# Development of Volumetric Methane Measurement Instrument for Laboratory Scale Anaerobic Reactors

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## ABSTRACT

In the present study, a newly developed VMMI (Volumetric Methane-Measuring Instrument) for laboratory scale anaerobic reactors is presented. The VMMI is a reliable, inexpensive, easy to construct, easy to use, corrosion resistant device that does not need maintenance, can measure a wide flow range of gas at varying pressure and temperature. As per the results of the error analysis, the accuracy of the VMMI is unilateral, i.e. -6.91%. The calibration of VMMI was investigated and a linear variation was found; hence, in situ calibration is recommended for this type of instrument. As per chromatographic analysis, it absorbs almost 100% of the carbon dioxide present in the biogas, results only the methane, and thus eliminates the need of cost intensive composition analysis of biogas through gas chromatograph.

Key Words: Volumetric, Methane Measurement, Instrument, Laboratory Scale, Anaerobic Reactor.

## 1. INTRODUCTION

he increasing cost of fossil fuels and increased pollution due to their combustion, the anaerobic digestion processes are attending an increasing interest in industry and government regarding the waste treatments in many countries worldwide. It is because of their capability to produce renewable energy, appropriate management of different types of organic wastes and recycling of important nutrients back to the soil [1-4]. In order to know the maximum methane potential, various organic materials are an aerobically degraded in the laboratory scale anaerobic reactors [5]. Anaerobic digestion of the put rescible organic material produces biogas, which is mainly a mixture of methane (55-75%) and carbon dioxide (30-45%), along with smaller quantities of the other gases including hydrogen, hydrogen sulfide, nitrogen, oxygen, and carbon monoxide [6-7].

For controlling and monitoring the laboratory scale anaerobic reactors, the pH of the substrate and volume of the biogas are most important variables. The measurement of pH is an easy task in comparison to the accurate volumetric measurement of gas [1]. The most significant features affecting the accuracy and sensitivity of volumetric measurement of biogas are the errors, generated due to the variation of temperature, pressure, vapor content, and solubility of carbon dioxide [8]. Various biogas meters have been discussed in the literature, and define that the ideal biogas meter must be precise, inexpensive, capable of automatic measurement, corrosion resistant, need slight or no maintenance, and can measure a wide range gas flow at varying pressure and temperature [9-12]. The most widely measurement systems used for the quantification of biogas for laboratory scale reactors is the volumetric gas measurement system, based on the

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liquid displacement method [13]. It usually uses water or brine as the working substance. Use of water or brine absorb some quantity of carbon dioxide and thus generates the error in the volumetric measurement of the biogas. Moreover, a higher proportion of the carbon dioxide in the biogas, dissolved readily into the substrate and results in the decrease of the pH of the substrate, thus result in the inhibition of the anaerobic digestion process [14].

In the anaerobic digestion process, the degradation of organic matter is represented in, either as the loss of volatile solids [15] or as loss of chemical oxygen demand [16]. As per mass balance analysis, the loss of volatile solids or chemical oxygen demand is converted into the biogas. Out of the main components of the biogas, methane is a combustible gas, thus higher concentration of methane represents a higher quality of biogas. After measuring the quantity of the biogas, its composition was tested in a gas analyzer, usually by gas chromatography [17-18], as it is the best analytical technique for analyzing the different components of the biogas [19]. The composition of the biogas throughout the digestion period does not remain constant, but it varies due to the unbalance of the acidogenic and methanogenic stages of the degradation of organic matter [20], thus analysis of biogas is required frequently. Moreover, the gas chromatography is the cost intensive process.

Keeping in view the aforementioned problems this research paper is aimed to present a new VMMI for measuring the methane produced from the laboratory scale anaerobic reactor. Moreover, the developed VMMI was also tested experimentally, to measure the quantity and quality of methane produced from the degradation of organic matter in a 5-liter CSTR anaerobic reactor treating buffalo dung and crop residue.

