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ENERGY GENERATION BY ANAEROBIC DIGESTION: RESISTIVE METHOD FOR BIOGAS MONITORING – PART II

GERAÇÃO DE ENERGIA POR DIGESTÃO ANAERÓBIA: MÉTODO RESISTIVO PARA MONITORAMENTO DO BIOGÁS – PARTE II

GENERACIÓNDE ENERGÍA POR DIGESTIÓN ANAEROBIA: MÉTODO RESISTIVO PARA MONITOREO DE BIOGÁS – PARTE II

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ABSTRACT

Data collection in laboratory experiments through automation can help with the operation and optimization of the process, and as a result, overcome any potential failures in the system, ensuring safety and complete control. In this sense, this work aimed to evaluate the automatic system developed for data collection during the anaerobic digestion process. Anaerobic digesters were used, supplied with bovine manure (BM), sewage sludge (SS), and mineral water (MW) under anaerobic digestion process in the proportions of 100:0 BM:SS, 0:100 BM:SS, 50:50 BM:SS, and 50:50 BM:MW. For monitoring the biogas concentration and temperature, the Biogas Monitoring Module (BMM) and Parameter Monitoring Module (PMM) were developed, which use the MQ-4 and DHT11 sensors, respectively. The results obtained with BMM and PMM were compared to those obtained by the use of Alfakit® and gas chromatography. It was concluded that BMM and PMM were effective in monitoring temperature and methane, but there is a need for adjustments in the applied methodology.

Keywords: automation, MQ-4 sensor, DHT11 sensor.

RESUMO

A coleta de dados em experimentos laboratoriais por meio da automação pode auxiliar no funcionamento e otimização do processo, e como consequência contornar eventuais falhas no sistema garantindo a segurança e total controle. Neste sentido, objetivou-se neste trabalho avaliar o sistema de automático desenvolvido para coleta de dados da produção durante processo de digestão anaeróbia. Utilizou-se biodigestores anaeróbicos abastecidos com dejetos de bovino (DB), lodo de esgoto (LE) e água mineral (A) sob processo de digestão anaeróbica nas proporções de 100:0 DB:LE, 0:100 DB:LE, 50:50 DB:LE e 50:50 DB:A. Para o monitoramento da concentração e temperatura biogás desenvolveu-se o Módulo de Monitoramento do Biogás (MMB) e o Módulo de Monitoramento de Parâmetro (MMP) que utilizam os sensores MQ-4 e DHT11, respectivamente. Os resultados obtidos com MMB e MMP foram comparados aos obtidos pelo uso do Alfakit® e cromatografia gasosa. Concluiu-se que o MMB e MMP foram eficazes no monitoramento da temperatura e metano, porém há necessidade de ajustes na metodologia aplicada.

Palavras-chave: *automação, sensor MQ-4, sensor DTH11*

RESUMEN

La recolección de datos en experimentos de laboratorio a través de la automatización puede ayudar en la operación y optimización del proceso, y como consecuencia, sortear posibles fallas en el sistema, garantizando seguridad y control total. En este sentido, el objetivo de este trabajo fue evaluar el sistema automático desarrollado para recolectar datos de producción durante el proceso de digestión anaeróbica. Los digestores anaeróbicos fueron alimentados con estiércol bovino (EB), lodos de depuradora (LD) y agua mineral (A) bajo proceso de digestión anaeróbica en las proporciones de 100:0 EB:LD, 0:100 EB:LD, 50:50 EB:LD y 50:50 EB:A. Para el monitoreo de la concentración y temperatura del biogás, se desarrollaron el Módulo de Monitoreo de Biogás (MMB) y el Módulo de Monitoreo de Parámetros (MMP), que utilizan los sensores MQ-4 y DHT11, respectivamente. Los resultados obtenidos con MMB y MMP se compararon con los obtenidos con Alfakit® y cromatografía de gases. Se concluyó que MMB y MMP fueron efectivos para monitorear la temperatura y el metano, pero es necesario realizar ajustes en la metodología aplicada.

Descriptores: *automatización, sensor MQ-4, sensor DHT11*

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INTRODUCTION

Biogas is composed of methane (CH_4), carbon dioxide (CO_2) and small amounts of other gases such as hydrogen sulfide (H_2S). Therefore, during the anaerobic digestion (AD) process, continuous monitoring of biogas components is necessary.

However, there are still many operational difficulties due to the lack of technologies in different stages of the production line. The optimization of the continuous collection of anaerobic digestion parameters through instrumental automation can assist in the operation of the process due to the possibility of automatic and continuous monitoring, ensuring safety and total process control.

