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Optimization of Process Parameters Affecting Biogas Production from Organic Fraction of Municipal Solid Waste via Anaerobic Digestion

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***Abstract*—**The aim of this study was to obtain the optimal conditions for biogas production from anaerobic digestion of organic fraction of municipal solid waste (OFMSW) using response surface methodology (RSM). The parameters studied were initial pH, substrate concentration and total organic carbon (TOC). The experimental results showed that the linear model terms of initial pH and substrate concentration and the quadratic model terms of the substrate concentration and TOC had significant individual effect ($p < 0.05$) on biogas yield. However, there was no interactive effect between these variables ($p > 0.05$). The highest level of biogas produced was 53.4 L/Kg VS at optimum pH, substrate concentration and total organic carbon of 6.5, 99gTS/L and 20.32 g/L respectively.

***Keywords*—**Anaerobic Digestion, Biogas, Optimization, Response Surface Methodology.

I. Introduction

MUNICIPAL solid waste (MSW) generation is significantly increasing in Indian urban areas and started creating enormous waste disposal problems in the recent past [1]. In India, MSW management is the duty of the local municipalities [2].More than 90 percent of the municipal solid waste (MSW) generated in India is dumped in an unsatisfactory way, what creates environmental hazards to water, air and land. At the same time the organic fraction of MSW is about 40-60 percent [3]. In Kerala, around 70% of the waste is compostable organics enabling high level of recycling in the form of manure or fuel [4], [5]. The anaerobic digestion is an attractive option for energy generation from the putrescible fraction of MSW as well as for reducing the disposal problem [6]. It has reduced environmental impact, especially with respect to the greenhouse effect and global warming.

Anaerobic digestion is a biological process wherein diverse group of microorganism convert the complex organic matter into a simple and stable end products in the absence of oxygen [7]. This process is very attractive because it yields biogas, a mixture of methane and carbon dioxide which can be used as renewable energy resources. AD of organic fraction of municipal solid waste (OFMSW) is used in different regions worldwide to reduce the amount of material being landfilled, stabilize organic material before disposal in order to reduce future environmental impacts from air and water emissions and recover energy. Several research groups have developed anaerobic digestion processes using different organic substrates [8]-[10]. In this view, anaerobic digestion of solid waste is a process that is rapidly gaining momentum to new advances especially in the area of dry anaerobic fermentation and has become a major focus of interest in waste management throughout the world. It appears to be the reliable and promising process for the treatment of organic solid waste. Moreover, when compared to other conversion technologies for treatment of the organic fraction of MSW, the economic, energy, and environmental benefits makes anaerobic digestion an attractive option [11].

In anaerobic digestion environmental factors such as substrate concentration, temperature, pH and metal ions [12] have great influences on methane production. A high concentration of VFAs has been reported to inhibit methane production from VFAs by mixed anaerobic microorganisms [13]. The optimum pH range for anaerobic digestion producing methane is 6.8–7.2 [14]. The growth rate of methanogens can be greatly reduced when the pH value is less than 6.6 [15]. An excessively alkaline pH can lead to the disintegration of microbial granules and subsequent failure of the digestion process [16]. Therefore, a buffer is needed in the methane production process in order to provide the resistance to significant and rapid pH changes in the system.

Optimization of various process factors affecting biogas production is a complex process with a number of interactive controlling parameters. At industrial level, even a small improvement in the process, gives a better yield which may be beneficial commercially, making process optimization a major area of research in the field of industrial biotechnology [16]. Therefore, there is a need for optimization of accurate process parameters which, improves the production of the biogas significantly.

In this study, optimization of process controlling factors was done by Response Surface Methodology (RSM). RSM is a statistical technique for analyzing the effects of several independent variables on the responses [17]. RSM is important in process design and optimization as well as for improving the performance of the system. Therefore, the aim of this study was to investigate the effects of initial pH, substrate concentration and total organic carbon as well as their

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interactive effects on biogas production from organic fraction of municipal solid waste using RSM.