## 2. METHODOLOGY

### 2.1 Design of VMMI

The most popular method for measurement of the biogas generated through the anaerobic digestion is based on the principle of liquid displacement [10,21]. For the measurement of the low volumes of the gas, the substitute to liquid displacement is the use of manometric methods [22-23]. In this research work a new gas meter was developed, which is based on both the liquid displacement and manometric methods as shown in Fig. 1. It consists of two concentric shells made of clear acrylic tubing. The inner shell was opened by both ends, while the outer shell was closed by both ends through clear acrylic cover sheets. The top cover sheet contains two ball valves for the inlet and exit of gas. The internal diameter of the inner shell was 4.0 cm. Unlike the gas meters reported in the literature [11,12,24-25], VMMI does not contain any moving parts. Anaerobic degradation of the organic material produces biogas, which is mainly consist of methane and carbon dioxide. The percentage of these gases does not only vary with the type of organic material, but also fluctuate throughout the period of digestion. If only the quantification of the methane is required, then an alkaline solution can be used to absorb carbon dioxide [23, 26]. In order to measure the volume of only methane through the VMMI, the acidic carbon dioxide was absorbed through an alkaline solution of sodium hydroxide (NaOH) [27].

The working principle of the VMMI is based on the difference of level of alkaline solution between the inner and outer shells. When the biogas enters into VMMI, the carbon dioxide will be absorbed into the alkaline solution, while the methane will uplift the alkaline solution into the inner shell. The volume of uplifted alkaline solution is equal to the volume of methane. The volume of the methane trapped in the VMMI is to be discharged manually by opening the exit valve, as it does not contain any siphon or solenoid valve as reported in the literature [10,28]. However, it is capable to measure the volume of methane at standard temperature (273.15K) and standard pressure (101.325 kPa) by using Equation (1), which was modified from the equation states by Veiga, et. al. [29].

$$VG_{inst} = \frac{\pi}{4} d^2 \frac{(h+H_2)}{101.325} \left[ \frac{273.15}{(273.15+T)} \right]$$
$$\left[ P + \frac{\rho g(h+H_1)}{100 \times 1000} - 0.133 \times \exp\left\{ 20.386 - \frac{5132}{(273.15+T)} \right\} \right]$$
(1)

Where  $VG_{inst}$  is the volume of gas measured by VMMI at standard temperature and pressure in mL;  $VG_{drum}$  is the volume of gas measured by drum gas meter at standard temperature and pressure in mL; d is the internal diameter of the inner shell in cm; h is the height of the liquid in inner shell in cm;  $H_1$  is the height of the liquid in outer shell before discharge of the gas in cm;  $H_2$  is the height of the liquid in outer shell after discharge of the gas in cm; T is the temperature of laboratory in °C; P is the atmospheric pressure in kPa;  $\rho$  is the density of liquid (1000 kg/m<sup>3</sup>), and g is the acceleration due to gravity (9.81 m/s<sup>2</sup>). Equation (1) not only contains the adjustment of the standard temperature and volume but also the vapor pressure of water, which was based on the modified Arden Buck Equation [30].

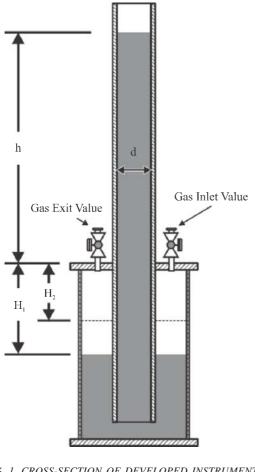


FIG. 1. CROSS-SECTION OF DEVELOPED INSTRUMENT (VMMI)

#### 2.2 Experimental Setup

The pictorial view of the experimental setup is shown in Fig. 2. It contains an air blower, VMMI, and drum type gas meter (Ritter TG-05) in series. The VMMI was filled with air through an air blower and after noting the data required for calculating the volume of gas through Equation (1), the air was discharged by first passing through the drum type gas meter. The function of the drum type gas meter in the experimental setup was the counter measurement of gas. It contains the gas pressure scale and the thermometer. As per calibration certificate of the manufacturer, the accuracy of the drum type gas meter by using the air as the calibration gas was +0.16%. The calculation of the gas by using the drum gas meter was carried out by Equation (2).

$$VG_{drum} = 1000 \times \left(R_f - R_i \left[\frac{273.15}{(273.15+T)}\right] \left[\frac{P + P_{gas}}{101.325}\right]$$
(2)

Where  $VG_{drum}$  is the volume of gas measure by drum gas meter at standard temperature and pressure in mL;  $R_i$  is the initial reading of the drum gas meter in liters;  $R_f$  is the final reading of the drum gas meter in liters; T is the temperature of laboratory in °C; P is the atmospheric pressure in kPa; and  $P_{gas}$  is the pressure of gas measured through built in pressure scale.



