

# **Optimization of biogas production from cassava peels mixed with urea by Central Composite Design methodology**

## **ABSTRACT**

The present study aims to know the optimal conditions for biogas production from biomethanization of cassava peels mixed with urea in mesophilic conditions during 14 days in sixteen reactors of 1 Litre capacity, using response surface methodology (RSM). Two parameters were studied, organic loading rate (OLR) and urea concentration (UC). The variations used for OLR was 5% and 15% TS and for UC, it was 0.01 % and 0.05 %. The variations obtained inside were determined by Central Composite Design (CCD) made from rsm package of R software 4.1.1. Based on experimental results analysis, it was found that urea concentration affect biogas production ( $P < 0.05$ ), the optimum value of Organic Loading Rate and urea concentration were 6.688%TS and 0.0067% respectively, with a optimal biogas yield of 3260.694 mL. The analysis of variance (ANOVA) showed a high coefficient of determination value ( $R^2 = 0.8146$ ) at 95% confidence level and a p-value of 0.002. The results show that urea concentration has a major impact on biogas production ( $P < 0.05$ ).

**Keywords:** *Optimization, biogas, cassava peels, urea, central composite design.*

## **1. INTRODUCTION**

Global environmental pollution which is increasing due to the human activities has led delegates, Heads of State and Government who participated in COP26 conference to reflect about on the severity of climate crisis that facing the world and to live up historical responsibility of setting the world on the path to address this existential challenge. They left Glasgow with a global compromise and agreement to work on reducing the persistent gap and keeping the temperature average to 1.5 degrees. Parties were encouraged to strengthen their emissions reductions and to align their national climate action pledges with the Paris Agreement [1].

Lignocellulosic solid wastes increase proportionally with the growth of world population. Unfortunately, they are indiscriminately discharged into the environment and amassed at waste dumps. The unpleasant odor, leachate and methane generated from disposal wastes enhance the risk of pollution to the environment which is dangerous for the living being and water resources [2].

To avoid the pollution rise according to Glasgow philosophy, organic solid wastes could be valued by producing fuel as biogas, bioethanol and briquette [3-6]. Cassava tubers as a main food stuff in sub-saharan Africa generate in peeling step, enough wastes which represent 1/3 per Kg of tubers, especially the peels [2]. Several researchers reported the possibility to value them by producing biogas [7] and bioethanol [5, 8].

We investigated biogas production from cassava peels mixed with urea at various concentrations. The highest result (80.79L/KgTS) were obtained only with 0.01% of urea [9]. Attempts have been undertaken to investigate the factors affecting that high biogas rate from cassava peels with urea. Therefore, fractional factorial design were used to study the four factors (initial pH, Organic loading rate, particle size and co-substrate type) assumed to affect biogas rate as obtained previously. It was seen that three factors: organic loading rate, particle size and co-substrate type were found to be statistically most significant ( $p < 0.05$ ), followed by two interactions: initial pH-organic loading rate and organic loading rate-particle size [2]. Based on our previous works, there is a need to optimize the biogas production from cassava peels mixed with urea under mesophilic conditions by using response surface methodology (RSM) with the variables as organic loading rate and urea concentration. This study aims to determine the optimum operating conditions from that two independent variables (organic loading rate and urea concentration) to optimize biogas yield.

## 2. MATERIAL AND METHODS

### 2.1. Material

Cassava peels used as substrate were collected from Réserve Stratégique Générale field at Bateke's plateau/Kinshasa province. Urea was brought at triangle market/Lemba area, Kinshasa, DR-Congo. After collecting, peels were pretreated and experiments were carried out under mesophilic conditions in 1L plastic bottle digester with 750mL working volume, as described previously [9]. The total and volatile solids, total organic carbon and ash of the feedstock and inoculum were pre-determined before their loading in biodigester.