Currently, there are analytical methods and equipment that can be used in detecting biogas components. However, they have limitations such as the high cost of equipment and lack of precision in the values detected by analytical methods. In addition, analytical methods require constant material acquisition, increasing the cost of analysis when a large number of samples are required. The lack of precision in the values detected by analytical methods is an important factor to be considered when working with scientific research.

Gas chromatograph is the most commonly used equipment in the laboratory for measuring the concentration of CH_4 and other components present in biogas (JAFARI et al., 2017; KRAUSE et al., 2018; YANG et al., 2019). However, the time-consuming measurement time between repetitions due to the cleaning and saturation process of the present gases makes the analysis slow, generating delays in data collection process and possible sample loss.

In this sense, the popularization of electronic prototyping boards has facilitated the development of automatic data collection systems for evaluation and monitoring of scientific experiments. The automation system through the Arduino Uno® electronic prototyping platform can be considered a low-cost technique with free software for programming, efficient and easy to use and handle. The Arduino Uno® platform can be used in conjunction with sensors, allowing the development of specific applications, such as automatic monitoring during the drying process of agricultural products (PAES et al., 2022) and anaerobic digestion (YANG et al., 2019; FAKRA et al., 2020).

Thus, aiming at the adoption of sustainable production processes associated with automation, the objective was to develop, implement and evaluate the automatic data acquisition system to continuously monitor the methane detection and temperature parameters in the biogas generated during the process.

MATERIALS AND METHODS

The experiment was conducted at the Multi-User Research Laboratory of the Rural Renewable and Alternative Energy Group (LabGERAR) of the Rural Federal University of Rio de Janeiro (UFRRJ), located in Seropédica - RJ, Brazil, with geographical coordinates of 22º 45’ 33” S and 43º 41’ 51” W. The climate of the region is classified as Aw according to the Köppen classification system, with an average annual temperature of 24.5 ºC.

The sewage sludge used in the experiment was obtained from the Palatinato Sewage Treatment Plant, owned by Águas do Imperados of the Águas do Brasil Group, located in the municipality of Petrópolis, Rio de Janeiro state. The bovine manure was collected from the Dairy Cattle Farm at UFRRJ.

The experiment was based on the anaerobic mono-digestion (MoAD) ratios of 100:0 and 0:100 for bovine manure:sewage sludge (BM:SS), and a ratio of 50:50 for bovine manure:mineral water (BM:MW) for anaerobic co-digestion (CoAD) experiments. The experiments were performed in triplicate.

The anaerobic biodigester used in the experiment was based on the Indian model and consisted of a water-seal chamber, fermentation chamber, gasometer, and a movable U-tube manometer with water manometric fluid, as described by Matos et al. (2017).

The biogas generated during the anaerobic digestion of the substrates under MoAD and CoAD was monitored using the Conventional Data Acquisition System (CDAS) and Automatic Data Acquisition System (ADAS), which includes the Parameter Monitoring Module (PMM) and Biogas Monitoring Module (BMM). After the measurement was completed in the anaerobic biodigester using CDAS, ADAS was initiated.

The CDAS was used to monitor and record the data on temperature and methane in the biogas produced during anaerobic digestion. The biogas and ambient temperature were obtained using a thermocouple connected to a millivoltmeter with an accuracy of ±0.1 °C.

The methane was quantified using the Biogas Analysis Kit® Alfakit and gas chromatography. In the first step, the carbon dioxide (%) was determined using the Orsat volumetric method. Then, the value was subtracted from 100% to obtain the methane content. For this purpose, 20 mL of biogas from each anaerobic biodigester was collected using a syringe. The biogas was then transferred to another syringe containing a solution called “Pre-Treatment 2.” The biogas and solution were agitated for two minutes and transferred to another graduated

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glass syringe to perform the carbon dioxide reading. Methane quantification was performed using gas chromatography in the Multi-User Unit in Geochemistry of Gases, Water, and Sediments at the Federal Fluminense University (GAS-UFF). A calibrated Varian Chrompack CP-3800 gas chromatograph was used, with methane standard from White Martins Gases Industriais Ltda. Nitrogen was used as the carrier gas and makeup gas. The column used was the RestekRt-Q-PLOT, with an internal diameter of 0.53 mm and a length of 15 m. The detector used was the flame ionization detector (FID), powered by hydrogen and synthetic air.

