18% for raw biogas to 95% ± 0.98% for biomethane which was ideal for liquefaction.

Keywords

Biogas, Bio-Methane, Catalysis, Purification, Liquefaction, Bio-Energy, Kinetics

1. Introduction

Global energy demand is increasing with fossil fuels expected to be depleted by the year 2070 [1] [2]. Overreliance on a few energy sources is a threat as fluctuation in the world market could destabilize economies. Kenya has intensified in research and exploitation of various energy productions such as geothermal, hydro and biogas to diversify energy sources to meet demand [3] [4] [5]. Seasonal fluctuations in the prices of synthetic energy have caused an upsurge in search of different energy opportunities to subsidize the existing options for industrial,

commercial and domestic applications. Extensive use of fossil energy has contributed to increased climatic events in myriad ways such as persistent drought, flooding, heat waves and emergence of resistant pathogens [6]. Due to climate change concerns, different countries have opted to utilization of renewable energy sources to supplement petroleum energy [7] [8] [9]. There has been increased focus on biogas exploitation and research with generation, cleaning; upgrading, storage and liquefication having been poorly addressed [10] [11] [12]. Biogas has been utilized in different applications such as; heating, lighting and running SI engines among others [13] [14]. **Figure 1** summarizes biogas energy generation, purification and liquefaction for domestic applications.

Kinetics and processes in biogas production try to study the rate generation proceeds and measure changes in volumes. Production can be hastened by application of a catalyst and process optimization [17] [18]. In this study *P. glaucum* has been used as enhancers in the fermentation of slurry with improved biogas generation. Multiplication of micro-organisms during fermentation process causes increased biogas yields [19] [20] [21]. CO_2 and H_2S are the main components to be purified to obtain CH_4 which is an essential element in biogas energy [22] [23] [24] [25]. [26] reported that LBG energy contains 99% CH_4, its high methane number, being utilized in the same way as LNG using the same infrastructure with its production and use contributing positively to the conservation of environment. Liquefaction of biogas refers to cooling purified gaseous biomethane to a temperature below its condensation point. [27] argued that the most common liquefaction techniques used are closed-loop and opened-loop cycles. [28] [29] point out that in open-loop cycle the refrigerant is part of the feed gas, whereas in closed-loop cycle biogas cooling and liquefaction is attained by an external refrigerant that flows continuously in a separate circuit. These liquefaction techniques have been used for several years in technical gas industry on a much larger scale in biogas plants. To increase usage in small scale applications, biogas should be value added as dealt with in this research.

2. Materials and Methods

The study used experimental research design in which four broad sections were explored which included determination of optimal biogas production conditions using *P. glaucum* as generation enhancers, development and testing of small-scale biogas purification and upgrading system, and determination of potential and conditions for biomethane liquefaction using liquid nitrogen as

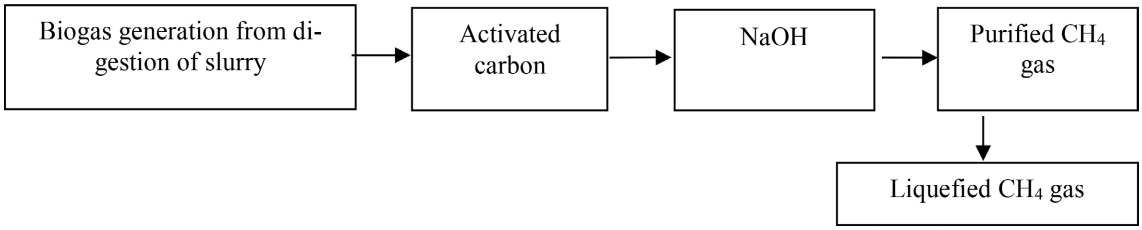

Figure 1. Schematic diagram for biogas generation, cleaning, upgrading and liquefication [15] [16].

cryogenic liquid.

