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Proposal for a new methodological approach to measure the concentration of methane in biogas using a low-cost sensor.

Felipe Fernandes Lopes
Instituto Federal de Minas Gerais - Campus Arcos
lopes.ff@outlook.com

Reginaldo Gonçalves Leão Junior
Instituto Federal de Minas Gerais - Campus Arcos
Universidade de Santo Amaro
reginaldo.junior@ifmg.edu.br

Abstract.
The use of biogas for several applications is dependent on its composition, especially the concentration of methane. To analyze biogas samples in small systems, it is proposed to develop a low-cost system, with a methodology based on a set of chambers, for diluting the biogas sample to the levels where a simple and low-cost semiconductive sensor can be used accurately. For interpretation of the signal extracted from the sensor, the methods provided in the manufacturers' manuals are evaluated, as well as methods developed particularly for this project. Results are cross-checked with external analyses to ensure system accuracy. Among the methods evaluated, those with the greatest potential for use in the system are those developed particularly for this application, and should be recalibrated and validated experimentally in the continuation of the project.

Keywords: *biogas, methane, low-cost sensor*

1. INTRODUCTION

Considering the current energetic and environmental scenario, alternative energy sources have been gaining more space, including biogas. Biogas is a gas with high calorific value resultant of anaerobic decomposition of organic matter composed mainly of methane and carbon dioxide. It can be used for heating, power generation, internal combustion engines and also in domestic applications. It is generally produced in reactors called biodigestors, a technology that is widespread in rural areas of India and China, but still little used in brazilian rural areas (Santos, 2016). This type of reactor can be loaded with different types of organic material (e.g. animal manure on small farms).

The chemical composition of biogas may vary significantly depending on production and processing conditions. In order to homogenize the quality of the gas, there are several purification methods that are used to eliminate the inert components, increasing the concentration of methane and therefore the energy potential of the gas (Deublein and Steinhauser, 2011). To determine the energy potential of the biogas generated under certain conditions, checking its suitability for use, it's necessary to monitor the quality variation over time, to assess the effectiveness and the need for purification methods, as well as to determine the composition of the biogas. To achieve theses purposes there are several fixed and portable equipment available on the market, however, the high cost of this equipment makes its use inaccessible for small farms, where the costs of the equipment exceed considerably the total cost of the biodigester building.

This cost mismatch motivated the authors to propose the development of a system to measure methane concentration based on low-cost sensors. This work deals with the procedures and methods that make it possible to measure the methane concentration with a precision and reliability consistent with a low budget production unit.

Low-cost biogas analysis devices, if widely available, would be an important tool for the development of purification methods, and therefore an important step in expanding the production and use of biogas. Thus, this project is justified by proposing a methodology that can be widely replicated and adapted in different researches.

The general objective of the project is to formulate a methodology for analyzing biogas samples from a set of MQ-4 sensors connected to an Arduino board (or other microcontroller) and inserted in a dilution chamber system. To formulate this methodology, the properties of biogas, the technical characteristics of the sensors and different methods of interpreting the sensor signal are evaluated. In addition, the objective of the project was also to calibrate and validate the obtained data by means of external analyzes of commissioned devices.