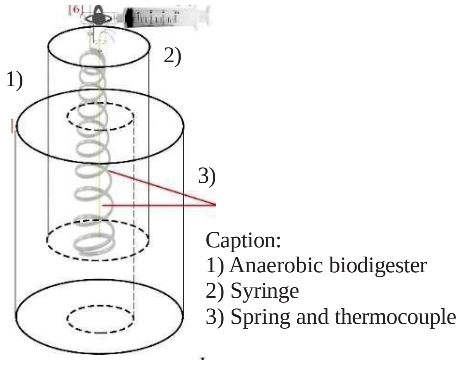
In ADAS, the Arduino Uno® electronic prototyping platform, electronic components (sensors), a 400-point auxiliary protoboard, and a distinct computer were used. The Arduino Uno® platform was composed of a programmable ATmega328P microcontroller with fourteen digital input/output ports, six of which were used as Pulse Width Modulation (PWM) outputs and six as analog outputs (ARDUINO INC, 2021).

The digital thermal sensor DHT11 measures temperature (T) in the range of 0 to 50 ºC and air humidity (RH) between 20 and 90% (Datasheet DHT11, 2022). The MQ-4 sensor, made up of sensitive tin dioxide material (SnO_2), measures CH_4 concentration in the range of 200 to 10,000 ppm (Datasheet MQ-4, 2022). The sensors have adjustable sensitivity via a potentiometer with digital and analog output, power supply, and Graduated Neutral Density Filter (GND). These electronic components were purchased with connection pins adapted to a module to facilitate connections with the protoboard. The module terminal of each sensor connected to the protoboard communicated with the Arduino Uno® platform through a jumper. The DHT11 sensor, through the GND port, communicated with the Arduino Uno® platform at the digital output port 2 and power supply port 3.3 V to supply power. The MQ-4 sensor communicated on the analog pin (A0), 5.0 V power supply port, and neutral connection on the GND pin. As methane concentrations were measured, analog ports were used, which detect conductivity variations reflected in the signal sent to the Arduino Uno® platform (ARDUINO INC, 2021).

To ensure the proper functioning of ADAS, it was necessary to adapt the available codes and libraries on Github (2008) and Geekstips (2016) for the DHT11 and MQ-4 sensors, respectively. This change was possible due to the free hardware characteristic of the Arduino Uno® platform and its libraries available for free on the internet.

With the proper programming for continuous measurement, the PMM for biogas temperature (DHT11) and BMM for methane concentration (MQ-4) were developed. For this purpose, 3.0 mL of biogas sample produced in the anaerobic biodigesters (Figure 1) were collected and injected into the experimental apparatus that constitutes the PMM and BMM.

Figure 1 - Biogas sample collection in the anaerobic biodigester



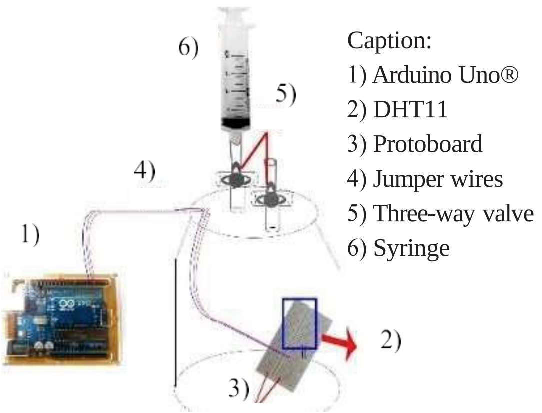
Source: Rural Renewable and Alternative Energy Group (GERAR)

The value of the 3.0 mL sample was defined to be used in collecting biogas samples injected into the gas chromatograph. At the base of the syringe, 5.0 mL of steel mesh was placed to prevent sensor corrosion by the biogas. The steel mesh is used to purify the biogas regarding H2S.

The PMM used the experimental apparatus called the “Pot Method”, consisting of a 0.3 L glass jar with a metal lid, Arduino Uno® platform, DHT11 attached to the protoboard, and a computer (Figure 2).

On the metal lid, two three-way valves and a hole for the passage of the jumper that connects the protoboard/sensor assembly inside the pot with the Arduino Uno® platform were installed. To seal the pot, a 6.2 cm diameter o-ring was used inside the lid, transparent acetic silicone adhesive and instant epoxy resin adhesive on the connections (Figure 2).

Figure 2 - Experimental apparatus for the Parameter Monitoring Module (PMM)



Source: GERAR

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The DHT11 sensor started its operation 10 s before the injection of biogas to read the ambient air inside the glass pot. Then, the biogas was injected to read the temperature every second, totaling 300 s.

The BMM was composed of three experimental apparatuses called Air Method (AM), Capsule Method (CM), and Pot Method (PM). Each experimental apparatus was individually composed of an Arduino Uno® platform, MQ-4, and three 10 ohm resistors coupled to the protoboard and a computer.