1) Determination of optimal biogas production conditions using *P. glaucum* as generation enhancers

Three sets of flexible batch reactors size 1 m^3 were run concurrently to produce biogas under similar mesophilic generation conditions. Cow dung was obtained at JKUAT cattle rearing farm and sorted. 1 m^3 tubular batch reactors fermented cow dung substrate for biogas generation. The reactors processed 100 kg, 200 kg and 300 kg of cow dung inoculated with 1 kg of powdered *P. glaucum* in the ratio 100:1 and 200:1 and 300:1; water was added in the ratio 1:1.5w/w as fermentation process and biogas generation began. All reactors were monitored for variations of pH, TDS and EC as they operated at mean temperatures of 35˚C throughout HRT period. Kinetics studies for enhanced biogas production were achieved with powdered *P. glaucum* inoculation at a ratio of 100:1w/w. Biogas samples were collected every third day of generation to monitor variations of pH, TDS and EC so as to establish generation condition in the reactors.

2) Development and testing of small-scale biogas purification and upgrading system

The purification layout was connected to the bioreactor to ensure biogas produced was continuously cleaned and upgraded to optimize CH_4 concentration level. Activated carbon impregnated KOH adsorbed H_2S, NaOH scrubbed CO_2 as CH_4 content optimized and silica gel absolutely dried the gas. The bioreactor was assembled in series with a 60 mm column of activated carbon impregnated 10 g KOH, a column packed with 10 g NaOH and drying column packed with 10 g silica gel. Biogas entered at the top and exited at the bottom of activated carbon column to adsorb H_2S as well as water vapor, entered the second column packed with NaOH to scrub CO_2 levels as CH_4 level optimized before total drying to improve flammability. Samples of raw and purified biogas were collected in 5 ml syringes for analysis using GC equipment. 1 ml of raw and purified biogas were injected in the GC with 10 minute retention time to display results in the GC paper. A gas outlet pipe was joint-sealed to ensure biogas tight seal as shown in Figure 2.

Biogas cleaning and upgrading had a significant effect on CH_4 concentration. To effectively analyze biogas required collection of samples in triplicate under tight-sealed conditions to prevent any leakage.

3) Determination of potential and conditions for biomethane liquefaction using nitrogen as cryogenic liquid

Purified biomethane samples of boiling point −162.8˚C were frozen in a 3 litre

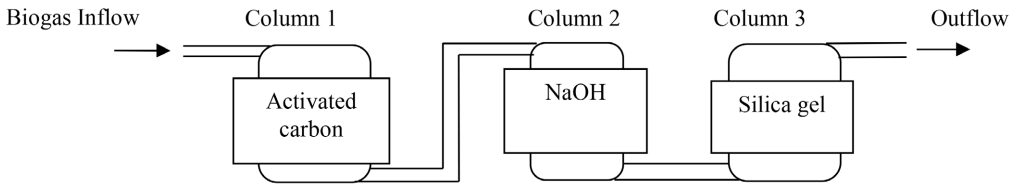

Figure 2. Biogas purification layout.

nitrogen liquid-filled cold box of boiling point −195.8˚C to obtain the liquid. 20 ml syringes collected biomethane samples, pressure gauge determined sample collection pressure; thermo-detector (100˚C) detected initial temperature of the collected sample and timer monitored duration at which freezing occurred. The temperature for purified biogas sample was 21˚C when packed in clear 20 ml syringes, which was tightly sealed before putting it in the cold box to be frozen to liquid.

3. Results and Discussions

1) Optimal biogas production conditions using *P. glaucum* as generation enhancers

Optimum generation was on 6th, 7th, 9th, 10th and 12th days with pH values of 7.63 ± 0.25, 7.76 ± 0.13, 7.89 ± 0.02, 7.85 ± 0.11 and 7.95 ± 0.25 respectively. There was a slowdown in growth and accumulation of acidogenic bacteria responsible for breakdown of TDS because reactors operated between alkaline and neutral conditions thus affecting generation volume. Raw biogas samples were drawn by 10 ml syringes at gas outlet point every 3rd day, analyzed for mean variations of pH, TDS and EC by pH a meter. Data obtained were compared and recorded for further analysis to determine optimum reactor operating conditions.

