

BIOGAS GENERATION THROUGH PREDICTIVE MODELS WITH EXPERIMENTAL DESIGN

Arturo Hernández Hernández, Joaquín Pérez Meneses, María Blanca Becerra Rodríguez and José Marcos Zea Pérez

SUMMARY

This article presents a development for the generation of biogas from cricket excreta, optimizing the formulation to increase energy production from the control of factors such as: temperature, pH, water and excreta. The experiments were carried out with different formulations, using Kitasato flasks through the water displacement method. The optimization analysis was performed by applying three experiment design strategies; 2^2 with blockage, 2^2 with increased blockage and $3^1 \times 2^2$, where the temperature and the cricket excreta were significant factors. As a result, the regression equation of the formulation was obtained to predict the generation of biogas.

Introduction

In recent years, the interest in the production of bioenergy has increased significantly. Various technologies and different processes are used to recycle, reuse organic waste and one of them is the generation of biogas. It generally refers to the gas that contains mainly methane, which is obtained from the anaerobic digestion of organic matter such as biomass, manure, sewage, municipal waste, green vegetables, and energy crops. Anaerobic decomposition (fermentation) of organic matter produces carbon dioxide, methane, hydrogen, and other gases. Biogas production is a technology that can make efficient use of organic matter despite the high-water content. The yield and composition of biogas depends on the nature or type of the substrate fed to the digester (Lorenzo *et al.*, 2005).

Small-scale biogas production has been promoted, mainly from bovine manure, in order

to obtain energy and organic fertilizer. It is estimated that there are more than 30 million domestic digesters in China, followed by India with 3.8 million, 0.2 million in Nepal, 60,000 in Bangladesh, and in African countries such as Kenya and Ethiopia more than 1,000 each (Arrieta *et al.*, 2016).

Statistical design of experiments (DOE) has gradually been considered in the industry as a set of statistical engineering tools, which allows achieving maximum process efficiency with minimum cost. A statistical DOE can solve problems for the optimization of manufacturing processes (Navarrete, 2018).

The appropriate potential of hydrogen (pH) must be in a range between $6.5 < \text{pH} < 7.5$ to avoid destabilization of the process due to the accumulation of volatile fatty acids and the formation of inhibitory compounds. The process temperature must be constant and when the volatile fatty acids (VFA)

decrease, the substrate inside the reactor is running out, which is why the biogas production rate decreases to the point where the production is zero, because the anaerobic microorganisms die and the process stops (Martínez, 2016). The pH before the experiment was found to be within the optimum range for biogas production which is 6.6 - 7.6. After the experiment, the pH values were observed to increase slightly (Momoh *et al.*, 2008). One of the main factors during the degradation of the organic matter to produce biogas is to take care of the pH of the formulation, one of the actions to regulate the pH is with a solution of sodium bicarbonate (Heredia *et al.*, 2015).

The temperature has a direct relationship with the dynamics of the process. Any significant variation in temperature translates into an imbalance in the process, which generates inhibition in the production of biogas. If this is achieved, greater

biogas production can be obtained, compared to systems without temperature control (Corona, 2007). In the investigations consulted, it was determined the average temperature for a study period being 26°C (Rivas *et al.*, 2010).

To assess the feasibility of biogas generation, many researchers have carried out tests with different materials that are often considered as organic waste whether domestic, industrial, or agricultural. The production of biogas from organic materials depends on substances that break down into CH_4 and CO_2 . The crude protein, crude oil, fiber, cellulose, hemicellulose, starch, and sugar are quite effective in methane formation (Senol, 2019). In a research work it was possible to evaluate the changes in the physicochemical properties of matooke hulls that improve the biogas production of different crops under environmental conditions of storage and optimization for different hull

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GENERACIÓN DE BIOGÁS A TRAVÉS DE MODELOS PREDICTIVOS CON DISEÑO DE EXPERIMENTOS

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RESUMEN

Este artículo presenta el desarrollo para la generación de biogás a partir de la excreta de grillo, optimizando la formulación para incrementar la producción de energía a partir del control de factores como son: temperatura, pH, agua y excreta. Las experimentos se realizaron con diferentes formulaciones, utilizando matraces Kitasato a través del método de despla-

amiento de agua. El análisis de optimización se realizó aplicando tres estrategias de diseño experimental; 2² con bloqueo, 2² con incremento de bloqueo y 3¹ x 2², donde la temperatura y la excreta de grillo son factores significativos. Como resultado se obtuvo la ecuación de regresión de la formulación para predecir la generación del biogás.