FIG. 2. PICTORIAL VIEW OF EXPERIMENTAL SETUP

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#### 2.3 Error Analysis

The measured values of gas as given in the Table 1 were validated by estimating the three types of errors between the gas measured by the drum gas meter ( $VG_{drum}$ ) and gas measured by VMMI ( $VG_{inst}$ ). The errors include RMSE (Root Mean Square Error), AAE (Average Absolute Error) and ABE (Average Bias Error). The RMSE was estimated by using Equation (3) [3], where n is the number of gas measurement readings. The AAE was estimated by using Equation (4), whereas the ABE was calculated by Equation (5) [31-32].

$$RMSE = \sqrt{\frac{\sum_{i=1}^{n} \left( VG_{inst,i} - G_{drum,i} \right)^2}{n}}$$
(3)

$$AAE = \frac{1}{n} \sum_{i=1}^{n} \left| \frac{VG_{drum,i} - VG_{inst,i}}{VG_{inst,i}} \right| \times 100 \tag{4}$$

$$ABE = \frac{1}{n} \sum_{i=1}^{n} \left| \frac{VG_{drum,i} - VG_{inst,i}}{VG_{inst,i}} \right| \times 100$$
(5)

#### 2.4 Gas Chromatographic Analysis

In order to know the absorption performance of the VMMI, it was connected to the laboratory scale anaerobic digester of 5-litter volume. The anaerobic reactor was treating crop residue and buffalo dung. The composition of the gas was analyzed in the gas chromatograph (GC-2010 plus SHIMADZU) with a TCD (Thermal Conductivity Detector) and using the capillary column (Rt-Q-BOND 30m, 0.53mm ID, 20 m df). A sample of 500 L was charged into the gas chromatograph by using the gas tight micro syringe (500 R-GSG, SGE analytical science). The gas chromatograph was set to inject mode by the split ratio of 18.0, whereas the injection, column and detector temperatures were adjusted at 250, 60 and 250°C, respectively. The nitrogen was used as the carrier gas with the column flow of 8.60 mL.min<sup>-1</sup>.

#### 3. **RESULTS AND DISCUSSION**

The data obtained for calculating the volumes of the gas through the VMMI, and the corresponding volumes of the gas measured through the drum type gas meter are given in the Table 1. The laboratory temperature was in the range of 28.5-30.5°C, the atmospheric pressure was in the range of 100.2-100.4 kPa,  $VG_{inst}$  was in the range of 156.4-459.4 mL, while the  $VG_{drum}$  was in the range of 142.6-430.2 mL. The relation between the  $VG_{inst}$  and  $VG_{drum}$  is shown in Fig. 3. The value of R2 reveals that both VG<sub>inst</sub> and VG<sub>drum</sub> have the strong linear relationship with each other. As per the result of the error analysis, the RMSE was 26.14 mL, while the AAE was only 6.91%. Referring to the value of ABE, i.e. -6.91% it is revealing that the VMMI overestimates the measured volume of gas by 6.91%, which is also reflected from the linear relation between VGinst and VGdrum as shown in Fig. 3 (y=0.931x). The accuracy of the Ritter Milli Gas Counter meter is bilateral, i.e.  $\pm 3\%$ [33], similarly the accuracy of the volumetric meter developed by Liu et al. is also bilateral, i.e.  $\pm 3.3\%$  [10]. On the contrary, the VMMI has only unilateral accuracy of -6.91%. The accuracy of the VMMI is lower than the accuracy of the gas meters reported in the literature, but as the VMMI generates unilateral error, thus its reading can easily be corrected by subtracting the 6.9% from the measured value.

