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# Full 4<sup>2</sup> Factorial Experimental Design of Biogas Production from Cow Dung

Mohammed Alhassan, Noah Abdulmumeen, Mohammed Umar Garba, Abubakar G. Isah

**Abstract** – In this research work, a full 4<sup>2</sup> factorial experimental design procedure has been used to optimize some parameters in biogas production from cow dung in a laboratory size anaerobic digester. The effect of parameters such as production temperature, residence time and the yield of biogas have been investigated and the results showed that production temperature ( $X_1$ ), residence time ( $X_2$ ) and the interaction ( $X_1X_2$ ) have significant effect on the yield of bio gas ( $Y$ ). The full mathematical model developed which includes the two main effects and interaction and the reduced model are introduced in the paper. The optimizer plot suggested that to optimize  $Y$ , the experiment should be conducted at a temperature and residence time of 60°C and 5 days respectively. Copyright © 2016 Praise Worthy Prize S.r.l. - All rights reserved.

**Keywords:** Biogas, Renewable Energy, Cow Dung, Modeling, Experimental Design

## I. Introduction

Biogas as an alternative source of energy is renewable. The fact that petroleum is not renewable source of energy and being put at our disposal over a long period of time, it has been predicted that the present energy (petroleum) reserve would not last long.

This uncertainty has caused a lot of developing nations to look back to the past methods of using biomass (cow dung) as one of the most viable remedy with the purpose of improving it. The raw material for biogas includes most agricultural and other organic wastes.

In Nigeria, biogas can be produced from animal and human excreta, crop residue, poultry droppings, cow, pig and horse dung as well as sludge. In the production of biogas, the biomass are allowed to decompose anaerobically at room temperature, producing a gaseous product which contains methane ( $CH_4$ ), carbon dioxide, hydrogen sulphide, and some traces of nitrogen and hydrogen [1]- [3].

The anaerobic decomposition of complex organic molecules to methane and carbon dioxide is carried out by many different species of organisms (bacteria) acting together in the total absence of oxygen. The biological polymers, which are usually, present in biomass such as polysaccharides, protein and lipids must be broken down to simpler substances before they could be converted to methane [4]. The bacteria responsible for the conversion are present in faeces. The decomposition can be perceived to be in three stages.

The first stage is hydrolysis in which the insoluble carbon hydrates, proteins and oils are converted to soluble substances including sugar, and alcohols. In second stage, the soluble material is converted to fatty acids, esters, carbon dioxide and hydrogen.

In the final stage called “methanogenesis” the intermediate components are converted to methane [5], [3].

Once the Anaerobic digester is in operation, its continued performance can be measured by its gas production. If the gas production decreases, it is a sign that the microbial activities has been inhibited and this could be as a result of a change in temperature, a change in loading rate, a change in pH, the undigested part of animal waste or the presence of toxic materials [6], [7].

Temperature is a primary factor affecting growth rate of micro-organisms, the inappropriate selection of temperature range for any particular digestion will inhibit digestion [8] - [10]. The volume of gas produced per unit weight increases rapidly with increase in retention time up to threshold limit. A longer retention period would need bigger tanks. A treatment for toxicity is to dilute the peel material with other digestible materials such as hay, straw or urine. In most cases, urine is used and this increase the gas production, although excess of urine can bring about excess formation of ammonia and in turn reduction in the amount of biogas produced. Anaerobic digestion will occur best within a pH range of 6.8 – 8.0.

More discussion on the optimum pH for decomposition of biomass can be found in [6].

A decrease in pH is associated with an increase in volatile acid concentration and means the digester activity is imbalanced [7]. The addition of lime is recommended for a pH decrease and should bring the process back to normal [11].

Many works have been done on biogas production from various agricultural wastes, and review on such can be found in [3], [12], [13]. However, only limited work can be found on the optimization of technical parameters affecting biogas production from animal waste.

It is therefore the aim of this research work to optimize the technical parameters such as temperature and residence time for biogas production from cow dung using a full  $4^2$  factorial experimental design method.

Design of experiment is a structured and organized method that is used to determine the relationship between the different factors affecting a process and the output of that process. This method was first developed in the 1920s and 1930, by Sir Ronald A. Fisher, the renowned mathematician and geneticist [14].

Design of experiment involves designing a set of some numbers of experiment, in which all relevant factors are varied systematically, when the results of these experiment are analyzed, they help to identify optimal conditions, the factor, that must influence the results, and those that does not, as well as the existence of interaction and synergies between the factors. The idea is to vary all relevant factors simultaneously over a set of planned experiments and then connect the results by means of a mathematical model. This model is then used for interpretation, predictions and optimization of the process. Design of experiment requires only a small set of experiment and thus helps to reduce cost.

The development of the model is based on the results of experimental products and various combinations of the levels of each input parameters [14]. Although, the basic calculations can be done on paper, it is highly desirable and convenient to use more sophisticated computer packages, such as MINITAB 14, SPSS13, statistics, MODE 8.0 or Design expert.