2) Optimal conditions for cow dung: water and slurry: *P. glaucum*

Biogas generation peaked at mesophilic temperatures of 30˚C, 35˚C and 40˚C respectively as reactors were operated in triplicate in a HRT of 13 days. Cow dung to water ratio was 1:1.5 w/w and 1 kg of inoculums (*P. glaucum*) were charged into three reactors whereas control experiment utilized cow dung water ratio 1:1.5. Biogas generation was monitored for 13 days HRT and samples were collected for pH, TDS and EC analysis. Catalyzed generation peaked at a mean volume of 577.9 ± 0.08 mL while for un-catalyzed was 302.4 ± 0.06 mL. Optimum generation occurred at a mean temperature of 35˚C ± 1˚C. With the onset of digestion, pH value was negligible since the reactors were new in the process. Digestion peaked with optimum generation being on the 6th, 7th, 9th, 10th and 12th days with pH values of 7.63 ± 0.25, 7.76 ± 0.13, 7.89 ± 0.02, 7.85 ± 0.11 and 7.95 ± 0.25 respectively. There was a slowdown in growth and accumulation of acidogenic bacteria to be utilized for a breakdown of TDS since the digester operated between alkaline and neutral conditions thus affecting reactor volume and reduced generation at mean values as presented in Table 1.

Data generated revealed that biogas production followed an exponential curve as shown in Figure 3.

Production for catalyzed substrate displayed increased volumes at different values of pH, TDS and EC, which is presented in Table 2. The catalyzed reactor used cow dung substrate with 1 kg of powdered *P. glaucum* in the ratio 100:1 mixed with water in a ratio of 1:1.5, which peaked early and gained stability. Data obtained revealed that optimum generation occurred on 1st, 2nd, 3rd , 4th , 5th ,

Table 1. Mean values for un-catalyzed process.

Day	Biogas Volume (mL)	pH	TDS	EC
0	0	0	0	0
1	65 ± 0.06	8.21 ± 0.24	3.10 ± 0.02	7.56 ± 0.03
2	110 ± 0.08	8.20 ± 0.15	3.55 ± 0.10	7.88 ± 0.01
3	140 ± 0.04	8.20 ± 0.12	3.55 ± 0.02	7.88 ± 0.05
4	250 ± 0.09	8.18 ± 0.32	3.85 ± 0.04	7.98 ± 0.04
5	287 ± 0.12	8.97 ± 0.22	4.08 ± 0.02	8.22 ± 0.02
6	310 ± 0.07	7.63 ± 0.25	4.48 ± 0.12	8.97 ± 0.12
7	398 ± 0.04	7.76 ± 0.13	4.66 ± 0.11	9.41 ± 0.08
8	421 ± 0.05	8.27 ± 0.23	4.35 ± 0.10	8.81 ± 0.11
9	440 ± 0.03	7.89 ± 0.02	4.37 ± 0.03	8.88 ± 0.04
10	450 ± 0.06	7.85 ± 0.11	4.56 ± 0.02	9.16 ± 0.06
11	500 ± 0.14	8.31 ± 0.01	3.82 ± 0.04	7.42 ± 0.03
	560 ± 0.02	7.95 ± 0.25	3.72 ± 0.03	7.50 ± 0.02
Mean	302.4 ± 0.06	7.50 ± 0.16	3.70 ± 0.05	7.67 ± 0.20

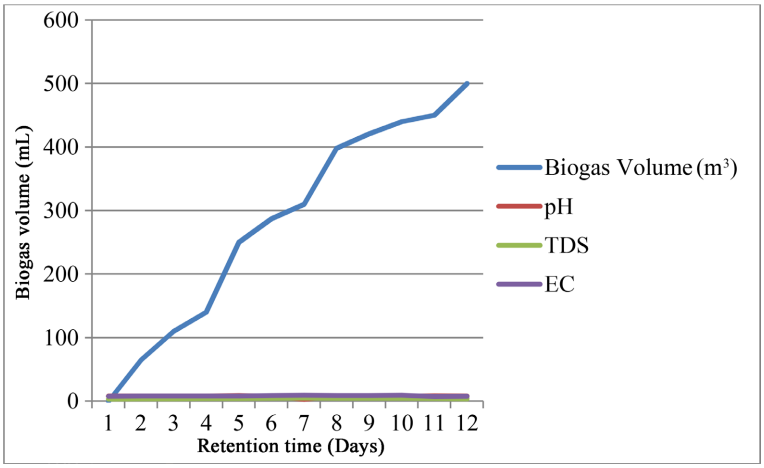

Figure 3. Curve for un-catalyzed process.