II. Materials and Methods

A. Experimental Reactors

The experiments were carried on batch laboratory scale reactor (aspirator bottle) with total capacity of 2 L. The reactor was made of borosilicate glass with bottom sampling outlet. The bottles were closed by rubber stoppers equipped with glass tubes for gas removal and for adjusting the pH. The glass tube was dipped inside the slurry to avoid gas loss during the pH adjustments. The effective volume of the reactor was maintained at 1.6L.Biogas production from the reactors was monitored daily by water displacement method. The volume of water displaced from the bottle was equivalent to the volume of gas generated. The reactor was mixed manually by means of shaking and swirling once in a day. The reactors were operated at room temperature.

The inoculum used in this study was fresh cattle dung which contains all the required microbes essential for anaerobic digestion process. The pH, total solid and volatile solid of the inoculum were 6.5, 24.2% and 85.4% respectively. The percentage of inoculum for acidogenic fermentation of the organic waste is approximately 30% of the working volume. The inoculum was collected and kept at 4°C until used.

B. Design of Experiments with Response Surface Methodology (RSM)

Design of experiment (DOE) is a well-accepted statistical technique able to design and optimize the experimental process that involves choosing the optimal experimental design and estimate the effect of the several variables independently and also the interactions simultaneously. Response surface methodology (RSM) is a statistical method used for experimental modeling and analyzing the relationship between the input and response variables [18]-[20]. In this study three process variables viz. initial pH, substrate concentration and TOC were selected to study the effect on biogas production. The full experimental plan with respect to their actual and coded forms is listed in Tables I & II.

Analysis of variance (ANOVA) was used for analysis of regression coefficient, prediction equations, and case statistics. The experimental results of RSM were fitted using the following second order polynomial equation:

$$Y= \beta o+\Sigma\ \beta iXi+\Sigma\ \beta iiXi2+\Sigma\ \beta ijXiXj$$

In this polynomial equation, Y is the predicted, Xi, Xj are independent variables, βo is the intercept term, βi is the linear coefficient, βii is the quadratic coefficient, and βij is the interaction coefficient.

In this study, the independent variables were coded as A, B and C. Thus, the second order polynomial equation can be represented as –

$$Y = \beta o + \beta_1 A + \beta_2 A + \beta_3 A +\beta_{11} A^2 + \beta_{22} B^2 + \beta_{33} C^2 + \beta_{12} AB + \beta_{23} BC + \beta_{13} AC$$

Diagnostics Plots and model graphs were obtained using the Stat-Ease software with Design Expert v.8 to analyze the effects of variables individually and their interactions to determine their optimum level. The point prediction method was used for optimization of the levels of each variable for maximum response.

III. Results and Discussion

A. Optimization of Biogas Production

The objective of this study was to investigate the effects of initial pH, substrate concentration and total organic carbon on biogas production from OFMSW. The optimal levels for the independent variables and the effect of their interaction on biogas production were further explored using the central composite design of RSM. By applying multiple regression analysis on the experimental data, the second-order polynomial equation (1) was derived to explain the biogas production.

$$\text{Biogas} =7343.15+96.03A -759.30B +88.78 C +69.28 AB +81.78AC - 41.76BC - 26.52 A^2 - 3241.51 B^2 - 209.95C^2 \quad (1)$$

where, A, B, C are the coded values for initial pH, substrate concentration and total organic carbon, respectively. Table I shows the coded values of independent variables experimental ranges. The full experimental plan of CCD design for studying the effects of three independent variables, viz. initial pH (A), substrate concentration (B) and TOC (C) are listed in Table II.