### 2.2. Design of experiments

Response surface methodology (RSM) with central composite design (CCD) was used to optimize the biogas production from cassava peels mixed with urea. Two independent parameters were evaluated in this study: Organic loading rate ( $x_1$ ) and urea concentration ( $x_2$ ), therefore the dependent variable was biogas yield ( $Y$ ) as a cumulative volume of biogas produced during the total retention time. The range and levels of the independent variables for biogas process are shown in Table 1. Therefore, 16 sets of experiments were generated including the  $2^2$  factorial experiments, 4 axial points and 8 replicates of centre points. R software (version 4.1.1) by rsm package was used to get the matrix of experiments with two factors (Table 1). Each factor was tested on five levels, the highest level was coded as +1, the centre point was coded as 0, and the lowest level was coded as -1. The outer design space points corresponding to  $\alpha$  were  $\pm 1.414$ .  $\alpha = 2^{k/4}$ , where  $k$  is the number of factor. In this case,  $k = 2$ . The response surface model was fitted to the response variable, namely biogas yield (mL). The second degree polynomial function is given by equation 1:

$$Y = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \beta_{1.2} x_1 x_2 + \beta_{1.1} x_1^2 + \beta_{2.2} x_2^2 + \varepsilon \quad (\text{Eq.1})$$

Where  $Y$  is the measured response or dependent variable,  $\beta_0$  is the intercept,  $\beta_1$  and  $\beta_2$  are linear coefficients while  $\beta_{12}$  is the interaction coefficient,  $\beta_{11}$  and  $\beta_{22}$  are squared coefficients and  $x_1$ ,  $x_2$ ,  $x_{12}$ ,  $x_1^2$  and  $x_2^2$  are the levels of independent variables (Khaled, 2015). Table 1, gives the experimental range and levels of the independent variables used in the CCD.

**Table 1. Experimental matrix of experiments**

Independent variable	Code Unit	Range and levels				
		Low level star point ( $\alpha = -1.414$ )	Low level factorial (-1)	Central point (0)	High level factorial (+1)	High level star point ( $+\alpha$ )

Organic loading rate	%	$x_1$	2.9289	5	10	15	17.0711
Urea concentration	%	$x_2$	0.0017	0.01	0.03	0.05	0.0583

### 2.3. Biogas production

A set of sixteen batch reactors labeled JC1-JC16 in duplicate were used to produce biogas from cassava peels mixed with urea. In each digester, the amount of peels to mix with a known concentration of urea, where done by following requirements of Table 2. After loading the digesters, insuring a neutral initial pH and airtighted, digesters were shaken manually twice daily morning and evening. The volume of biogas produced was measured by the quantity of water displaced. In each bottle, a specific amount of distilled water, substrate and inoculum were put and sealed with a plastic stopper and kept for digestion. The lid of bottle used as digester was drilled in the middle to serve as an outlet for biogas which was led to another 330 mL plastic bottle used as gas storage filled with caustic soda aqueous solution 0.01M to purify biogas obtained. All perforations were properly sealed and the two bottles connected with plastic tubing. Table 3 shows details of 16 digesters loaded and responses obtained.

### 2.4. Statistical analysis

All measurements of substrate characterization, were used to obtain the mean and standard deviation. The one-way Analysis of variance (ANOVA) was used to evaluate the adequacy of the model. The F-test (ANOVA) was exploited to find the statistical significance of the second-order polynomial model (Eq. 2). The rsm package of R 4.1.1 Software was used to analyze experimental data and to calculate the predicted response by using Eq.1. The predicted and experimental responses are shown in 2-D or 3-D plots. The significance evaluation between the mean of yield of biogas in different experimental runs were statistically defined.

## 3. RESULTS AND DISCUSSION

### 3.1. Physico-chemical characterization of feedstock used

Table 2 reports results of physico-chemical characterization of cassava peels used.