GERAÇÃO DE BIOGÁS ATRAVÉS DE MODELOS PREDITIVOS COM DESENHO EXPERIMENTAL

Arturo Hernández Hernández, Joaquín Pérez Meneses, María Blanca Becerra Rodríguez e José Marcos Zea Pérez

RESUMO

Este artigo apresenta o desenvolvimento para a geração de biogás a partir de excrementos de grilos, otimizando a formulação para aumentar a produção de energia através do controle de factores como: temperatura, pH, água e excretas. Foram realizadas experiências com diferentes formulações, utilizando matraces Kitasato através do método de deslocamento de água.

A análise de optimização foi realizada através da aplicação de três estratégias de concepção experimental; 2² com bloqueio, 2² com incremento de bloqueio e 3¹ x 2², em que a temperatura e os excrementos dos grilos são factores significativos. Como resultado, foi obtida a equação de regressão da formulação para prever a produção de biogás

particle size systems (Tumutegereize, 2011). In the experiments consulted, formulations were used with materials such as: Tomato residues, corrugated pumpkin leaf residues, chicken manure, sheep manure, cow manure, water and bacterial sludge. The effect of the substrate on the biogas yield was investigated and the results showed that sheep manure provided the highest cumulative volume of biogas per 100g of substrate with 240ml, followed by cow manure with 200ml, the smallest one being with waste of tomatoes and chicken manure with accumulations of 170ml each (Adamu, 2014). Some researchers make their formulations with one or more organic materials. In a study, organic solid residues were obtained from potato peel, vegetable peel (cucumber, carrot, and spinach), fruit peel, reduced to a particle size of 2mm, and 400g of said sample were taken together with 1200ml of distilled water. Adding 400ml of cow and horse inoculum. The following

operating conditions were maintained: Stirring every 8 hours for 5 minutes at 120rpm, system temperature to 30°C through the control system (Martínez, 2016).

A researcher collected cow dung from slaughterhouses, flying wish paper, and water hyacinth from rivers. Pretreatment of the operations involved 500g of freshly harvested water hyacinth allowing it to dry in the sun for a period of 30 days, then they were dried in an oven at 600°C for 5 hours. This water hyacinth is kiln dried and ground to a fine particle using a mill. These biomasses were mixed with 250ml of water. The contents of the digesters were left to ferment for a period of 62 days, stirring twice a day, in the morning and evening hours, respectively. The measurement of biogas was carried out using the water displacement method. It could be seen that the effect of waste paper in a fixed amount of cow manure and water hyacinth increases biogas production in a

parabolic way. In this investigation, a paper concentration of 17.5g was the maximum amount of wastepaper necessary to combine with 5g of cow manure and 5g of water hyacinth for maximum biogas production (Momoh *et al.*, 2008). In another work, the amount of residue produced from slaughtered cattle was refined. It was estimated that a cattle could produce waste of: 6.4kg of tissue, 8.0kg of intestinal content, 11.8kg of bone and 12.6 kg of blood. These residues have the potential to generate a total energy of 246,027.01kWh/year (Sindibu *et al.*, 2018).

The particle size of the organic matter is an important parameter to produce biogas, being smaller contributes so the bacteria can efficiently digest the organic matter, and this is reflected in the amount of energy generated. One researcher was able to observe that the optimum particle size of banana (matooke) peels for anaerobic digestion was 6.73mm; however, it was

shown that further work is required to validate this optimally projected particle size (Tumutegereize *et al.*, 2011). In another work, organic solid residues of potato peel, vegetable peel (cucumber, carrot, and spinach), and fruit peel were used, which were crushed until obtaining a particle size of less than 2 mm to improve hydrolysis (Martínez *et al.*, 2016).