6th, and 8th day with variations in pH values of 7.93 ± 0.41, 7.93 ± 0.22, 7.81 ± 0.31, 7.86 ± 0.67, 7.88 ± 0.34, 7.84 ± 0.31 and 7.56 ± 0.42 were favorable for increased generation and methane accumulation.

Biogas generated was cleaned and upgraded continuously with mean values resulting from reaction in the digester displayed by sigmoidal curve in **Figure 4**.

Catalyzed substrates displayed higher daily volumes with reactors operating at mean pH of 7.08 ± 0.05, which was closer to neutral conditions and hence favorable for accumulation of microbes for digestion process. Use of enhancers increased production volumes since it improved fermentation process. Cumulative

Table 2. Mean yields for catalyzed process.

Day	Biogas Volume(mL)	pH	TDS	EC
0	0	8.25 ± 0.32	2.98 ± 0.01	6.85 ± 0.97
1	66 ± 0.09	7.93 ± 0.41	3.15 ± 0.21	7.01 ± 0.98
2	120 ± 0.10	7.93 ± 0.22	3.15 ± 0.36	7.01 ± 0.42
3	164 ± 0.08	7.81 ± 0.31	3.37 ± 0.32	7.43 ± 0.25
4	248 ± 0.10	7.86 ± 0.67	4.30 ± 0.31	8.20 ± 0.23
5	454 ± 0.10	7.88 ± 0.34	4.39 ± 0.42	8.86 ± 0.12
6	569 ± 0.09	7.84 ± 0.31	4.74 ± 0.52	9.49 ± 0.32
7	679 ± 0.10	8.25 ± 0.51	4.53 ± 0.67	9.04 ± 0.25
8	769 ± 0.08	7.56 ± 0.42	4.68 ± 0.20	9.36 ± 0.22
9	859 ± 0.10	9.90 ± 0.35	4.52 ± 0.12	9.00 ± 0.32
10	1098 ± 0.10	8.55 ± 0.21	3.69 ± 0.33	7.49 ± 0.53
11	1186 ± 0.09	8.14 ± 0.30	3.71 ± 0.42	7.36 ± 0.24
12	1300 ± 0.10	8.25 ± 0.42	2.98 ± 0.51	6.85 ± 0.26
Mean	577.9 ± 0.08	8.17 ± 0.37	3.86 ± 0.34	8.0 ± 0.39

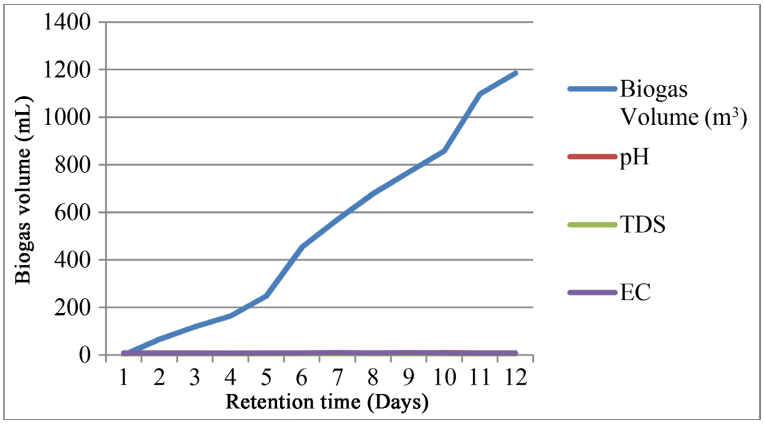

Figure 4. Mean production curve.

yields for catalyzed and un-catalyzed substrates for 13 day generation period are presented in Table 3.

Data presented in Figure 5 denoted an exponential increase in volume for inoculated reactor as fermentation process increased rapidly over the generation period.

3) Small scale biogas purification and upgrading system

Methane production for catalyzed substrate was 56.07% ± 0.18% whereas for un-catalyzed was 48.7% ± 0.2%. Catalyzed substrate heightened biogas generation with optimum methane content is displayed in Table 4.

NaOH reacted with CO_2 which resulted in chemical equation: $2NaOH_{(aq)} \pm CO_{2(g)} = NaCO_{3(aq)} \pm H_2O_{(l)}$.