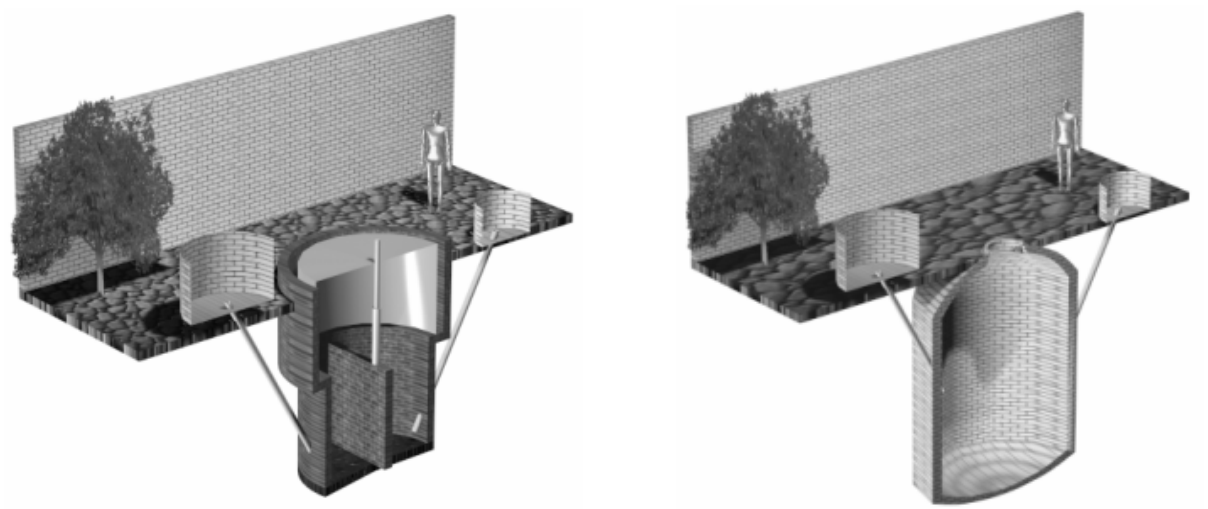
1.1 The Biogas

According to Souza (2006), the first scientific studies on anaerobic digestion of agricultural residues dates from 1808. The first facility to produce fuel gas was built in India in 1857, after this, the biodigesters spread throughout the country. At the same time in China, the biogas was being developed in rural areas as an energy source for cooking food and domestic lighting (Souza, 2006) (Seixas *et al.*, 1981).

Biogas is a renewable source of energy resulting from the anaerobic digestion of organic waste. It consists of a mixture of gases, with a predominance of methane and carbon dioxide. The composition of biogas varies according to the organic material used and the biodigestion process, with methane corresponding to values between 20 and 80% and 20 to 60% for carbon dioxide (Santos, 2016). In addition biogas usually contains between 0.3 and 2% of H_2S and traces of hydrogen and nitrogen (Coelho *et al.*, 2006).

The generation of biogas inside the biodigester takes place through biodigestion, which occurs in four stages, namely, hydrolysis, acidogenesis, acetogenesis and methanogenesis, when acetates are transformed into methane (Chernicharo *et al.*, 1997) (Deublein and Steinhauser, 2011).

The low-cost biodigestors, can be characterized as intermittent or continuous assembly. Intermittent biodigestors retain organic matter for a certain time, so that all biomass is decomposed, with periodic feeding (Fernandes *et al.*, 2012). Among the intermittent biodigester models the Indian and the Chinese model stand out. The Indian model has a loading box, an unloading box and a fermentation chamber. Its main features is the hood which is responsible for keeping the gas pressure constant and the central wall responsible for promoting the movement of organic matter inside the fermentation chamber (Fig. 1a) (Deganutti *et al.*, 2002).



(a) Indian model. (b) Chinese model.

Figure 1: A schematic representation of Indian and Chinese biodigesters.

The Chinese model has a loading box, a discharge box and a fermentation chamber. Its operating principle is the movement of organic matter through pressure gradients in the fermentation chamber caused by the accumulation of biogas. (Fig. 1b) (Deganutti *et al.*, 2002). On the other hand, continuous biodigestors have a constant volume of organic matter throughout the period of operation, with all volume variations at the inlet implying volume variations at the outlet Fernandes *et al.* (2012).

1.2 The Biogas Analysis

The analysis technique most commonly used today is based on gas chromatography (CG) instrumentalized by a flame inization detector (FID) (Poole, 2003). This technique has numerous advantages for industrial and field use, such as; high resolution, high temporal reading rate and excellent sensitivity to CH_4. However, it's expensive, with access costs generally incompatible with the construction costs of small plants, as previously mentioned (Kolb and Ettre, 2006).

Although not commonly used as reliable options for gas chromatography, methane sensors, that are more simpler than FID (mainly semiconductors) are easily found sold as electronic components for the detection of leaking flammable gases, among these components the most used is the the MQ4.

The MQ4 gas sensor (Fig. 2) consists of ceramic micro tubes made of AL_2O_3 (Fig. 2 - 5) with a sensory layer consisting of SnO_2 (Fig. 2 - 1).