In the works carried out to produce biogas, a Batch type reactor with a capacity of 3 liters was used, it had:

- A compression system made up of a motor and a propeller.
- A biogas outlet valve to take out the samples.
- A visualization point to extract substrate during the experiment, allowing to measure and monitor its pH and, in turn, measure the concentration of volatile fatty acids (VFA).
- A Dallas DS18S20 temperature sensor which allows monitoring the internal temperature of the reactor (Martínez *et al.*, 2016).

The reactor heating system was composed of:

- A water jacket.
- An electric pump (to recirculate the water through the heater and the reactor).
- An 800W electrical resistance (heater).
- A phase angle variation actuator module to vary the voltage on the heater (which allows to vary its temperature) by means of a control signal (0-5V). Figure 1 shows the characteristics of the Batch type reactor (Martínez *et al.*, 2016).

Another researcher used the water displacement method. The formulation consisted of feeding the bottle of the biodigester which was connected to the gas collection bottle filled with water and then attached to a graduated container. The biogas produced is measured by looking at the amount of water displaced from the gas collector to the graduated container (Adamu, 2014) as seen in Figure 2.

Another investigation used a set of five discontinuous reactors as digesters, using biogas measurement through the water displacement method, the material used was Kitasato flasks (Momoh *et al.*, 2008) as seen in Figure 2.

In summary, the problem consists of determining the

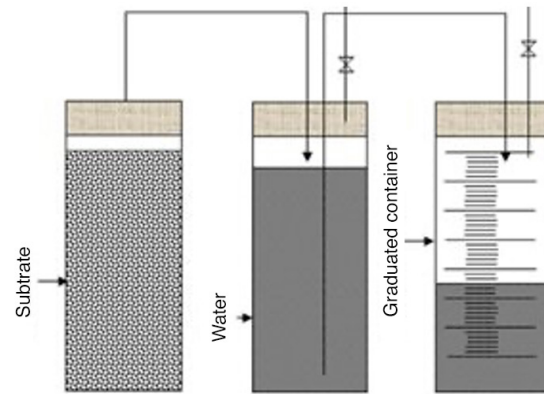


Figura 2. Water Displacement Method.



proportion to obtain the formulation and the volume necessary to produce liters of biogas. To achieve this, it was necessary to control factors such as: Temperature, dosage of organic matter, amount of water and pH.

Methodology

The purpose of this work was to study the production of biogas as a byproduct of the anaerobic digestion of cricket excreta, with the objective of establishing a statistical predictive model to obtain the optimal formulation at the laboratory level.

The methodology used for the development of this

research work is as follows (Gutiérrez *et al.*, 2012).

Planning and design

- Define the problem to be solved and establish the objective of the work.
- Determine the factors to study and the characteristics to produce biogas.
- Select the experimental design according to the needs of the problem.
- Plan the experimental work.

Experimentation

- Carry out experiments.
- Perform tests.

Analyze and interpret statistical data

- Study the results.

Optimization of results

- Define if the characteristics are optimal, otherwise return to the planning stage.

Final conclusions

- Determine the optimal operating conditions of the process.
- Documentary results.

Experimentation

Equipment /Apparatus

For this research, the following equipment was used: Digester built with Kitasato flasks, analytical scale,

crucibles, beaker, pH paper strips, pipette, test tube, muffle, stove, plastic funnel, digital thermometer, electric extension, spotlight, digital pH meter and glass box for temperature control.

Material

The material used for this project: Water, cricket droppings and cow droppings.

Details of the experiment

In the first experimentations carried out, it was not known how biogas was generated in the absence of oxygen, called anaerobic disability. Tests were carried out in a plastic container with cricket and cow excreta, as shown in Figure 3.