Table 3. Cumulative volumes for catalyzed and un-catalyzed processes.

Day	Catalyzed process	Un-catalyzed process
0	0	0
1	66 ± 0.09	65 ± 0.06
2	186 ± 0.10	110 ± 0.08
3	248 ± 0.10	140 ± 0.04
4	350 ± 0.08	250 ± 0.09
5	454 ± 0.10	287 ± 0.12
6	569 ± 0.09	310 ± 0.07
7	679 ± 0.10	398 ± 0.04
8	769 ± 0.08	421 ± 0.05
9	859 ± 0.10	440 ± 0.03
10	1098 ± 0.10	450 ± 0.06
11	1186 ± 0.09	500 ± 0.14
12	7513.13 ± 1.0	3931.8 ± 0.78

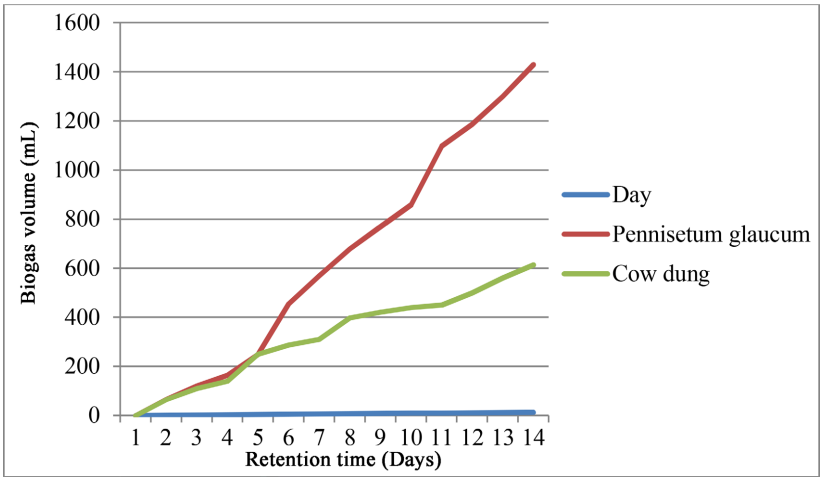

Figure 5. Daily production volume.

Table 4. Biogas composition for catalyzed and un-catalyzed process (n = 3).

Component	Catalyzed substrate	Un-catalyzed substrate
Methane	56.07 ± 0.36	48.7 ± 0.22
Carbon dioxide	27.09 ± 0.32	33.7 ± 2.10
Hydrogen sulphide	0.01 ± 0.03	0.22 ± 0.14

Purification process reaction optimized CH_4 content to about 95% ± 0.98%.

Samples were analyzed by gas chromatography for raw and purified biogas; raw biogas displayed a mean value of 56.07% ± 0.18% while the purified one showed a mean of 95% ± 0.98% CH_4 as shown in Table 5.

Experiments were done in triplicate. NaOH reacted with CO_2 which resulted in increased CH_4 content in biogas is summarized in Table 6.

Results obtained showed that duration for biogas purification in the layout had a significant effect on CH_4 concentration. Activated carbon impregnated KOH reacted with H_2S resulting in potassium sulphate and water formation is displayed in the following chemical equation:

$$H_2S_{(g)} + KOH_{(aq)} = K_2S_{(s)} + H_2O_{(l)} \quad (1)$$

NaOH reacted with CO_2 which resulted in the formation of aqueous solution of sodium carbonate and water is indicated in the chemical equation:

$$2NaOH_{(aq)} + CO_{2(g)} = NaCO_{3(aq)} + H_2O_{(l)} \quad (2)$$

Ionically,

$$2OH_{(aq)} + CO_{2(g)} = CO_{3\ (aq)}^{2-} + H_2O_{(l)} \quad (3)$$

CO_2 absorption increased as biogas upgrading progressed with optimal CH_4 percentage. Total dryness of biogas was achieved using silica gel as the gas passed through it. Increased CH_4 content in biogas from 56.07% ± 0.18% to 95% ± 0.98% denoted progressive absorption of CO_2. Its absorption was rapid at the beginning of the process but declined afterwards in about 20 minutes due to saturated NaOH material. The purification layout could be regenerated by hot flushing of the activated carbon and drying silica gel in the air. It was denoted that combustion of raw biogas took $5^{1/2}$ minutes to heat 1 lit of water from a temperature of 4˚C to 50˚C while biomethane took 3 minutes to achieve the same results as summarized in Table 7.