	Parts	Materials
1	Gas sensing layer	SnO_2
2	Electrode	Au
3	Electrode line	Pt
4	Heater coil	Ni-Cr alloy
5	Tubular ceramic	Al_2O_3
6	Anti-explosion network	Stainless steel gauze (SUS316 100-mesh)
7	Clamp ring	Copper plating Ni
8	Resin base	Bakelite
9	Tube Pin	Copper plating Ni

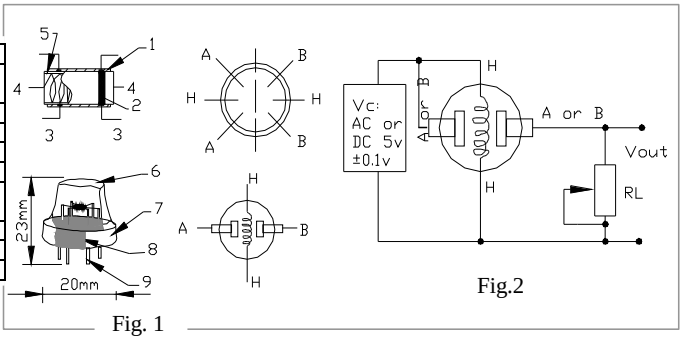


Figure 2: Technical data MQ4 gas sensor (Hanwei, 2007)

This sensor is able to detect the concentration of methane in its environment by increasing the conductivity of its sensitive components, which is low in clean air, continually converting the variation of conductivity into an output signal.

The MQ-4 sensor features high sensitivity for methane and natural gas, with a concentration sensitivity range for methane between 200 ppm and 10000 $ppm(1\%)$, a scale incompatible with the CH_4 concentrations found in the biogas (Hanwei, 2007).

The use of sensors like these would be conditioned to the controlled dilution of the initial sample in a larger volume chamber, until the sample reached concentrations compatible with the reading scale of the MQ4, that was the approach used in this paper.

Only three other works were found using this approach. In Frattaroli (2017) the method of signal interpretation used did not return satisfactory results for the sensors employed, keeping, in the second part of its discussion, the focus on signal processing methodologies from different sensor manufacturers. Yang *et al.* (2019) obtained great results showing the feasibility of using the sensor, but does not present a methodology of signal interpretation and calibration that can be surely replicated. Fakra *et al.* (2020) focuses his discussion on the measurement protocols comparing results between the MQ4 a nd MQ8 sensors. It reports promising results that are close to those obtained in this work with respect to the dimensionality of the CH_4 sample.

Despite the success reported by Fakra *et al.* (2020), the present work diversifies the modeling alternatives. It introduces the possibility of heuristic search for solutions to expand the possibilities of adaptability of the modeling, considering that a computational model has static numerical behavior , whereas an intelligent computational method can be adaptive operating in a more elastic way under different conditions.

2. METHODS & DESIGN

This project is motivated by the construction of a medium size biodigester of the Indian model. As the construction of the biodigester was executed at low-cost, any integrated control and analysis system will necessarily be low-cost too. So, the first option evaluated is to perform the biogas composition analysis using the MQ4 sensor. As concentrations close to 60 and 80% are expected in the gas and the sensor saturates in concentrations above 10,000 ppm, it's necessary to develop a measurement method that is sufficiently precise for its intended purpose despite the limitation of the sensor (Colatto and Langer, 2011).

2.1 Gas dilution

The proposed system consists of two storage chambers, one filled with biogas and the other filled with ambient air, free of methane. Accurate measurement of the methane concentration in the biogas sample is obtained by diluting the sample in air, reducing the concentration to levels where the sensor gives reliable results. The measurement obtained by the sensor corresponds to the number of methane moles in relation to the total number of moles of the system. The objective of the process is to obtain the methane concentration in the sample, defined by the number of methane moles in the system in relation to the number of moles in the sample.

To calculate the number of mols (n) in each chamber it was necessary to know the gas mass (m) and the molecular weight (mw) of the gas (Boles and Çengel, 2013).

$$n = \frac{m}{mw} = \frac{v \cdot d}{mw} \tag{1}$$

Table 1 shown the relationships between densities (d) and molecular weights are approximately constant (0.3% variation), so that the number of mols in each chamber can be calculated on the basis of the volume of gas alone (v), regardless of the composition of the biogas sample (data obtained by the software Coolprop).

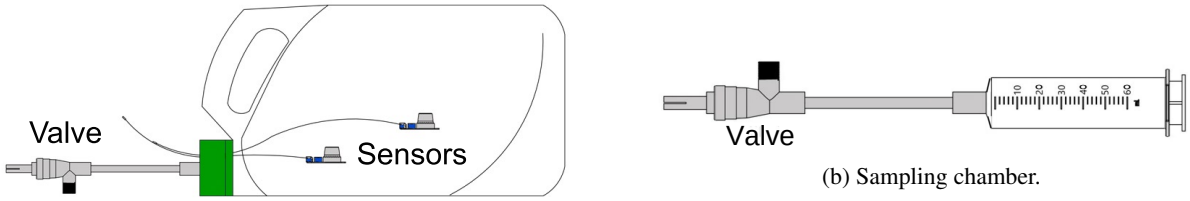
Table 1: Gas properties

Gas	Density (kg/m^3)	Molecular weight (kg/mol)	d/mw (mol/m^3)
CH_4	0.657	0.016	40.946
CO_2	1.808	0.044	41.081