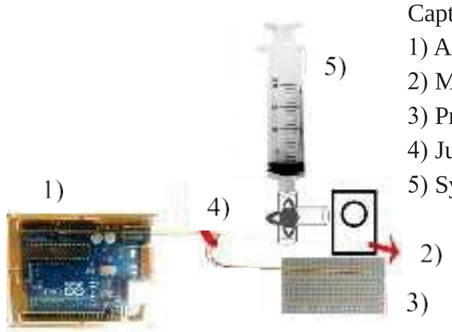
Initially, for analysis by BMM, the MQ-4/protoboard assembly was connected to the Arduino Uno® platform only by the power supply and GND port for 24 h. This procedure was performed for heating, expansion, and stabilization of the internal resistance of the sensor (R0), as recommended by the manufacturer.

After this stage, R0 was read in the ambient air. This procedure was employed to calibrate the sensor for use in the adapted code before each methane concentration reading. This process is essential because it ensures the stability of R0 in the sensor and, consequently, the methane concentration values. Therefore, after the calibration stage, data collection by MQ-4 was initiated.

The methodology for methane quantification by BMM followed the same as that adopted in PMM, in which the Arduino Uno® platform together with the MQ-4 sensor started its operation 10 s before the injection of biogas to read the ambient air.

In the AM, the biogas was injected directly into the MQ-4 sensor for 30 s, with data collection by the system every second (Figure 3).

Figure 3 - Biogas Monitoring Module by Air Method



Caption:
1) Arduino Uno®
2) MQ 4
3) Protoboard
4) Jumper wires
5) Syringe

Source: GERAR

In the CM, the MQ-4 sensor was fitted into a rigid PVC hose with dimensions of 2.2 cm in height and 2.4 cm in diameter to prevent air leakage from the

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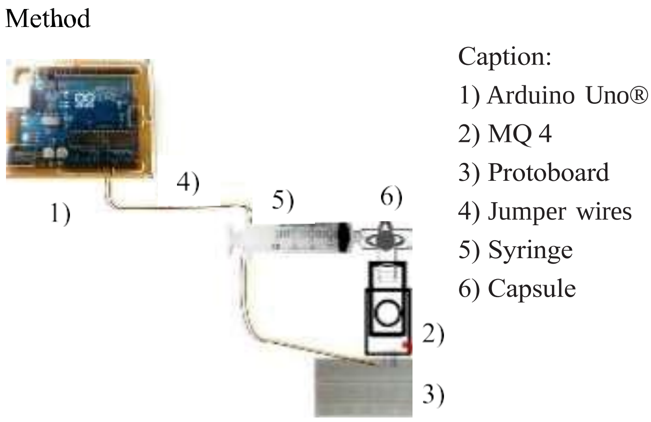
lower base. The upper base of the capsule was sealed with a plastic cap with a diameter of 2.7 cm and a butyl rubber septum for perfusion. A three-way valve was installed in the middle of the rubber septum for biogas injection (Figure 4).

To ensure complete sealing of the experimental apparatus, transparent acetic adhesive silicone was used on the lower and upper base of the capsule, as well as on the connection between the three-way valve and the butyl rubber septum (Figure 4).

Figure 4 - Biogas Monitoring Module by Capsule



Source: GERAR

The PM followed the apparatus developed for the PMM. In the CM and PM, a metal structure was used to hold the syringe in place, avoiding sudden movements during injection of the biogas.

After data collection, the anaerobic biodigester was emptied, and the experimental apparatuses of the PMM and BMM were opened. Data collection by CDAS and ADAS was performed once a week over the period of anaerobic digestion, in triplicate for each ratio under study.

To evaluate the results at different substrate ratios, the mean and standard deviation (SD) of the data obtained by ADAS for each collection day were calculated.

RESULTS AND DISCUSSION

The original code developed by GITHUB (2008) can be used by different sensors in the DHT line to measure temperature and relative humidity. Thus,

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the ‘comment’ command (//) was placed in the original code, which prevented the measurement of relative humidity. Only the measurement of temperature was obtained through the DHT11. The digital port 2 was defined on the Arduino Uno® platform for data reading.

Low SD was observed in all ratios studied over 11 weeks of anaerobic digestion, indicating homogeneity in the collected data.