The statistical significance of the second-order polynomial equation was checked by an F-test (ANOVA). All the corresponding data are shown in Table III. The Model F-value of 445.8 implies the model is significant. There is only a 0.01% chance that a "Model F-Value" this large could occur due to noise. In addition, the ANOVA of the quadratic regression model demonstrated that the model was highly significant ($p<0.05$) (Table III). The linear model terms of initial pH (A) and substrate concentration (B) and the quadratic model terms of the substrate concentration (B^2) and TOC (C^2) were significant ($p<0.05$), indicating that these two variables had an individual effect on biogas yield. However, the linear model terms of substrate concentration was insignificant ($p>0.05$), suggesting that there was no linear effect of this variable on biogas yield. The interactive effects for all of these factors were found to be insignificant ($p>0.05$) (Table III).Additionally, the experimental biogas production was close to the predicted value using (1) (Fig. 1).

For biogas production, the correlation coefficient (R^2) of polynomial equation was found as 0.9975. The R^2 value indicated a measure of variability in the observed response values which could be described by the independent factors and their interactions over the range of the corresponding factor. This implied that the sample variation of 99.75% of the total variation could be explained by the model and only 0.25% of it was not explained by the model. So, quadratic model was chosen for this analytical work.

TABLE I
CODED VALUE OF INDEPENDENT VARIABLES AND EXPERIMENTAL RANGES

Factor	Name	Coded lower limit	Coded Higher limit	Real lower limit	Real higher limit
A	Initial pH	-1	+1	6.00	7.00
B	Substrate Concentration(TS-g/L)	-1	+1	83.00	115.00
C	TOC(g/L)	-1	+1	16.76	23.87

TABLE II
CCD MATRIX FOR THREE VARIABLES WITH ACTUAL BIOGAS PRODUCTION

Run	Initial pH A	Substrate (TS-g/L) B	TOC(g/L) C	Biogas production(mL)	Biogas yield (L/Kg.VS)
1	6.50	99.00	20.32	7400	53.8
2	7.00	83.00	16.76	4500	37.8
3	7.00	115.00	16.76	3150	20.1
4	7.34	99.00	20.32	7450	54.2
5	6.50	115.00	20.32	3200	20.4
6	7.00	115.00	23.87	3400	21.6
7	6.00	83.00	16.76	4600	38.7
8	6.50	83.00	20.32	4800	40.3
9	6.50	99.00	20.32	7415	53.9
10	6.50	83.00	20.32	4820	40.5
11	6.00	115.00	16.76	3100	19.7
12	6.50	99.00	16.76	800	49.5
13	5.66	99.00	20.32	7000	50.9
14	6.00	83.00	23.87	4700	39.5
15	6.50	99.00	20.32	7380	53.7
16	6.50	99.00	20.32	7400	53.8
17	6.50	99.00	20.32	7415	53.9
18	6.50	99.00	20.32	7380	53.7
19	7.00	83.00	23.87	4800	40.3
20	7.00	115.00	23.87	3410	21.7

TABLE III
REGRESSION ANALYSIS FOR THE PRODUCTION OF BIOGAS FOR QUADRATIC RESPONSE SURFACE MODEL FITTING (ANOVA)

Source	Sum of Squares	Degree of freedom	Mean Square	F Value	p-value Prob> F	
Model*	5.717E+007	9	6.496E+006	445.80	< 0.0001	Significant
A-Initial pH	*5.846E+007*	*1*	*93395.21*	*6.41*	*0.0298*	
B-Substrate Concentration(TS)	*93395.21*	*1*	*4.736E+006*	*324.99*	*< 0.0001*	
C-TOC	*4.736E+006*	*1*	*47296.67*	*3.25*	*0.1018*	
AB	*47296.67*	*1*	*24097.24*	*1.65*	*0.2274*	
AC	*24097.24*	*1*	*33577.45*	*2.30*	*0.1600*	
BC	*33577.45*	*1*	*9899.15*	*0.68*	*0.4290*	
A2	*9899.15*	*1*	*9608.27*	*0.66*	*0.4357*	
B2	*9608.27*	*1*	*3.030E+007*	*2079.35*	*< 0.0001*	
C2	*3.030E+007*	*1*	*1.111E+005*	*7.62*	*0.0201*	
Residual	1.457E+005	10	14571.16			
Lack of Fit	*1.444E+005*	*4*	*36107.07*	*168.81*	*< 0.0001*	*Significant*
Pure Error	*1283.33*	*6*	*213.89*			
Cor Total	5.861E+007	19				