2.2 Sizing of the chambers

To perform the chamber sizing a specific case with 100% methane concentration in the sample and reading of $10.000\ ppm$ was taken as a reference. Once the volume of the expansion chamber has been defined, a procedure based on Amagat's Law is used to calculate the corresponding volume for the sampling chamber. The result is a dilution ratio of 99.14 for biogas (Boles and Çengel, 2013).

For a test model it was defined that the chamber would have 3 liters of volume (Fig. 3a).



(a) Expansion chamber.

(b) Sampling chamber.

Figure 3: Set of cameras used during the experiment.

Depending on the dilution ratio, the sample chamber volume should be 30.3 milliliters. For this volume it's proposed to use a syringe of 60 millilitres for the sampling chamber (Fig. 3b).

The complete system used for the sensor tests is shown in Fig. 4.

Figure 4: Test system

The tests performed in the setup shown in Fig. 4 followed the ensuing steps:

1. Collection of a 60 milliliter of biogas by the sampling chamber.
2. Elimination of residues in the expansion chamber through an air flow.
3. Chamber coupling.
4. Initial reading in clean air.
5. Handling of the valves and insertion of the sample into the expansion chamber in discrete volumes of 5 milliliters.
6. Readings for volumes from 5 to 60 milliliters.
7. Record and analysis of collected data.

2.3 Signal Interpretation

Each of the two MQ-4 sensor returns an analog reading that can range from 0 to 1023 which corresponds to a variation between 0 and 5 volts. To obtain the concentration in ppm from the sensor reading some conversion method must be used.

The manufacturer of the sensors used in the test system does not provide a method for conversion to analog signal. Therefore, methods based on manuals from other manufacturers were evaluated, as well as the development of a new method, both described in the sequence of this section.

As a basis for the interpretation of the analog signal, an experiment was performed on a sample whose composition was analyzed externally by a project partner. In this experiment the analog sensor signal was recorded for discrete sample

volumes added in 5 ml increments. Additionally, two sequences of three experiments each were performed to check the consistency of the data, it can be considered that the samples have the same chemical composition, as they were collected in a short time between them.

2.3.1 Concentration from eletric voltage

According to Sparkfun (2016) and Winsen (2015), the particle concentration per million can be determined after converting the analog reading to the corresponding voltage by means of:

$$\varphi_e = 10.938 \cdot e^{1.7742 \cdot V} \tag{2}$$

Where φ_e is the concentration of methane in the expansion chamber in ppm and V is the voltage in volts.
The Eq. (2) is shown in Fig. 5, where the error bars show the difference between the models of the two references.

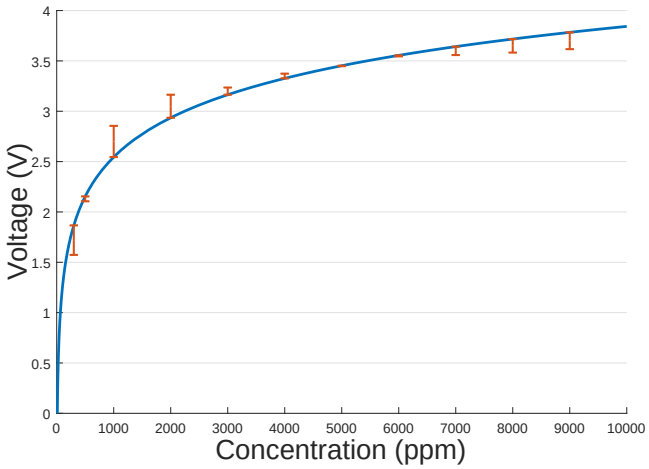


Figure 5: Voltage as a function of concentration - Adapted from (Sparkfun, 2016) and (Winsen, 2015)

2.3.2 Concentration from resistance

According to Hanwei (2007), The methane concentration in the sensor can be determined based on the relationship between the current resistance and the reference resistance. The current resistance (Rs) is given by the Eq. (3). Where Ve is the input voltage of the circuit, Vs is the voltage corresponding to the sensor reading and Rl is the charging resistance used in the sensor circuit.

$$Rs = \left(\frac{Ve}{Vs} - 1\right) \cdot Rl \tag{3}$$

The function suggested by Hanwei (2007) to determine the corresponding methane concentration is shown in Fig. 6.