**Table 2. Characterization of cassava peels used as substrate**

	Parameter	Value (%)
1	Moisture	$8.9 \pm 0.14$
2	Total solids	$91.1 \pm 0.14$
3	Volatile solids	$96.75 \pm 0.13$
4	Total Organic Carbon	$55.60 \pm 0.13$
5	Ash	$3.25 \pm 0.13$

The results of table 2 show that cassava peels are good raw matter for producing biogas regarding its volatile solids (96.75%) and ash content (3.25%) which are the organic matters from which biogas is produced [3, 9]. It has been found that cassava peels used contains mineral elements which are necessary for the living and growing of microorganisms [2]. But, their excess concentration could have an inhibitory effect on methanogen bacteria [10]. The ash content of peels used in this study was a little different than our previous studies [2, 9] The variation of chemical content could be explained by various factors as origin, age and the period of harvest [2].

### 3.2. Optimization of biogas production with ccd

Table 3 gives ccd matrix and responses obtained for each run.

**Table 3. Ccd matrix for two variables with responses obtained**

Run	OLR(%TS)	UC (%)	Y (mL)
1	10	0.03	1720
2	15	0.01	2082
3	5	0.05	0
4	10	0.03	2692
5	10	0.03	2866
6	5	0.01	4041
7	15	0.05	230
8	10	0.03	2971
9	2.9289	0.03	880
10	10	0.03	2293
11	10	0.03	2876
12	10	0.0017	2402
13	10	0.03	2606
14	10	0.03	2782
15	17.0711	0.03	2360
16	10	0.0583	0

The result of Table 3 show that biogas yield of each run was different. The highest biogas produced was in reactor 6 with the yield of 4041mL (107.76L/KgTS) under the following conditions: 5%TS and 0.01% urea concentration. The result of biogas production in Table 3 could be inputted for calculation with RSM in R software 4.1.1 which then resulted in equation 2.

### 3.3. Statistical analysis

Tables 3 and 4 give details about regression model and ANOVA results of experimental result analysis.

**Table 4. Regression analysis for the production of biogas**

	Estimate	Std.Error	t value	Pr(> t )	
(Intercept)	2600.750	219.900	11.8270	3.349e-07	***
$x_1$	45.505	219.900	0.2069	0.8402152	
$x_2$	-1159.828	219.900	-5.2743	0.0003606	***
$x_1 \cdot x_2$	547.250	310.985	1.7597	0.1089515	
$x_1^2$	-446.437	219.900	-2.0302	0.0697830	.
$x_2^2$	-653.938	219.900	-2.9738	0.0139549	*

Signif. codes: 0 '\*\*\*' 0.001 '\*\*' 0.01 '\*' 0.05 '.' 0.1 ' ' 1  
 Multiple R-squared: 0.8146, Adjusted R-squared: 0.7218  
 F-statistic: 8.785 on 5 and 10 DF, P-value: 0.002

From Table 3,  $x_1$  is the organic loading rate and  $x_2$  is the urea concentration. The results show that the model used and urea with its quadratic coefficients are all significant ( $p < 0.05$ ). The  $R^2$  value obtained in this study was 0.8146 and the p-value was 0.002, imply that the model is significant. Mohd et al., [11] and Ibrahim et al., [12] showed that  $R^2$  value of at least 0.6 is acceptable while more than 0.70 is considered accurate and satisfactory. Therefore, the  $R^2$  value obtained in this study was higher than 0.70 indicating that the regression model explains at 81.46%, the response variations observed.  $R^2$  (0.8146) and  $R^2$ -adjusted (0.7218) values

was acceptably closed to 1. It shows that the model used describes the relationship between dependent variable (response) and the inputs (independent variables) and could help to determine optimum parameter. The model employed for this process based on the results of Table 3, could be expressed as:

$$Y = 2600.750 + 45.505x_1 - 1159.828x_2 + 547.250x_1x_2 - 446.437x_1^2 - 653.938x_2^2 \quad (\text{Eq.2})$$

Where Y is the predicted response,  $x_1$  is the organic loading rate (%TS) and  $x_2$  is the urea concentration (%). The positive coefficient of  $x_1$  (organic loading rate) means that the total solids will linearly increase the biogas production while the negative coefficient of  $x_2$  means that biogas yield subside when urea concentration increases.