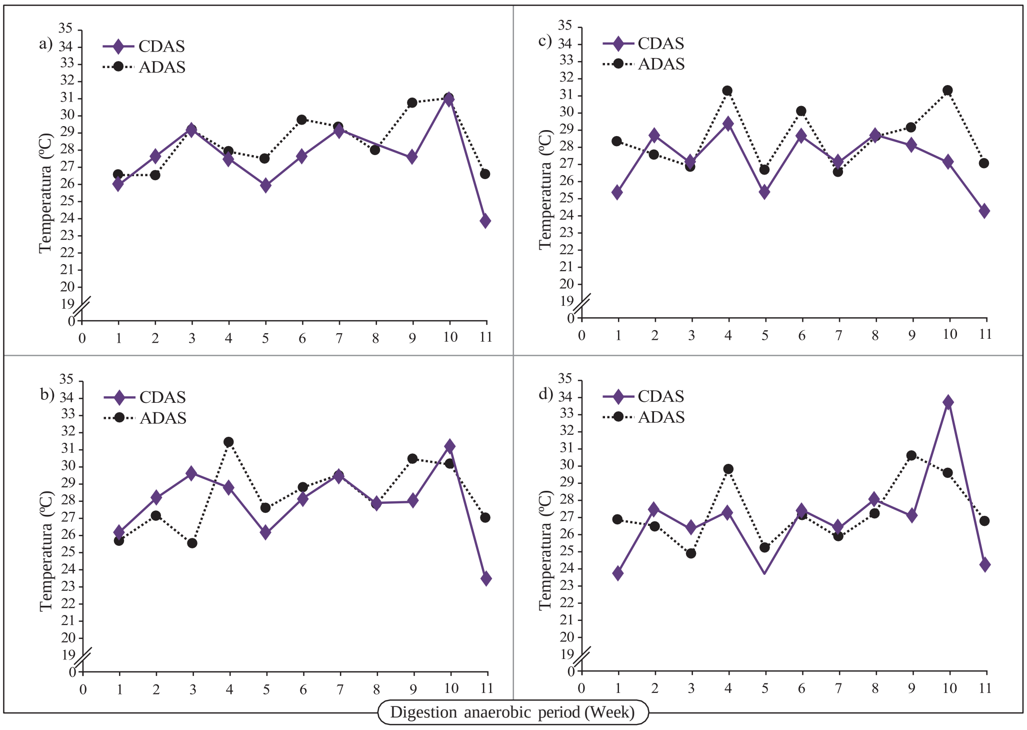
Table 1 - Mean and Standard Deviation (SD) of ADAS readings for 11 weeks of anaerobic digestion (AD)

Proportion	100:0	BM:SS	0:100	BM:SS	50:50	BM:SS	50:50	BM:SS
AD period	Mean	SD	Mean	SD	Mean	SD	Mean	SD
1	26.5	0.06	25.6	0.13	28.4	0.08	26.7	0.12
2	26.4	0.10	27.0	0.08	27.6	0.05	26.3	0.08
3	29.1	0.04	25.4	0.12	26.9	0.49	24.7	0.22
4	27.8	0.12	31.3	0.05	31.3	0.08	29.7	0.06
5	27.4	0.04	27.5	0.05	26.7	0.06	25.0	0.07
6	29.7	0.08	28.7	0.06	30.1	0.13	27.0	0.08
7	29.3	0.18	29.4	0.09	26.6	0.04	25.7	0.05
8	27.9	0.30	27.7	0.09	28.6	0.23	27.0	0.16
9	30.7	0.07	30.4	0.07	29.1	0.18	30.5	0.19
10	31.0	0.08	30.1	0.10	31.3	0.05	29.5	0.05
11	26.5	0.05	26.9	0.04	27.0	0.36	26.5	0.05

It can be observed that the ADAS, aimed at obtaining temperature data, presented values similar to those collected by the CDAS in all proportions studied over 11 weeks of anaerobic digestion (Figure 5). Paes et al. (2022) reported that the use of Arduino, a low-cost microcontroller, for automation of monitoring activities allowed for the estimation of air drying temperature through DHT22, with good correlation between automatic and conventional readings.

For the adaptation of the code developed by Geekstips (2016), the variables sensor_volt (sensor voltage), RS_air (air resistance), and sensorValue (analog variable of resistance reading) were defined to obtain R0, and gas_sensor (sensor voltage) and RS_gas (gas resistance) to obtain the methane concentration reading in biogas. The variable sensor_value received the gas_sensor variable readings and was converted to voltage, resulting in the sensor_volt variable.

Figure 5 - Temperature values collected by ADAS and CDAS for a) 100:0, b) 0:100, c) 50:50 BM:SS and d) 50:50 BM:MW



Then, this was used to calculate the biogas resistance, named RS_gas. The ratio variable received the final converted value of methane concentrations. In addition, values for the m and b variables were defined, which were used in the conversion calculation to ppm.