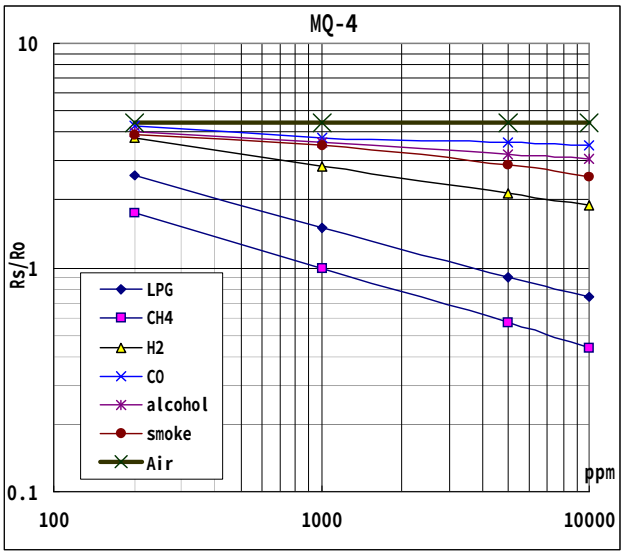


Figure 6: Sensitivity characteristics of MQ4 gas sensor (Hanwei, 2007)

The corresponding equation is:

$$ln\left(\frac{Rs}{Ro}\right) = 2.4606 - 0.3562 \cdot ln\varphi \tag{4}$$

A function can also be defined from the data collected with known concentration, as presented in the following section.

2.3.3 Concentration from analog signal - Mathematical modelling

The curves of readings presented by Sparkfun (2016) and Winsen (2015) describe an asyntotic exponential behavior, so that volume increments produce smaller reading increments when the reading approaches saturation (Fig. 5). This behavior can be modeled by means of a first order differential equation. The hypothesis adopted for the variation of the analog reading in relation to the volume of biogas in the system is presented by the Eq. (5).

$$\frac{dL}{dv} = \lambda(L_{sat} - L) \tag{5}$$

Where λ is a constant related to the concentration of methane in the sample and also the characteristics of the sensor, L_{sat} is the maximum sensor reading for a specific condition (indicates sensor saturation) and L is the current sensor readout. Using the integral factor method for the resolution of the differential equation, it is obtained (Çengel and Palm, 2013):

$$L(v) = L_{sat} - (L_{sat} - L_0)e^{-\lambda v} \tag{6}$$

2.3.4 Concentration from analog signal - Metaheuristics

Another option addressed to determine the methane concentration in the sample is based on the equation:

$$\varphi_a \cdot \frac{n_a}{n_a + n_{ar}} - \varphi_e = 0 \tag{7}$$

Where φ_a is the concentration of methane in the sample in ppm, n_a is the number of mols in the sample, n_{ar} is the number of air mols in the expansion chamber and φ_e is the concentration of methane in the expansion chamber in ppm.

An algorithm was developed to minimize the absolute value of the equation, using the metaheuristics Simulated Annealing (SA). The collected analog reading for 30 ml of diluted biogas and the calibration profile obtained by the experiment with the sample of known composition were used.

The implemented code starts from Eq. (7) considering that the number of air moles in the expansion chamber (n_a) and the methane concentration in the expansion chamber (φ_e) points after sample injection can be safely determined, the first value by thermodynamic methods and the second by reading sensor corrected by the calibration curve. These premises reduce the number of variables in the objective function to just two, the number of moles in the sample (n_a) and the concentration of methane in the sample (φ_a), where the latter is the desired value.

The implementation of the SA used can be briefly describedby the Tab. 2.