## II. Experimental Procedure

### II.1. Design of Experiment

Before using MINITAB 14, all pre-experimental planning was conducted to determine the influence of process variables that affects the value of response variable (Y). A new design was created in MINITAB 14, and the factorial design worksheet created was applied to run the experiment and collect the response data which was then analyzed using analysis factorial design tab to fit a model in the experimental data.

The display plots were used to look at the design, the effects and to display main effects and interaction plots. To optimize the process response, response optimizer or overlaid contour plot were used to obtain a numerical and graphical analysis on the conditions that; the measure of the model consistency (p-value) is assumed to be at most 0.05 and hence, any of the main effects or interactions with p-value greater than 0.05 is rejected and not shown in the reduced model, and that all terms are free from a liaising, and their effects can be determined separately.

### II.2. Biogas Production

The laboratory scale digesters consisting of  $500\text{cm}^3$  conical flask were filled with cow dung used for the digestion. The flasks were corked to prevent inlet and

outlet of air, so as to serve as laboratory anaerobic digester. The delivery tube was connected from the digester to the gas collector (1 litre of empty bag).

The pH was maintained at 6.8 – 8.0 before the onset of biogas production. This pH range was selected based on the reported literature value for the optimum production of biogas from a related feed stock [6].

Each of the sample of biogas collected in the bag were weighed by weighing balance to know the amount of biogas produced based on factorial method used (Full  $4^2$  factorial).

This was done throughout for all the factors.

## III. Result and Discussion

When a design is screened, the objective is to select factors that have large effects on the output of the process (response). The factorial design was tested and the response data collected, a full mathematical model was fitted to the results and some plots were generated to evaluate the significance of the effects.

The output from fitting a full mathematical model was used, and normal probability plot of the residual and surface plot of production rate were used to determine which factors are important for improving the production rate of biogas from cow dung and the results are presented in Table I to Table IV, and in Fig. 1 and Fig. 2.

The experimental results and the factorial experimental design are shown in Table I, and it shows that the experimental consist of 4 levels and two factors i.e. temperature ( $^{\circ}\text{C}$ ) and residence time (days).

TABLE I  
THE FACTORIAL TEST ( $4^2$ ) AND EXPERIMENTAL RESULTS  
FOR THE BIOGAS PRODUCTION RATE

Run	Temperature ( $^{\circ}\text{C}$ )	Time (Days)	Biogas production rate
1	70	5	16.40
2	70	4	15.00
3	60	5	15.74
4	60	3	12.37
5	70	3	13.37
6	50	5	17.77
7	50	4	16.80
8	70	2	14.40
9	60	2	10.87
10	40	5	19.44
11	40	3	17.24
12	50	2	13.50
13	60	4	12.87
14	40	2	15.62
15	50	3	17.07
16	40	4	17.92

Temperature was tested at 4 levels i.e.  $70^{\circ}\text{C}$ ,  $60^{\circ}\text{C}$ ,  $50^{\circ}\text{C}$ , and  $40^{\circ}\text{C}$  while time was tested at 2, 3, 4, and 5 days. In total, 16 sets of experiment were conduct and the highest amount of bio gas was observed to be obtained at temperature of 40 and at residence time of 5days.

Table II shows the result of fitting a full mathematical model which includes 2 linear main effects, 2 square effects and one interaction. The values in the p column were used to determine which of the effects are

significant. Using  $p = 0.05$ , the main linear values of temperature, time and temperature square were found to be significant; that is, their p-values were less than 0.05.

The square, time and their interaction values were found to have no significant effects on the production rate, which indicates that p-value are higher than 0.05. Reference [15] reported a similar observation.

The full model equation can therefore be represented as follows:

$$Y = 14.3441 - 1.7415X_1 + 1.7782X_2 + 1.7438X_1^2 + 0.1457X_2^2 - 0.2466X_1X_2 \quad (1)$$

The significant P-value or p-value is 0.05, any value greater than this is regarded as insignificant and can be done away with. This implies that the reduced first order regression equation can be re-written thus:

$$Y = 14.3441 - 1.7415X_1 + 1.7782X_2 + 1.7438X_1^2 \quad (2)$$

The reduce equation indicated above illustrates the coefficient of the full regression equation and their numerical significance. The equation illustrates the actual impact of each model term with positive and negative coefficients which have a synergistic and antagonistic effect on the biogas production respectively.

Terms that are synergistic are said to contribute to the overall biogas production when increased while terms that are antagonistic work against biogas production when increased.

Hydraulic retention time has a synergistic effect on the response equation while temperature has an antagonistic effect on the conversion.

TABLE II  
ESTIMATED REGRESSION COEFFICIENTS FOR PRODUCTION RATE (CODED UNITS)

Term	<sup>1</sup> Coef	<sup>2</sup> SE Coef	<sup>3</sup> t	<sup>4</sup> p
Constant	14.3441	0.6722	21.338	0.000
Temperature	-1.7415	0.4441	-3.922	0.003
Time	1.7782	0.4441	4.004	0.003
Temperature × temperature	1.7438	0.7447	2.341	0.041
Time × Time	0.1547	0.7447	0.208	0.840
Temperature × Time	-0.2466	0.5958	-0.414	0.688

<sup>1</sup>Coefficient

<sup>2</sup>Coefficient of the square effects

<sup>3</sup>Student T-test

<sup>4</sup>Probability Value

Table III shows the Analysis of variance for production rate which indicates the p-values of Regression and the linear terms to be less than 0.005, meaning that they have significant effects on the production, while square and interaction are insignificant as their P-values are greater than 0.05. The statistical significance of the model equation checked by F-test and P-value implies the model is highly significant.