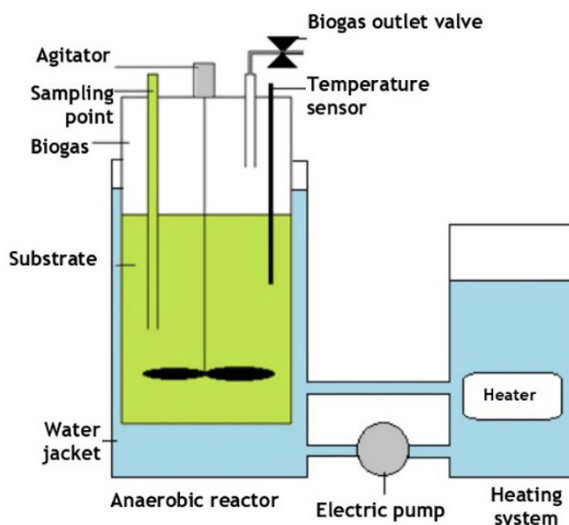


Figure 1. Batch type reactor (Martínez *et al.*, 2016).



Figura 3. Exploratory tests for biogas production.

These tests allowed to visually compare the production of biogas from both organic materials, a similar action was observed in both organic materials, but since the containers do not have sufficient resistance to the pressure generated, another option was sought.

In the first experiment carried out in this research, the behavior of the anaerobic process was evaluated, based on the results obtained, a second experimental phase was developed through the water displacement method that helped evaluate the production of biogas by measuring the amount of water moved as shown in Figure 2.

The process begins by mixing the water and the excreta of the cricket in a Kitasato flask connected through a rubber hose to another Kitasato flask where the liquid to be displaced is stored, beginning the tests later for the generation of biogas.

Three experimental design strategies were developed:

- The first strategy consists in a 2^2 block design of experiments, which seeks to determine the significance of the possible factors.

- In the second strategy, a 2^2 design of experiments with blocking increment was carried out to determine the feasibility of converting the noise variable into a control factor.

- In the third strategy, a $3^1 \times 2^2$ design of experiments was carried out to determine the level of scaling when verifying the significance of the factors.

These experiment design strategies are shown in Figure 4.

Results and Discussions

With the experiments carried out, it was shown that there is a direct relationship in the production of biogas when the scales vary, as shown in Figure 5, for this reason it is possible to determine the formulation to estimate the production of biogas.

The first strategy was carried out by making a 2^2 design

with blocking as shown in Table I where block 1 was made at an average temperature of 28°C , block 2 was made at an average temperature of 17°C and block 3 at an average temperature of 24°C , the formulations were made with a minimum amount of water of 80 ml represented with the number -1 and a maximum amount of 160 ml represented with the number 1 and a minimum amount of excreta of cricket of 10 gr represented with the number -1 and a maximum of 20 gr represented by the number 1, once the design of experiments was established, the experiments continued, obtaining the displacement of water.

It was found that the block and the excrete are significant, therefore, there is a model that has an R-sq of 93.69%, R-sq (adj) of 88.44%, because the difference between these two values is greater than 3%, it indicates that there is overfitting, as it can be seen there are variables that are not significant within the regression, the prediction level leads to 74.78%, as shown in Figure 6.

In Table II, the second design strategy 2^2 with increased blocking was performed, ordering the blocks according to temperature, assigning block 1 to the experiments carried out at low temperatures, block 2 at intermediate temperature and block 3 at the higher temperature.

This design was developed as a design of experiments controlling the temperature variable, observing that temperature and excreta are also significant and that the temperature effect, which is a quadratic effect, is also significant, in the R-sq regression model is 96.04% and the R-sq (adj) is 89.12%, and since the difference between these two values is greater than 3%, it indicates that there is overfitting, which means that there are variables that are not significant within the regression, the level of the prediction leads to 49.78%, as shown in Figure 7.

Once the blocks with temperature were completed, the

third strategy was carried out to determine the significance of the factors, performing the

reduction of terms to determine the significant factors in the formulation.

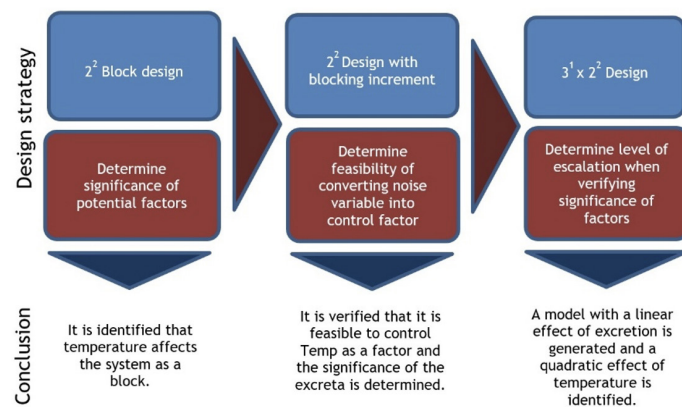


Figure 4. Design strategies.