* SD=120.71; Mean=5506.00; R-Square=0.9975; Adj R-Squared=0.9953; C.V.%=2.19; Pred R-Squared=0.9169; PRESS=4.870E+006; Adeq Precision =52.185

“Adeq Precision" measures the signal to noise ratio. A ratio greater than 4 is desirable. The ratio found here 52.185 which indicated an adequate signal for this study. This model was used to navigate the design space. The adjusted R^2(0.9953) was also very high, which indicated the higher significance of the model. The "Pred R-Squared" value of 0.9169 showed the reasonable agreement with the "Adj R-Squared" value of 0.9953. This indicated a good agreement between the observed and the predicted values. The percentage of coefficient of variation (CV %) is a measure of residual variation of the data relative to the size of the mean. Usually, the higher the value of CV, the lower is the reliability of experiment. Here a lower value of CV (2.19%) indicated a greater reliability of the experiment. The Predicted Residual Sum of Squares (PRESS) was a measure of how well the model fitted each point in the design. The smaller the PRESS statistics, better would be the model fitting the data points. Here the value of PRESS found as 4.870E+006.Moreover the "Lack of Fit F-value" of 168.81 implies the Lack of Fit is significant. There is only a 0.01% chance that a "Lack of Fit F-value" this large could occur due to noise. The model showed standard deviation and mean values of 120.71 and 5506.00, respectively.

Graphical representations of the response surface are shown in Figs. 2 and 3; to view the effects of initial pH, Substrate concentration and TOC on biogas production. Fig. 2

represents the 3D surface plot. The contour plots (Fig. 3) determined the interaction of the process parameters and optimum value of each component for maximum response. Those plots were obtained from the pair-wise combination of the independent factors, while keeping other factors at its centre point level. Figs. 2 (a) & 3 (a) shows the effect of substrate concentration, initial pH and their interactive effects on biogas yield with the optimum level of TOC (20.32).The optimum values of the substrate concentration and initial pH for biogas yield is indicated at the top of the surface (Fig. 2 (a)). The biogas yield increased with increase in the substrate concentration from 89 to 107g/L when the initial pH and TOC kept at their centre values. The maximum biogas production of 53.9L/Kg VS was obtained with substrate concentration of 99g/L and initial pH 6.5 respectively. The effects of TOC, initial pH and their interactive effects, with optimum level of substrates concentration (99g/L) on biogas yield were shown in Figs. 2 (b) & 3 (b). Figs. 2 (c) and 3 (c) show the effects of substrate concentration. TOC and their interactive effects on biogas yield with the optimum level of pH (6.5). It was found that biogas yield increased with increase in substrate concentration from 89 to 107g/L. Highest biogas yield of 53.9 L/Kg VS was obtained with an initial substrate concentration of 99 g/l. An inhibitory effect of high substrate concentration generally occurs in anaerobic digestion process, depending on the types of substrates and microorganisms.

The optimum conditions for maximizing the biogas yield calculated by the obtained model (1) were a substrate concentration of 99g TS/L, aninitial pH of 6.5 and TOC of 20.32g/L from point prediction method. Under the optimum conditions, the predicted maximum biogas yield of 53.4 L/KgVS was obtained from the quadratic regression model. The experimentally determined production values (Table II) were close agreement with the statically predicted on which confirmed that the RSM with CCD analysis was a useful technique to optimize the biogas yield from the organic fraction of municipal solid waste through anaerobic digestion.