The results of Anova second order model data are shown in Table 5.

**Table 5. Analysis of Variance Table**

Response: y	Df	Sum Sq	Mean Sq	F value	Pr(>F)	
FO( $x_1, x_2$ )	2	10778181	5389090	13.9308	0.001285	
TWI( $x_1, x_2$ )	1	1197930	1197930	3.0966	0.108952	
PQ( $x_1, x_2$ )	2	5015526	2507763	6.4826	0.015655	
Residuals	10	3868477	386848			
Lack of fit	3	2673636	891212	5.2212	0.033211	significant
Pure error	7	1194841	170692			

By employing ANOVA multiple comparison test as summarized in Table 4, it has been shown that first model and pure quadratic term are significant because their *P*-values were less than 0.05. In fact, *p*-value less than 0.05 express that the term is statistically significant otherwise it is not statistically significant [13]. Furthermore, a significant lack of fit obtained in Table 4, indicates that the variation of the replicates about the mean values is less than the variation of the design points about the predicted values. In other word, the runs replicate well, and the variance is small [11]. The lack of fit F-value of 5.2212 implies that the lack of fit is significant. There is only 3.3211% chance that this large "Lack of Fit F-value" could occur due to noise [14]. This means that there was some constraints which was neglected especially urea concentration. In fact, beyond the concentration of 0.03%, the biogas rate decrease sensibly which led to the high deviation between predicted and experimental values of biogas yield. Thereby, it was advisable to reduce the high value of urea concentration to 0.03% and add other factors such as hydraulic retention time and temperature to increase the adequate precision of the model. Abubakar et al., [12] showed that a little change in the parameters might affect the fit of models and Choo et al., [15] evoked that adding more factors could make the lack of fit become desirably insignificant.

### 3.4. Stationary points

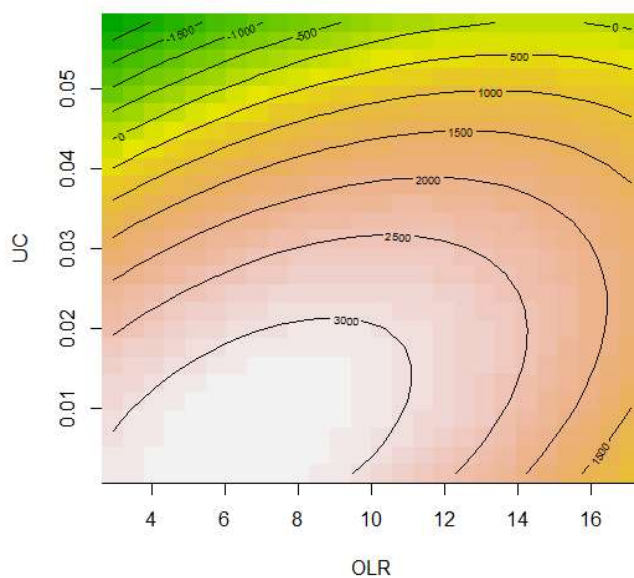
Furthermore, to find the levels of variables  $x_1$  and  $x_2$  which optimize the predicted response, partial derivatives were applied to regression model (Eq.1) equal to zero [16]. By replacing regression coefficients, mathematical equations are obtained and resolved to determine the values of variables corresponding to stationary points. Table 5 gives the stationary point of surface response in coded and original values.