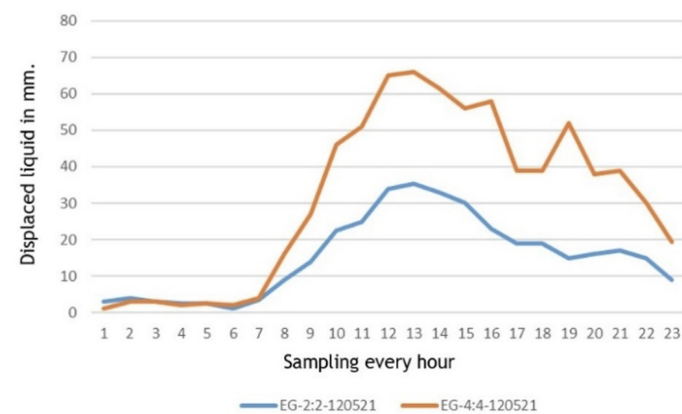


Figure 5. Scalability in biogas production.

TABLE I
BLOCK TESTS

Test	Block	Water	Excrement	Water displacement
1	1	1	1	349.0
2	1	1	-1	160.0
3	1	-1	1	252.0
4	1	-1	-1	241.0
5	2	1	1	12.5
6	2	1	-1	4.0
7	2	-1	1	23.0
8	2	-1	-1	11.0
9	3	1	1	310.5
10	3	1	-1	222.0
11	3	-1	1	277.5
12	3	-1	-1	187.0

The first reduction of terms was performed to optimize the model: Values not significant were removed, including water interactions, therefore we have

a model with an R-sq of 95.96%, R-sq (adj) of 91.12%. Since the difference is 4.84%, greater than 3%, it indicates that there is still an overfitting,

which means that there are variables that are not significant within the regression, resulting in a prediction level of 74.69%, as shown in Figure 8.

It can be stated that a better model is being found.

A second reduction was carried out where the interaction of the water was eliminated, which was not significant, therefore, we have a model that has an R-sq of 94.36%, R-sq (adj) of 91.14%. Since the difference is 3.22%, greater than 3%, although there is very little variation with respect to 3%, there are still variables that are not significant within the regression, resulting in a prediction level that leads to 81.87%, as shown in Figure 9, it can be stated that a better model is being found.

The third reduction of terms showed that the quadratic temperature and the excreta are significant, with an R-sq of 92.09%, R-sq (adj) of 89.12%. Since the difference is 2.97%, less than 3%, it results in a prediction level of 82.20%, as shown in Figure 10, it can be stated that a better model for prediction has been found.

Therefore, Equation 1 shows the regression that will help predict biogas production, based on the experimental design carried out by blocks.

$$\text{Liq. disp.} = -459.4 + 590 \text{ Temp.} + 33.3 \text{ Excreta} - 117.7 \text{ Temp.*Temp.} \quad (\text{Eq. 1})$$

It can be seen in Table II of the temperature control block, that the largest liquid displacement was 349 ml, which was obtained in block 3. To obtain the value of displaced liquid using (Eq. 1) and substituting the variables of temperature by 3 (higher temperature), the excreted by 1 (larger excreted portion), a value of 284.6 ml of displaced liquid is obtained, when comparing both values a percentage of 81.55% can be observed, which is very close to the 82.20% shown in Figure 10 of the third reduction of terms.