It can be observed that in the three repetitions of each proportion studied, methane detection occurred immediately after injection of biogas directly into the MQ-4 sensor (Figure 6). The methane concentration peak in all proportions occurred between 10 - 15 s of reading, except for 50:50 BM:MW.

In the BM and MW, it was identified that the three repetitions did not present the same biogas concentration profile, as verified in the others. This result is probably related to instability in data collection by the sensor when used in short intervals between one analysis and another.

As observed in the AM (Figure 6), there was immediate detection of methane on the MQ-4 sensor after the injection of biogas into the capsule (Figure 7). However, the CM did not show a peak concentration of methane. There was a steady increase in the methane content reading until reaching a constant concentration (Figure 7).

This fact may indicate saturation by CH_4 in the environment inside the capsule. Considering that there was no decrease in methane concentration after 60 s of

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reading, it can be inferred that the capsule seal was efficient. It should also be noted that the data collected among the three repetitions were not homogeneous for the same proportion, which may characterize sensor instability when used in the CM.

Figure 6 - Profile of data collection by Air Method for a) 100:0, b) 0:100, c) 50:50 BM:SS and d) 50:50 BM:MW

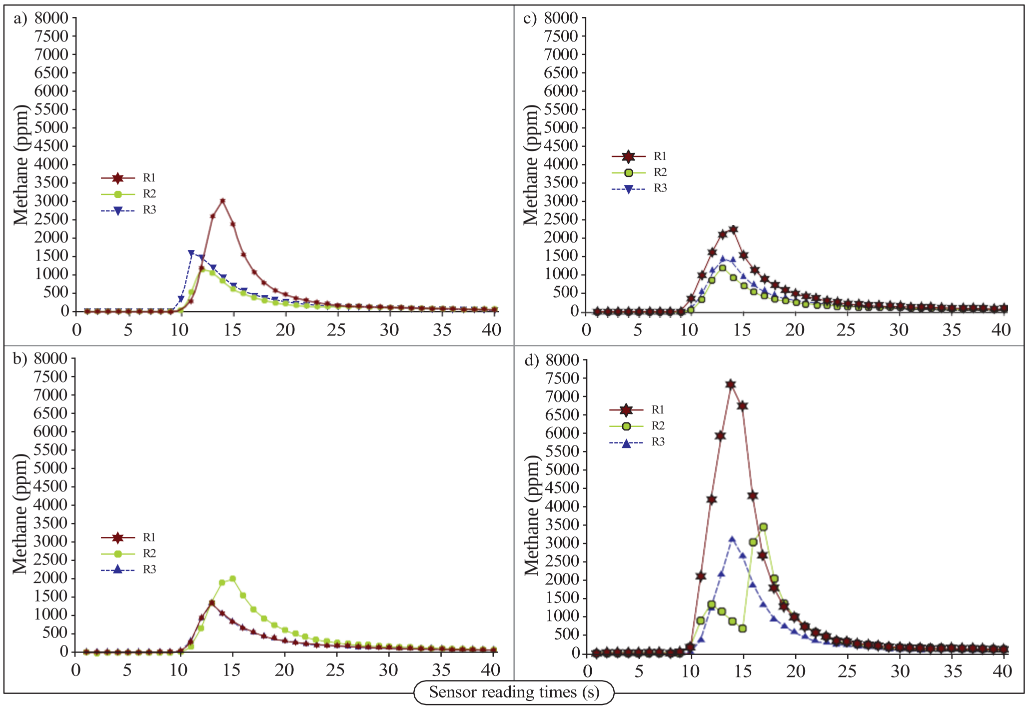
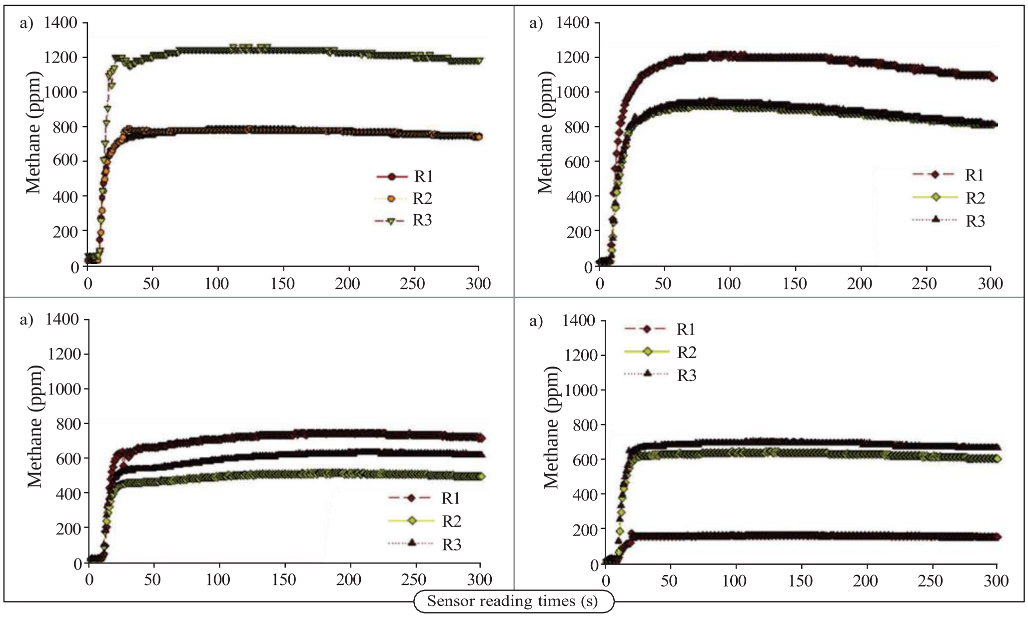