Table IV shows the statistical precision parameters of the full model, in which the R-square value obtained was 78.8% which is high, suggesting that the relationship between the biogas production parameters (temperature, time) and the response is approximately linear.

TABLE III  
ESTIMATED REGRESSION COEFFICIENTS FOR PRODUCTION RATE (CODED UNITS)

Source	DF <sup>a</sup>	Seq SS <sup>b</sup>	SS <sup>c</sup>	Adj MS <sup>d</sup>	F-value
Regression	5	65.0526	65.0526	13.0105	7.42
Linear	2	55.0666	55.0666	27.5333	15.71
Square	2	9.6856	9.6856	4.8428	2.76
Interaction	1	0.3003	0.3003	0.3003	0.17
Residual Error	10	17.5284	17.5284	1.7528	
<b>Total</b>	<b>15</b>	<b>82.5810</b>			

a: Degree of freedom, b: sum of square, c: Adjusted sum of squares, d: adjusted square mean, F: F-test, P: probability value

TABLE IV  
THE VALUES OF THE STATISTICAL PRECISION PARAMETERS OF THE FULL MODEL

Statistical Parameters	Statistical values
S	1.324
R - Sq	78.8%
R - Sq (adjusted)	68.2%

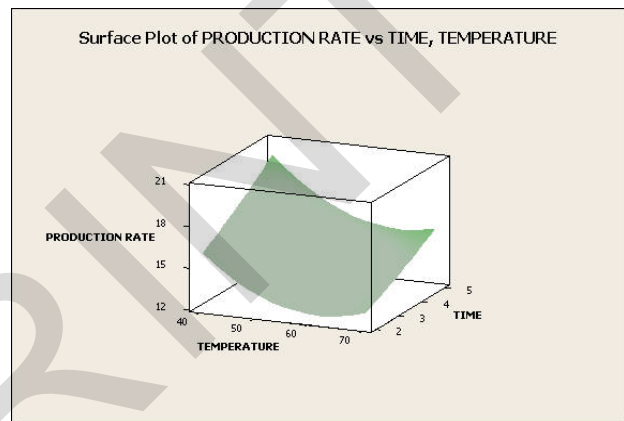


Fig. 1. Surface plot of production rate versus time, temperature

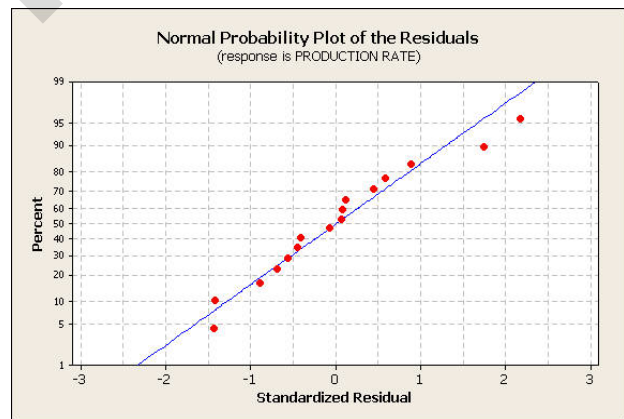


Fig. 2. Normal probability plot of the residuals

The adjusted R-square of 68.2% implies that only 68.2% of the variability in the response could be captured and explained by the full model [16].

Fig. 1 shows the surface plot of production rate versus time, temperature. This plot shows that the optimum yield of biogas for this research is at the temperature of 40 and residence time of 5 days. This observation confirm that the methanogenic bacterial that are believed to aid methane generation strive very well at temperature below 40°C. Reference [17] reported a similar

observation.

Fig. 2 shows the probability plot for the set of data to ascertain whether or not the data set is approximately normally distributed. For the biogas yield data, in Table I, residuals appeared to roughly follow a straight line indicating it is normal. This shows no evidence of non normality or unidentified variable exists.

This result is in agreement with the null hypothesis that residuals are normally distributed on a straight line (Mandel, 1984).

#### IV. Conclusion

A full  $4^2$  factorial experimental design of production of Biogas from cow dung has been carried out. The effects of factors such as temperature and time have been investigated. Analysis using factorial design experiment showed that the approximate model equation for production rate is:

$$Y = 14.3441 - 1.7415X_1 + 1.7782X_2 + 1.7438X_1^2 + 0.1457X_2^2 - 0.2466X_1X_2$$

and the reduced model is:

$$Y = 14.3441 - 1.7415X_1 + 1.7782X_2 + 1.7438X_1^2$$

Using surface plot of production rate versus time and temperature suggested that to obtain high production, the experiment should be conducted at a temperature of 40°C and at the retention time of 5 days.

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