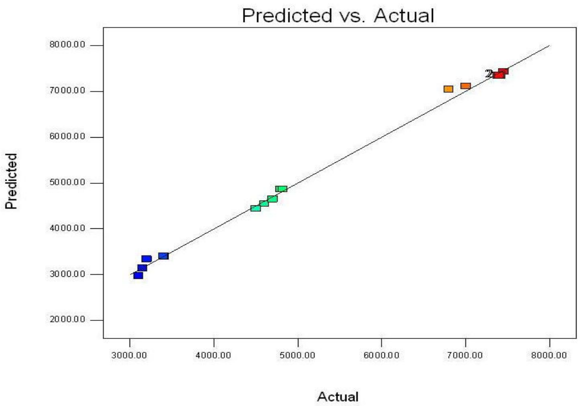
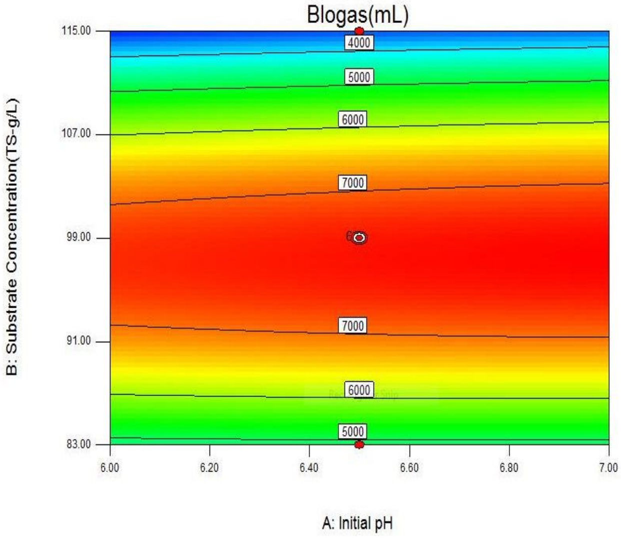
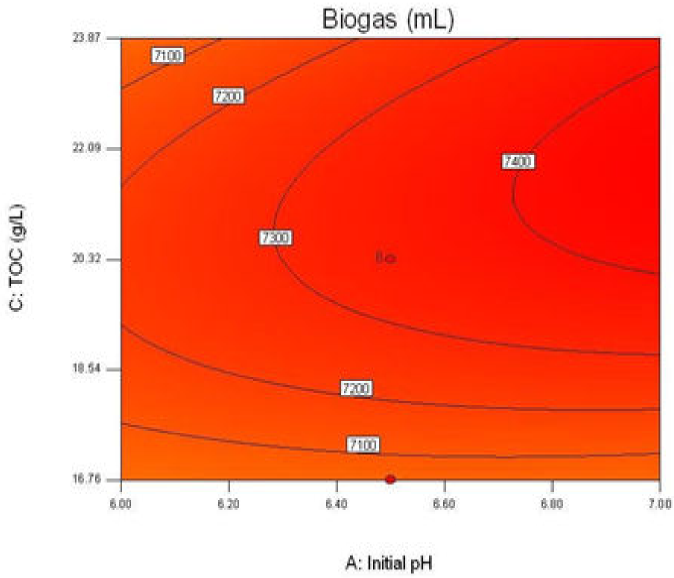


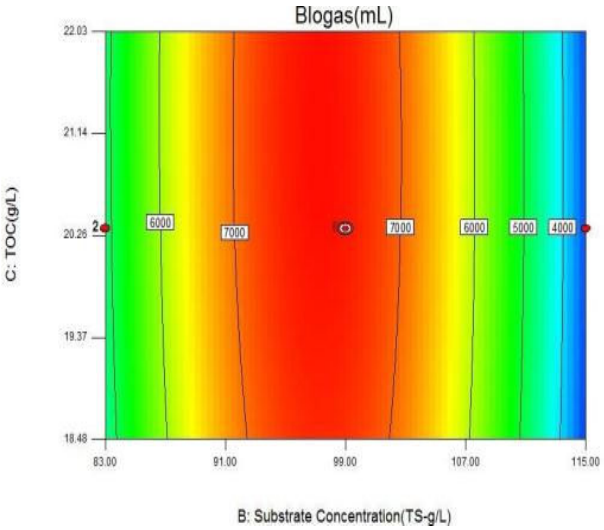
Fig. 1 Predicted vs. experimental biogas values