Table 2: SA algorithm steps

STEP 1.	Initializes the code by loading the objective function, the restrictions (upper and lower limits to search for each variable) `conj_sol`, the cooling rate of the heuristic method `raz_resf`, the number of temperature iterations `n_inter_temp`, the initial temperature `t_0` and the dummy suggestions of n_a and ϕ_a, `phi` and `n` respectively;
STEP 2.	Iterate in temperatures until the minimum temperature (stopping criterion) is achieved.
STEP 3.	For each iteration in temperature, perform a nested sequence of iterations determined by `n_inter_temp`.
STEP 4.	In each iteration, random values belonging to `conj_sol` are raised by the code and the adequacy of these new values compared to the adequacy of the current values of `phi` and `n`. The checks of adaptation take place through the solution of the objective function and the evaluation of its proximity (`delta_sol`) with zero. Survives the pair of solutions (either the old one or the one drawn in the current iteration) with better proximity to the objective value of the function.
STEP 5.	If a new pair of solutions resulting from the random draw passed through verification in 4, then a control variable of the population accepted by temperature `neighbors` is incremented unitarily, if not, the Boltzmann's criterion is accessed, and the `delta_sol` evaluated by $x < exp(-delta_{sol}/t_0)$, where `x` is drawn at randomly each time the Boltzmann criterion is accessed, ending the current iteration for the actual temperature.
STEP 6.	If `neighbors` is less than 60% of `n_inter_temp`, then the temperature is increased by the factor $1+gamma*\sqrt{(t)}$, otherwise is decremented by the factor $t/(1+gamma*\sqrt{(t)})$, where (gamma) is a internal constant to control heating and cooling rates.
STEP 7.	Once the new temperature is determined, repeats steps 3 to 6 until the iterations in temperature have reached the stopping criterion $t = 1 \cdot 10^{-6}$.

3. RESULTS & DISCUSSION

To check the compatibility of each model with the sensor samples used in the tests, a comparison was made between the results of each model and the results of the external analysis.

Three samples were collected for external analysis, performed with the apparatus OPTIMA 7 Biogas Analyzer. The results of the analysis are presented in Fig. 7 and on the Tab. 3. According to MRU (2018) the accuracy of OPTIMA 7 for methane analysis is $\pm$ 4 or 5 % of the measured value.

Figure 7: External analysis

Table 3: Analysis results

	Sample 1	Sample 2	Sample 3	Average
Concentration of CH4 (%)	58.63	57.79	57.94	58.12
Concentration of H2S (ppm)	103	98	102	101
Concentration of CO2 (%)	35.49	34.78	35.18	35.15
Concentration of O2 (%)	1.6	1.9	1.7	1.7
Lower Calorific Power (MJ/Kg)	17.7	17.4	17.4	17.5
Superior Calorific Power (MJ/m^3)	23.3	23.0	23.1	23.1

Between samples 1 and 2 sent for analysis, the 60ml sample was collected for internal analysis using the system presented by Fig. 4. In order to draw a calibration curve for the sensor, the sensor responses to different concentrations of methane, obtained by injecting discrete volumes of gas with known concentration in a controlled manner, were evaluated. The results of this reference experiment are presented by Fig. 8.

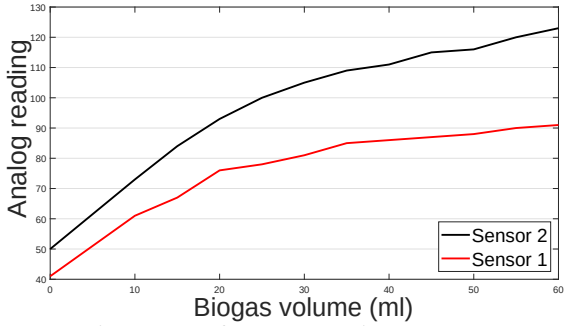


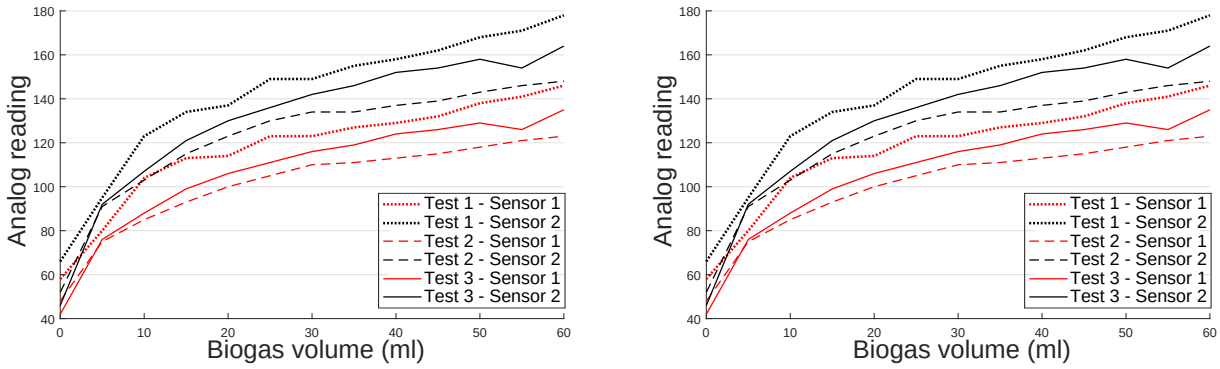
Figure 8: Reference experiment - 21/02

The concentration of the sample used in the experiment can be estimated at 58.21% according to the Tab. 3, which means that the sensors were exposed to a concentration of 5.821 ppm when 30ml of the sample had been inserted.