Moreover, due to the linear variation of measured gas for this type of instrument, in-situ calibration is recommended. The main reason of error in the VMMI is reading the heights of inner and outer shells. The smallest measurable height in VMMI was 0.1 cm for both inner and outer shells. As per Walker, et. al. [21] the volumetric gas meter designs, where displaced water is weighed to determine the volume are the most versatile. However, in the weighted gas meters, the displaced water is to be refilled again, while in case of VMMI, the water is to be filled once and can be reutilized again after releasing the measured gas. The error due to the measurement of the height of the shells can be minimized, if a smaller deviation scale is used, like 0.01 cm or even less. However, for smaller deviations, the reading of the scale becomes difficult.

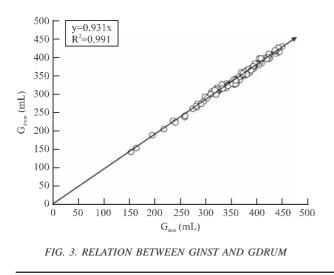
TABLE 1. GAS MEASUREMENT FROM DRUM GAS METER AND CORRESPONDING DATA FROM VMMI														MMI	
No.	h (cm)	H <sub>1</sub> (cm)	H <sub>2</sub> (cm)	T (°C)	P (kPa)	VG <sub>inst</sub> (mL)	VG <sub>drum</sub> (mL)	No.	h (cm)	H <sub>1</sub> (cm)	H <sub>2</sub> (cm)	T (°C)	P (kPa)	VG <sub>inst</sub> (mL)	VG <sub>drum</sub> (mL)
1.	34.6	8.5	2.9	30.0	100.35	418.62	384.80	51.	30.6	8.0	2.9	28.5	100.25	375.08	343.31
2.	37.4	8.9	2.9	30.0	100.35	453.10	416.94	52.	28.5	7.8	2.9	28.5	100.25	350.77	322.69
3.	36.0	8.7	2.9	30.0	100.22	434.37	397.68	53.	27.3	7.7	2.9	28.5	100.25	336.93	316.41
4.	37.4	8.9	2.9	30.0	100.35	451.29	417.84	54.	25.8	7.5	2.9	28.5	100.25	319.66	302.07
5.	33.2	8.4	2.9	30.0	100.32	402.28	370.41	55.	31.7	8.2	2.9	28.5	100.25	387.89	364.82
6.	38.1	9.0	2.9	30.0	100.32	459.36	430.21	56.	30.3	8.0	2.9	28.5	100.25	371.61	345.10
7.	29.4	7.9	2.9	30.0	100.32	358.41	332.03	57.	25.8	7.5	2.9	28.5	100.25	319.66	302.97
8.	35.8	8.7	2.9	30.0	100.32	432.48	409.68	58.	22.3	7.2	2.9	28.5	100.25	279.62	260.84
9.	28.4	7.8	2.9	30.0	100.32	346.94	318.64	59.	31.5	8.2	2.9	28.5	100.25	385.57	351.37
10.	31.9	8.2	2.9	30.0	100.32	387.22	353.45	60.	26.6	7.5	2.9	28.5	100.25	328.83	317.31
11.	33.8	8.5	2.9	30.0	100.2	408.76	382.44	61.	31.9	8.3	2.9	28.5	100.25	390.25	364.82
12.	37.3	8.9	2.9	30.0	100.32	450.00	413.25	62.	30.5	8.0	2.9	28.5	100.25	373.92	338.82
13.	35.0	8.6	2.9	29.5	100.32	424.36	398.73	63.	15.2	6.1	2.9	28.5	100.25	199.21	188.24
14.	27.0	7.6	2.9	29.5	100.32	331.84	314.70	64.	25.5	7.5	2.9	28.5	100.25	316.23	300.28
15.	24.4	7.3	2.9 2.9	29.5 30.0	100.32	302.11	281.62	65.	36.8	8.8 8.5	2.9 2.9	28.5	100.25	447.56	419.50
16. 17.	36.9 35.4	8.8	2.9	29.5	100.32 100.32	445.30 429.01	409.68 396.05	66. 67.	33.9 30.4	8.5 8.0	2.9	28.5	100.25 100.25	413.57 372.77	397.98 346.89
17.	29.4	8.6 7.9	2.9	29.5	100.32	359.43	334.36	68.	29.7	7.9	2.9	28.5 28.5	100.25	364.64	340.89