Table 5. Methane yields for raw and purified biogas (n = 3).

No.	Biogas	CH_4 content (%)
1	Raw biogas	56.07 ± 0.18
2	Cleaned biogas	95 ± 0.98

Table 6. Results of purified biogas content (n = 3).

Main element	Raw biogas (%)	Purified biogas (%)
CH_4	56.07 ± 0.18	95 ± 0.98
CO_2	27.09 ± 0.32	0.46 ± 0.41
H_2S	0.01 ± 0.03	0.006 ± 0.36

Table 7. Flame performance test.

No.	Biogas	Biogas pressure (Kpa)	Amount of water (lit)	Time (minutes) for temperature rise to 50˚C
1	Raw biogas	4.0	1	5 1/2
2	Purified biogas	4.0	1	3

Flame performance indicated that biomethane possessed higher energy density to raise water temperature from 4˚C to 50˚C.

4) Potential and conditions for biomethane liquefaction using nitrogen as cryogenic liquid

Purified samples were frozen in a 3-litre nitrogen liquid-filled cold box of boiling point −195.8˚C as biogas of boiling point −162.8˚C was frozen. Samples were filled in syringes and carefully inserted in the cold box as retention time was observed for change of state. The results obtained are summarized in Table 8.

The results indicated that a retention time of 10 minutes was ideal for 20 ml biomethane to liquefy into 8ml liquid.

a) Efficiency of purified biogas

Cold water was poured in aluminum container and carefully heated to 50˚C as temperature as time taken was recorded. Heat energy consumed by boiling water using biogas is given by the equation;

$$Q = mc\left(T_2 - T_1\right) \tag{i}$$

where: Q—heat flow, m—mass of water (1 lit = 1 kg) at 4˚C, c_w—specific heat capacity of water, 4200 J/kg K, c_a—specific heat capacity of aluminum vessel, 400 J/kg K , mass of aluminum vessel = 1.5 kg, T_2—higher temperature 50˚C (323 K) and T_1—lower temperature 4˚C (277K).

Therefore, Heat energy consumed = Heat of the boiling water + Heat of the boiling vessel (aluminum)

$$Q = 1\times 4200\times\left(323-277\right)+1.5\times 400\times\left(323-277\right)=165.6\text{ KJ}$$

165.6 KJ is the amount of heat energy required to raise the temperature of water to 50˚C.

For 1 hour Q = 165.6 KJ/60minutes = 2.76 KJ/h.

$$\text{Volumetric consumption of purified biogas (m}^3\text{/h)} = \frac{Q}{CV} = \frac{2.76}{26} = 0.106\text{ m}^3\text{/h} \tag{ii}$$

Volumetric flow rate by fire appliance was 0.106 m^3/h as temperature of water rose from 4˚C through to 50˚C.

Table 8. Biogas liquefaction.

Biomethane Sample (ml)	Pressure (Kpa)	Temperature (˚C)	Time (min)	Results	Deductions
20	1.019	−196	5	No change of state	No change occurred in vessel.
20	1.019	−196	10	20 ml biogas liquefied to 8 ml liquid.	Samples frozen in the vessel.
20	1.019	−196	15	20 ml biogas liquefied to 8 ml liquid.	Samples had frozen in the vessel.

b) Testing of liquefied sample

- **pH test of biomethane liquid**

Litmus paper tested the pH of liquefied biomethane sample which indicated that blue litmus paper turned red. When tested by a universal indicator displayed a value of 6.9.

- **liquefied sample flame test**

On being taken out of the cold box, the frozen liquefied sample ignited with some difficulty because of its coldness condition. Combustion occurred when its temperature normalized, which ignited producing a flame that extinguished in a duration of 4 seconds. The short lasting duration of the flame was caused by the size of the sample.

Acknowledgements

The authors acknowledge the contribution of the biogas laboratory technicians for technical assistance during biogas generation process, and to the Food Science Laboratory for analysis of the biogas.

Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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