**Table 6. Stationary point of surface response**

Variable	Optimal value (with coding)	Optimal value (in original)
$x_1$	-0.6624565	6.687717702

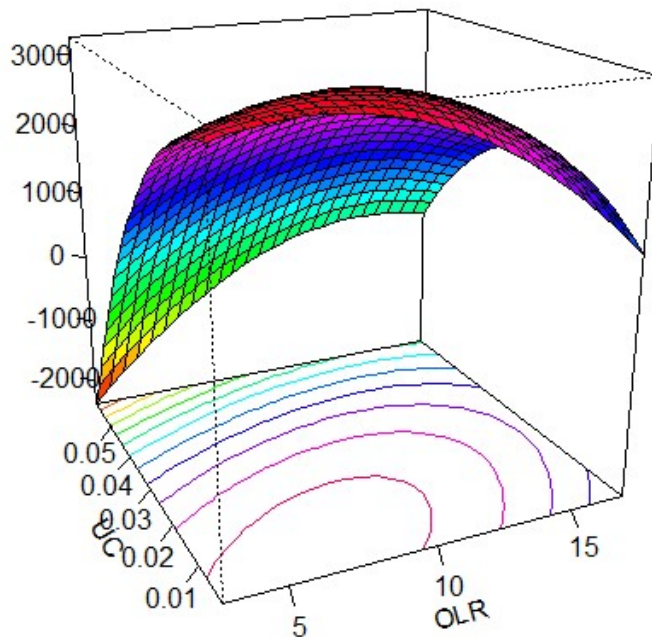
Where  $x_1$ = organic loading rate (%) and  $x_2$ = urea concentration (%). To get the surface of the response, eigen values ( $\lambda$ ) were calculated from the Hessian matrix obtained. The eigen values are given below:  $\lambda = [-257.5534 \ -842.8216]$ . As all two eigen values are negatives, it was concluded that the surface shape is the maximum response. The contour and 3D plots from pair of two factors used (figures 1 and 2) could be constructed. Based on data analysis with response surface method, it is concluded that the peak flow will reach maximum value of organic loading rate with 6.6877 %TS and urea concentration of 0.0067 % with a maximum biogas yield of 3260.694 mL.

Figures 1 and 2 show graphical representations of the response surface which help to see the effects of organic loading rate and urea concentration, on biogas production from cassava peels. Figure 1 shows the contour plot which is a 2D surface plot. It determines the interaction of the independent parameters and optimum value of each component for maximum response. The two plots (Fig.1 and 2) were obtained from rsm package of R software 4.1.1.



**Figure 1. Contour plot**

Figure 1 shows the area of maximum biogas yield located from 3 to 11 %TS (organic loading rate) and from 0 to 0.02% of urea. It is evident that, increasing urea concentration and organic loading rate beyond 0.02% and 10%TS respectively, would decrease biogas yield. According to our previous studies, the highest biogas yield obtained was 3030mL (80.79LTS) at 0.01% of urea and 5%TS, but particle size was not specified [9]. Later as we showed, the small particle size (<2mm) was appropriate to enhance biogas yield because of their easy degradation by methanogen bacteria [2]. From the present study, it has been seen that in the same conditions as previously [9], with the small particle size (<2mm), the maximum biogas yield obtained was 4041 mL (107.76L/Kg Ts). We concluded that the process is optimized and the highest biogas yield obtained by Nkodi et al., [9] could be justified that being in the area of maximum biogas yield. The figure 2 shows the 3D plot of the contour plot.



**Figure 2. Surface plot**

#### **4. CONCLUSION**

The present study focuses on optimization of independent variables used (organic loading rate and urea concentration). The response surface methodology especially central composite design was used to determine the optimum condition for biogas production from cassava peels mixed with urea in mesophilic conditions in a batch reactor. The analysis of regression model and ANOVA showed that urea concentration and quadratic term was significant ( $P < 0.05$ ). Quadratic model was used to predict all the responses. The optimal conditions determined were organic loading rate of 6.6878 %TS and urea concentration of 0.0067% with a biogas yield of 3260.694 mL. The coefficient of determination was 0.8146, thus it is suggested that the model obtained could be used to optimize biogas production from cassava peels mixed with urea.

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