18.	31.2	8.1	2.9	29.5	100.32	380.21	358.50	69.	29.7	7.9	2.9	28.5	100.25	363.49	338.82
20.	35.6	8.7	2.9	29.5	100.32	431.38	400.52	70.	33.8	8.5	2.9	28.5	100.25	412.40	3380.06
20.	27.0	7.6	2.9	29.5	100.32	331.60	309.12	70.	33.5	8.3 8.4	2.9	28.5	100.25	408.87	383.64
21.	32.3	8.3	2.9	29.0	100.23	393.68	357.84	72.	31.8	8.2	2.9	28.5	100.25	389.05	358.54
23.	36.6	8.8	2.9	29.0	100.22	443.85	415.10	72.	33.2	8.4	2.9	28.5	100.25	405.38	383.64
23.	21.0	6.9	2.9	29.0	100.22	263.91	237.92	74.	32.9	8.3	2.9	28.5	100.25	401.86	375.57
25.	24.6	7.3	2.9	29.0	100.2	304.87	292.48	75.	29.5	7.9	2.9	28.5	100.25	362.33	339.72
26.	27.2	7.7	2.9	29.0	100.2	334.69	313.05	76.	31.6	8.2	2.9	28.5	100.25	386.73	363.03
27.	28.2	7.8	2.9	29.0	100.2	346.18	316.63	77.	35.2	8.6	2.9	28.5	100.25	428.76	396.19
28.	30.0	8.0	2.9	29.0	100.2	366.94	328.26	78.	29.3	7.9	2.9	28.5	100.25	360.03	328.07
29.	33.5	8.4	2.9	28.5	100.2	408.67	383.45	79.	31.0	8.1	2.9	28.5	100.25	379.74	353.17
30.	37.2	8.9	2.9	28.5	100.2	452.06	426.45	80.	26.4	7.5	2.9	28.5	100.25	326.54	299.38
31.	33.8	8.5	2.9	29.5	100.2	409.91	383.08	81.	30.0	8.0	2.9	28.5	100.25	368.14	333.45
32.	31.0	8.1	2.9	29.5	100.2	377.45	348.25	82.	27.4	7.7	2.9	28.5	100.25	338.08	327.17
33.	25.8	7.5	2.9	29.5	100.2	317.72	307.18	83.	37.2	8.9	2.9	28.5	100.25	452.29	425.77
34.	28.5	7.8	2.9	29.5	100.2	348.65	329.50	84.	23.8	7.2	2.9	28.5	100.25	296.71	274.29
35.	19.0	6.8	2.9	29.5	100.2	240.64	227.70	85.	24.9	7.4	2.9	28.5	100.25	309.33	289.52
36.	26.8	7.6	2.9	29.5	100.2	329.15	315.21	86.	32.1	8.3	2.9	28.5	100.2	392.37	368.22
37.	26.7	7.6	2.9	29.5	100.2	328.01	303.60	87.	26.4	7.5	2.9	28.5	100.2	326.37	313.57
38.	29.9	8.0	2.9	30.0	100.2	363.74	327.17	88.	31.7	8.2	2.9	28.5	100.2	387.70	354.78
39.	31.1	8.1	2.9	30.0	100.2	377.53	359.27	89.	29.8	7.9	2.9	28.5	100.2	365.61	327.90
40.	11.5	5.8	2.9	30.0	100.2	156.44	142.64	90.	34.2	8.5	2.9	28.5	100.2	416.85	397.79
41.	17.4	6.5	2.9	30.0	100.2	222.00	205.04	91.	31.1	8.1	2.9	28.5	100.2	380.71	356.57
42.	23.3	7.2	2.9	30.0	100.2	288.42	263.88	92.	19.3	6.8	2.9	28.5	100.2	245.38	223.98
43.	27.0	7.6	2.9	30.5	100.2	329.55	311.50	93.	23.0	7.2	2.9	28.5	100.2	287.45	267.88
44.	32.9	8.3	2.9	30.5	100.2	397.17	371.13	94.	29.8	8.0	2.9	28.5	100.2	365.65	336.86
45.	28.9	7.8	2.9	30.5	100.2	351.23	324.85	95.	32.5	8.3	2.9	28.5	100.2	397.01	364.64
46.	30.2	8.0	2.9	28.5	100.25	370.45	336.13	96. 07	12.4	5.8	2.9	28.5	100.2	167.78	154.10
47. 48.	23.2 37.3	7.2 8.9	2.9 2.9	28.5 28.5	100.25 100.25	289.87 453.46	274.29 415.91	97. 98.	21.0 27.6	6.9 7.7	2.9 2.9	28.5 28.5	100.2 100.2	264.64 340.21	241.90 316.26
48.	33.1	8.4	2.9	28.5	100.25	404.22	375.57	98. 99.	26.2	7.5	2.9	28.5	100.2	324.08	310.20
49. 50.	34.0	8.5	2.9	28.5	100.25	414.73	387.23	100.	36.6	8.8	2.9	28.5	100.2	444.99	419.29
50.	54.0	0.5	2.9	20.5	100.23	-TI4./J	501.25	100.	50.0	0.0	2.9	20.3	100.2	777,77	T17.47