(a)

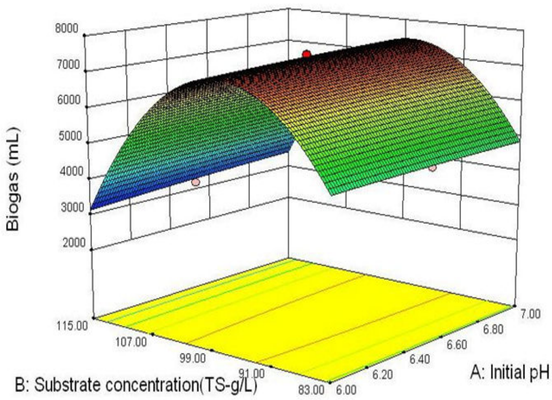


(b)

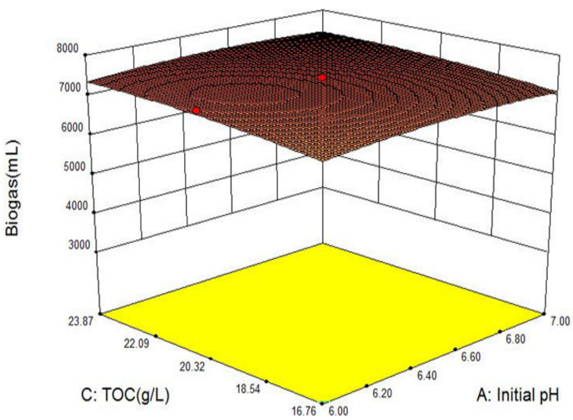


(c)

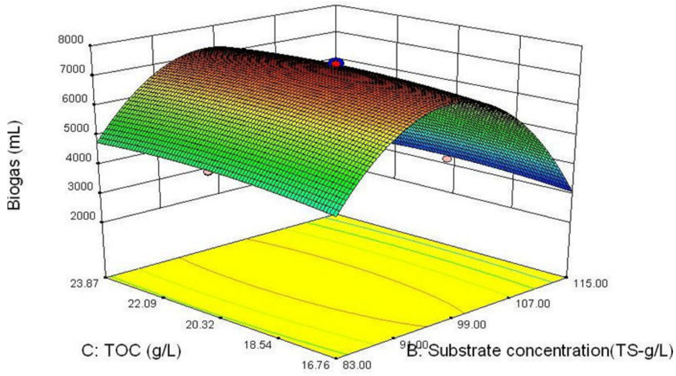
Fig. 2 Contour plot of biogas production as a function of (a) Substrate concentration (B) and initial pH(A),(b)TOC (C) and Initial pH (A), (c) Substrate concentration(B) and TOC(C)



(a)



(b)



(c)

Fig. 3 3D plot of biogas production (a) interaction between Substrate concentration (B) and initial pH (A), (b) interaction between TOC (C) and Initial pH (A), (c)interaction between Substrate concentration (B) and TOC(C)

IV. Conclusions

The present study focuses on the optimization of process parameters such as substrate concentration, initial pH and TOC for the maximal biogas production .Optimization of those variables was carried out by Response Surface Methodology using Central Composite Design. Only the initial pH and substrate concentration had significant individual effects on bio gas yield. The interactive effects for all of these factors were found to be insignificant ($p > 0.05$). The optimum conditions for maximizing the biogas yield were a substrate concentration of 99g TS/L, an initial pH of 6.5 and TOC of 20.32g/L in which maximum biogas yield of 53.4 L/KgVS mL was obtained. The maximum generation of biogas found experimentally using the optimized condition is 53.8 L/KgVS, which is in correlation with the predicted values 53.4 L/KgVS. It can be concluded that the RSM with CCD analysis was a useful technique to optimize the biogas yield from the organic fraction of municipal solid waste through anaerobic digestion.

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