Flammability tests were conducted during the experiments in order to verify the viability to produce heat energy with cricket excreta, as shown in Figure 11. Due to the amount of gas produced, chromatographic tests will be

Coded Coefficients

Term	Coef	SE Coef	T-Value	P-Value	VIF
Constant	170.8	12.5	13.70	0.000	
Blocks					
1	79.7	17.6	4.52	0.004	1.33
2	-158.2	17.6	-8.97	0.000	1.33
Agua	5.5	12.5	0.44	0.672	1.00
Excreta	33.3	12.5	2.67	0.037	1.00
Agua*Excreta	14.4	12.5	1.15	0.293	1.00

Model Summary

S	R-sq	R-sq(adj)	R-sq(pred)
43.1832	93.69%	88.44%	74.78%

Figure 6. Significance of possible values.

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Model	5	166252	33250.3	17.83	0.002
Blocks	2	150103	75051.6	40.25	0.000
Linear	2	13669	6834.3	3.66	0.091
Agua	1	369	368.5	0.20	0.672
Excreta	1	13300	13300.0	7.13	0.037
2-Way Interactions	1	2480	2479.7	1.33	0.293
Agua*Excreta	1	2480	2479.7	1.33	0.293
Error	6	11189	1864.8		
Total	11	177440			

Coded Coefficients

Term	Coef	SE Coef	T-Value	P-Value	VIF
Constant	249.2	20.9	11.90	0.000	
Temp.	118.9	14.8	8.03	0.001	1.00
Agua	5.5	12.1	0.46	0.671	1.00
Excreta	33.3	12.1	2.75	0.051	1.00
Temp.*Temp.	-117.7	25.7	-4.59	0.010	1.00
Temp.*Agua	4.2	14.8	0.28	0.791	1.00
Temp.*Excreta	22.4	14.8	1.51	0.204	1.00
Agua*Excreta	14.4	12.1	1.19	0.300	1.00

Model Summary

S	R-sq	R-sq(adj)	R-sq(pred)
41.8954	96.04%	89.12%	49.78%

Figure 7. Design of experiment with control of variables.

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Model	7	170419	24346	13.87	0.012
Linear	3	126838	42279	24.09	0.005
Temp.	1	113169	113169	64.48	0.001
Agua	1	369	369	0.21	0.671
Excreta	1	13300	13300	7.58	0.051
Square	1	36934	36934	21.04	0.010
Temp.*Temp.	1	36934	36934	21.04	0.010
2-Way Interactions	3	6648	2216	1.26	0.400
Temp.*Agua	1	140	140	0.08	0.791
Temp.*Excreta	1	4028	4028	2.29	0.204
Agua*Excreta	1	2480	2480	1.41	0.300
Error	4	7021	1755		
Total	11	177440			

Coded Coefficients

Term	Coef	SE Coef	T-Value	P-Value	VIF
Constant	249.2	18.9	13.17	0.000	
Temp.	118.9	13.4	8.89	0.000	1.00
Agua	5.5	10.9	0.51	0.634	1.00
Excreta	33.3	10.9	3.05	0.029	1.00
Temp.*Temp.	-117.7	23.2	-5.08	0.004	1.00
Temp.*Excreta	22.4	13.4	1.68	0.154	1.00
Agua*Excreta	14.4	10.9	1.32	0.245	1.00

Model Summary

S	R-sq	R-sq(adj)	R-sq(pred)
37.8449	95.96%	91.12%	74.69%

Figure 8. Regression with a first reduction of terms.

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Model	6	170279	28380	19.82	0.002
Linear	3	126838	42279	29.52	0.001
Temp.	1	113169	113169	79.02	0.000
Agua	1	369	369	0.26	0.634
Excreta	1	13300	13300	9.29	0.029
Square	1	36934	36934	25.79	0.004
Temp.*Temp.	1	36934	36934	25.79	0.004
2-Way Interactions	2	6507	3254	2.27	0.199
Temp.*Excreta	1	4028	4028	2.81	0.154
Agua*Excreta	1	2480	2480	1.73	0.245
Error	5	7161	1432		
Total	11	177440			

carried out in later stages of the project.

According to the results obtained, a 3D biodigester was designed in the SolidWorks software as shown in Figure 12, which will be manufactured in the next stage, allowing tests to

be carried out on a pilot plant scale.