Figure 7 - Profile of data collection by Capsule Method for a) 100:0, b) 0:100, c) 50:50 BM:SS e d) 50:50 BM:MW



The PM presented the lowest response values in the methane concentration curve by the BMM (Figure 8) when compared to the other methods. This result may be related to the high amount of components in the air in the same environment, which can compromise the data reading by the sensor. In addition, the distance traveled by the biogas to the sensor in this method is greater when compared to other methods.

According to Fakra et al. (2020), methane, being lighter than air, supposedly does not adequately cover the sensor, so it is necessary to reduce the space between biogas injection and the sensor.

The AM provided the highest values by the BMM. However, as it is in a free environment, there is no control over the diluted volume. Therefore, there is a limitation on application due to the physical characteristics of biogas, in which it dissipates soon after the peak, with a low reading duration time.

In Figure 7, it is possible to observe that the CM reached higher values when compared to the PM (Figure 8), but lower than the AM (Figure 6). Like the PM, the CM was able to keep the biogas inside its environment, but its volume deviates from the dilution proposal of Yang et al., 2019 and Fakra et al. (2020). However, the collected results were consistent with the sensor range (200 - 10000 ppm), even without using the formulas proposed by the authors.

Figure 8 - Profile of data collection by Pot Method for a) 100:0, b) 0:100, c) 50:50 BM:SS e d) 50:50 BM:MW