In order to characterize the behavior of the sensors, two sequences of experiments were performed, consisting of three experiments each, in which were measured the analog responses of the sensors for increments of 5ml of biogas in the system.

The actual concentration of the sample was unknown in these experiments, however, the collection of samples with minimal time lag supports the hypothesis that in each sequence the three experiments had samples of the same chemical composition. To eliminate errors due to residual readings, a forced ventilation procedure was inserted over the sensors and over the expansion chamber before each experiment.

The data collected in the sequences of experiments are presented by Fig. 9a and 9b.



(a) First sequence - 07/03.

(b) Second sequence - 14/03.

Figure 9: Analog reading of MQ4 as a function of the biogas volume injected into the expansion chamber.

3.1 Concentration from eletric voltage

To check the validity of the method proposed by Sparkfun (2016) and Winsen (2015) the analog readings were converted into voltage and then methane concentration in parts per million by the Eq. (2). The values obtained are presented in the Tab. 4.

Table 4: Results obtained

Test date	Voltage	Eq. (2)
21/02	0.63	33.48
07/03	0.61	32.01
14/03	0.45	24.50

The divergence of the values presented in the Tab. 4 for the previously calculated values indicate that the Eq. (2)

extracted from the sensor manuals is not suitable for the MQ4 sensor copies used.

3.2 Concentration from resistance

The experimental data compose the calibration curve presented by the Fig. 10.

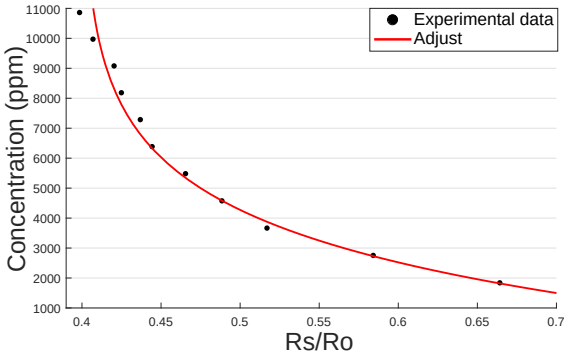


Figure 10: Calibration curve for resistance

The function obtained from the experimental data is:

$$\varphi_e = -2522.07 \cdot ln\left(\frac{Rs}{Ro} - 0.4002\right) - 1539.63 \tag{8}$$

The results obtained from the resistance are presented in the Tab. 5

Table 5: Results obtained

Test	Rs/Ro	Eq. (4)	Eq. (8)
21/02	0.47	8459.669	5269.64
07/03	0.37	15997.23	-
14/03	0.41	12595.04	11613.34

The results obtained by the Eq. (4) do not match the methane concentration range for this type of biogas and not even the operating range of the sensor, and is therefore inadequate for the interpretation of the sensor signal. The results of the Eq. (8) are reasonably accurate for the calibration point, however they cannot be applied for certain values of Rs/Ro and presented values outside the operating range of the sensor and should be recalibrated for a proper evaluation of its applicability.

3.3 Concentration from analog signal - Mathematical modelling

To test the format of the obtained equation, the curve fitting tool was employed (*software* Matlab®). The model showed good compatibility with experimental data sets.

In particular, for the experiment with known sample composition (02/21/20) the model of adjustment was extended to the variation of the reading as a function of the concentration. The results obtained are presented in Tab. 6 and by the Fig. 11 (where R-square is a parameter that indicates the compatibility of the adjustment to the data, being 1 the maximum value).