Conclusion

With the methodological process shown, the benefit achieved was that being an experimental scheme at

laboratory level, a pilot plant can be carried out being the basis for future research. The following impacts were achieved:

Scientific impact

- In the consulted literature, no previous works were found using cricket excreta as organic matter for the generation of biogas, thus revealing new knowledge.

- The feasibility of using the organic matter of the cricket excreta as an energy source for the generation of flammability at a laboratory scale was verified.

Technological impact

- The factors that intervene in the production of biogas were analyzed, based on the results obtained with the experimental designs, by establishing three temperature blocks, achieving reductions of



Figure 11. Flame generation.

TABLE II
TEMPERATURE CONTROL BLOCK

Test	Block	Water	Excrement	Water displacement
1	1	1	1	12.5
2	1	1	-1	4.0
3	1	-1	1	23.0
4	1	-1	-1	11.0
5	2	1	1	310.5
6	2	1	-1	222.0
7	2	-1	1	277.5
8	2	-1	-1	187.0
9	3	1	1	349.0
10	3	1	-1	160.0
11	3	-1	1	252.0
12	3	-1	-1	241.0

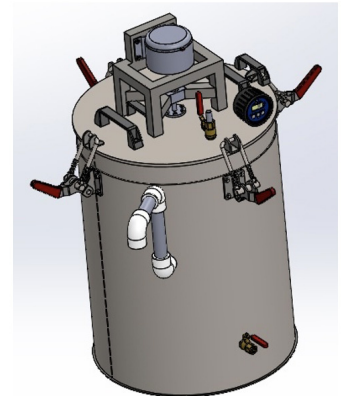


Figure 12. Biodigester design at pilot plant scale.

Coded Coefficients

Term	Coef	SE Coef	T-Value	P-Value	VIF
Constant	249.2	18.9	13.18	0.000	
Temp.	118.9	13.4	8.90	0.000	1.00
Excreta	33.3	10.9	3.05	0.019	1.00
Temp.*Temp.	-117.7	23.2	-5.08	0.001	1.00
Temp.*Excreta	22.4	13.4	1.68	0.137	1.00

Model Summary

S	R-sq	R-sq(adj)	R-sq(pred)
37.8142	94.36%	91.14%	81.87%

Figure 9. Regression with a second reduction of terms.

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Model	4	167431	41858	29.27	0.000
Linear	2	126469	63235	44.22	0.000
Temp.	1	113169	113169	79.14	0.000
Excreta	1	13300	13300	9.30	0.019
Square	1	36934	36934	25.83	0.001
Temp.*Temp.	1	36934	36934	25.83	0.001
2-Way Interactions	1	4028	4028	2.82	0.137
Temp.*Excreta	1	4028	4028	2.82	0.137
Error	7	10009	1430		
Total	11	177440			

terms to obtain the most significant factors: Cricket excreta and temperature.

- The predictive model is established to estimate the generation of biogas from the volume used in the formulations at the laboratory level.

- Confirmatory tests were performed with the same proportions established in the predictive model. Resulting in the ability to reproduce the amount of biogas generated.

- In Figure 5, the stability of the graph can be observed when stirring the formulation of organic matter during the generation of biogas.

Environmental impact

- Reduction of the pollution caused by cricket excreta, with a new disposal of such residues as a source of biogas generation (four sacks or eighty kilograms approximately are produced per week on a single farm).

Coded Coefficients

Term	Coef	SE Coef	T-Value	P-Value	VIF
Constant	249.2	20.9	11.90	0.000	
Temp.	118.9	14.8	8.03	0.000	1.00
Excreta	33.3	12.1	2.75	0.025	1.00
Temp.*Temp.	-117.7	25.7	-4.59	0.002	1.00

Model Summary

S	R-sq	R-sq(adj)	R-sq(pred)
41.8881	92.09%	89.12%	82.20%

Figure 10. Third reduction of terms.

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Model	3	163403	54468	31.04	0.000
Linear	2	126469	63235	36.04	0.000
Temp.	1	113169	113169	64.50	0.000
Excreta	1	13300	13300	7.58	0.025
Square	1	36934	36934	21.05	0.002
Temp.*Temp.	1	36934	36934	21.05	0.002
Error	8	14037	1755		
Total	11	177440			

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