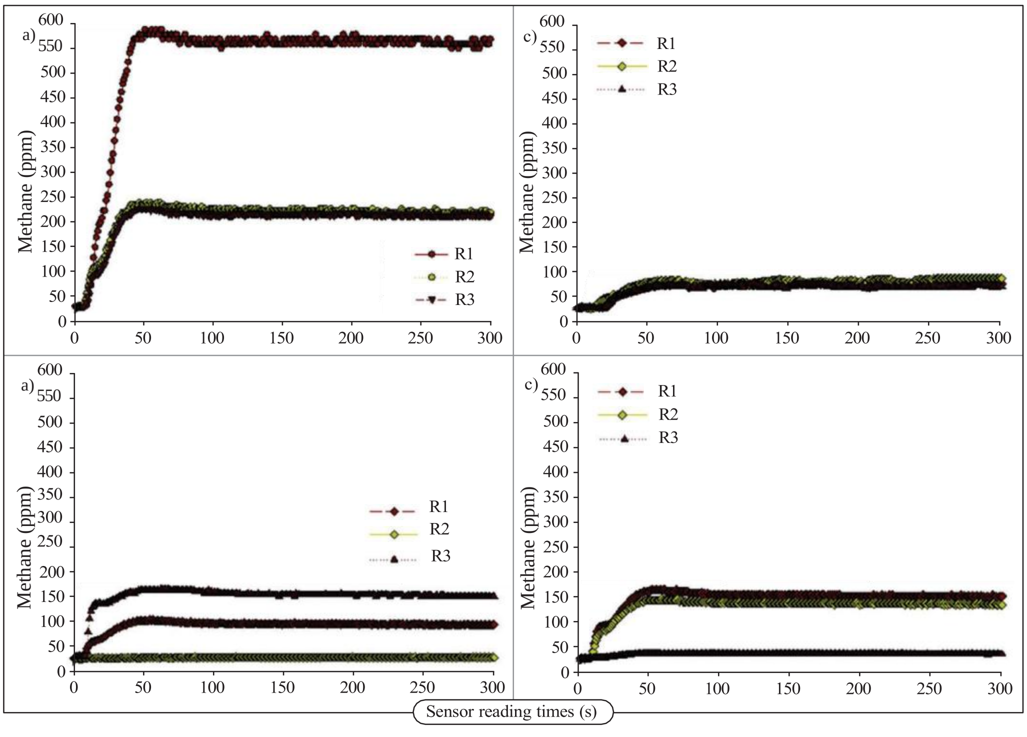


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Table 2 shows the results of the values obtained by the sensors and their respective averages and standard deviation (SD) using the BMM by the three methods (AM, CM, PM). When comparing these concentrations with the data collection profile observed by the ADAS, the same reading behavior of the MQ-4 sensor was identified, in which the methane concentration decreased with each repetition.

Table 2 - Quantification of methane concentration by sensors using the BMM

Proportion	Repetition	Values obtained by the sensors (ppm)		
		Air Method	Capsule Method	Pot Method
100:0 BM:SS	R1	17,205.6	227,228.36	161,283.56
	R2	8,576.80	227,626.22	65,216.71
	R3	11,195.56	361,175.89	62,837.27
	Mean	12,326.11	272,176.82	96,445.85
	SD	4,424.27	77.508.72	56.163.71
0:100 BM:SS	R1	15,598.75	211,324.60	29,298.12
	R2	15,598.75	147,629.33	9,589.76
	R3	10,662.48	178,382.89	46,958.69
	Mean	13,953.33	179,112.27	28,615.52
	SD	2,849.96	31,853.90	18,693.81
50:50 BM:SS	R1	16,967.31	342,190.71	21,689.97
	R2	8,759.86	258,065.60	24,033.88
	R3	11,091.08	261,903.24	20,974.82
	Mean	12,272.75	287,386.52	22,232.89
	SD	4,229.40	47,500.59	1,600.17
50:50 BM:MW	R1	42,613.91	47,998.67	45,616.04
	R2	20,731.08	186,517.91	40,950.85
	R3	18,639.77	203,626.34	11,496.09
	Mean	27,328.25	146,047.64	32,687.66
	SD	13,279.00	85,342.69	18,500.08

The result may be related to the immediate use of the values obtained by the sensors, without prior use of the formula recommended by the literature. According to Yang et al. (2019) and Fakra et al. (2020), to obtain the methane concentration values obtained by MQ-4, a methane dilution formula was used for the Capsule Method and Pot Method.

In addition, according to Fakra et al. (2020), the values obtained by the MQ-4 sensor in the Air Method and Capsule Method were not in the sensor's reading range. However, it was verified that the values obtained by the Air Method and Capsule Method in the present study were within the sensor's reading range. For the 100:0 and 50:50 BM:SS ratios, it was observed that the values were very similar, needing only to convert them all to the same unit (Table 3).

Table 3 - Results of methane concentration quantification by CDAS using Alfakit and Gas Chromatography

Proportion	Alfakit (%)	Alfakit (ppm)	Chromatography (ppm)
100:0 BM:SS	70	700,000	686,080.80
0:100 BM:SS	85	850,000	367,505.00
50:50 BM:SS	45	450,000	430,188.00
50:50 BM:MW	65	650,000	270.56

Gas chromatography is the common method among the authors, being the main measurement method. In addition, the common unit used is percentage. Although the common units are in percentage (Alfakit and Chromatography), gas chromatography also presents results in ppm, just like MQ-4 (Table 4). Therefore, due to the practicality of the analysis, it was decided to keep the chromatography values in ppm.

Table 4 - Literature review about the methods and units used for methane concentration acquisition in biogas

Reference	Method	Unit
Weber et al. (2014)	Portable gas analyzer Drager®	m^3 day^{-1} %
Daniel (2015)	Gas Chromatography	%
Zanato (2014)	Gas Chromatography	%
Córdoba et al. (2017)	Gas Chromatography	mL g_{VS}^{-1}
Varol & Ugurlu (2016)	Gas Chromatography	%
Luo et al. (2017)	Gas Chromatography	% mL g_{TS}^{-1}
Chou & Su (2019)	Gas Chromatography	% e mL

CONCLUSION

It is concluded that all actions arising from the adapted code were recognized by the Arduino Uno® platform, and consequently by the sensor, and successfully implemented in the monitoring of biogas concentration and temperature.

The developed modules were able to perform the analyses with satisfactory results, indicating the proper functioning of the system developed.

The PMM and BMM functioned as a monitoring system, with values found as expected by chromatography, although the methane concentration by the sensors was in the appropriate range when compared to Alfakit.

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