Table 6: Adjustment results

	L_{sat}	L_0	λ	R-square
Sensor 1	129.95	49.43	$2.0825{\cdot}10^{-4}$	0.9973
Sensor 2	93.11	40.78	$2.7544{\cdot}10^{-4}$	0.9962
Data average	111.15	45.12	$2.3481{\cdot}10^{-4}$	0.9977

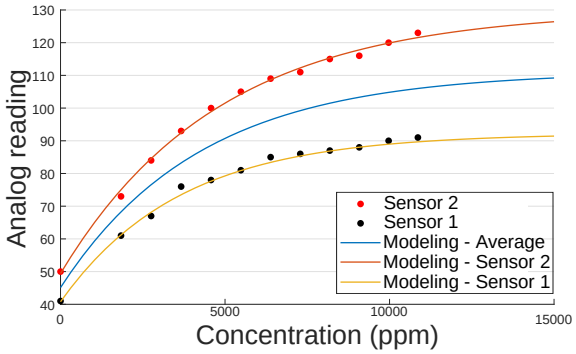


Figure 11: Adjust

Attempts to extrapolate the variation of the reading of each sensor according to the concentration to the test sequence did not show coherent results. So, possibilities of normalising the reading were evaluated. The first one of the options considered was the normalisation by the maximum reading established by the adjustments (Fig. 12)

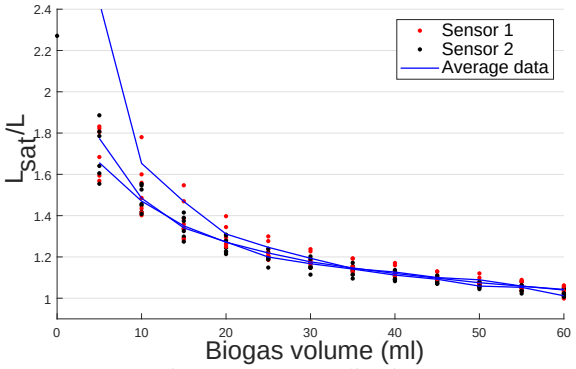


Figure 12: Normalisation

The behaviour of the standardised reading by the maximum reading showed potential for estimation of the sample concentration through the readings, therefore the adjustment for the average of the data was made in order to eliminate or soften the noise in the readings (Fig. 13).

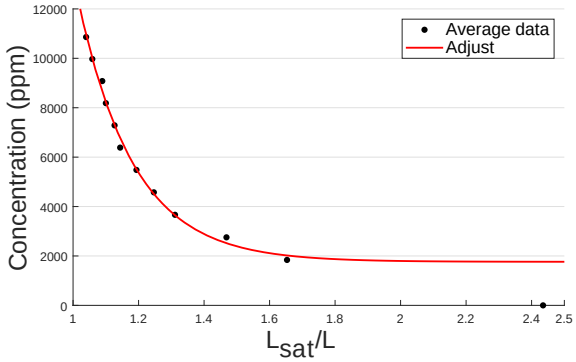


Figure 13: Adjust

The corresponding equation is:

$$\varphi_e = 3.964 \times 10^6 \cdot e^{-5.835 \cdot L} + 1763 \tag{9}$$

Using the Eq. (9) it is possible to estimate the concentration of the samples from the reading corresponding to the volume of 30 millilitres of biogas in the system.

Table 7: Estimated methane concentration - Analog signal

Test date	Estimated concentration	Gas analyzer	Deviation
21/02	58.21%	58.63%	-0.42%
07/03	65.30%	57.79%	+7.51%
14/03	63.62%	57.94%	+5.68%

The results obtained are inconclusive as there is no reference for validation other than the data used for calibration.

3.3.1 Concentration from analog signal - Metaheuristics

The results of the Simulated Annealing algorithm for the experimental data are presented by the Tab. 8.

Table 8: Estimated methane concentration - Metaheuristics

Test date	Estimated concentration	Gas analyzer	Deviation
21/02	64.03%	58.63%	+5.40%
07/03	61.70%	57.79%	+3.91%
14/03	64.23%	57.94%	+6.29%

Similarly to the modelling method, the results obtained by metaheuristics are not conclusive.

4. CONCLUSION

The project had as objective the development of a low cost system for analysis of methane concentration in biogas, based on a semiconductive sensor with a limited sensitivity range. A dilution system has been proposed to know the levels at which the sensor gets accurate readings.

The data collected in tests with the system showed incompatibility between the sensor behavior and the signal analysis methodology suggested by the manuals of different manufacturers. Different signal analysis methods were then evaluated from different parameters and based on experimental data. From the analyses made it is possible to conclude that three methods have the potential to compose an on-board system capable of performing methane concentration analyses on biogas samples with satisfactory precision, they are those based on resistance, mathematical modeling and metaheuristics.

For the completion of the project, combinations of external and internal analysis must be performed to recalibrate and validate the methods for interpreting the sensor readings.

As external analyzes are foreseen by different devices, an analysis of uncertainties will be conducted in the next stages of the project too.

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