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The main disadvantage of the manometric method is that deviation in the pressure of the headspace of the anaerobic reactor, which increases the absorption of the carbon dioxide in the liquid phase of the reactor and thus alter the pH and affect the experimental conditions [13]. This shortcoming was overcome in the VMMI by using the alkaline solution of NaOH, which absorb the carbon dioxide and thus eliminates the variation of pH due to the absorption of the carbon dioxide. Additionally, the composition of the biogas was also analyzed in the gas chromatograph. As per the results, the gas sample contains about 98-99% of methane, 1-2% of hydrogen sulphide and 0% of carbon dioxide. Usually, in the composition of the biogas, only methane and carbon dioxide are considered, and smaller quantities of other gases are neglected [34-35]. Thus, the developed VMMI eliminates the need of biogas analyze for its composition, and consequently does not require cost intensive gas chromatograph.

The VMMI is made from the acrylic plastic, which not only has high clarity but also has a very good resistance to chemicals, heat, impact loads and high pressures. Further, the acrylic plastic is solvent bondable, and highly rigid [36]. These characteristics of acrylic plastic are also reflected in VMMI, and thus make it suitable for biogas measurement. Besides, the VMMI does not contain moving parts, which makes it virtually maintenance free. The material cost of the gas metering system for measurement



of low gas rates from laboratory reactors as given by the Angelidaki, et. al. [9] was US \$100. The cost of the drum type gas meter use to calibrate the VMMI in the present study (Ritter TG-05) is US\$5000, and for the small size wet gas meter (Ritter MGC) is US\$2000. On the contrary, the cost of the VMMI is only US\$20. Thus VMMI is inexpensive volumetric gas measuring meter.

The VMMI is the versatile methane measuring instrument that can not only be used at varying temperature and pressure situations, but also adjust the gas measuring errors due to the absorption of the carbon dioxide [13]. Additionally, during calculation of the gas, vapor pressure of the water can also easily be compensated. The VMMI can be scaled up and scaled down to measure the gas flow rates ranging from 10-4000 mL/day or even more. Another exciting option is the connecting two VMMI in the series. The first VMMI will be large and contains a non-absorbent material, while the second will be relatively smaller and contain absorbent material. This configuration will not only measure both biogas and methane, but also be useful to understand the degradation of the organic material. Moreover, VMMI can be upgraded to measure methane and carbon dioxide automatically using digital data storing and processing.

#### 4. CONCLUSIONS

In this piece of work, a newly developed volumetric methane measuring instrument for laboratory scale anaerobic reactor is presented, which is based on the combined water displacement and manometric methods. From the results, it is concluded that newly volumetric methane measuring instrument is a reliable, inexpensive, easy to construct, easy to use, corrosion resistant device that does not need maintenance, can measure a wide range of gas flow at varying pressure and temperature and adequately accurate to measure the volume of methane generated from the laboratory scale anaerobic reactors. It is capable to calculate the methane at standard temperature and pressure and almost eliminates the need of cost intensive composition analysis of biogas through gas chromatograph or in any other gas analyzer.



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