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Optimization of process parameters for the economical generation of biogas from raw vegetable wastes under the positive influence of plastic materials using response surface methodology

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Abstract

In this experimental study, optimization of selected process parameters were assessed as controlling factors for the efficient generation of biogas by Response Surface Methodology (RSM) using raw vegetable wastes (RVW) and waste plastics (WAP) as the key constituents. The four factors: WAP, height to diameter (h/D) ratio, water content (WAC) and digestion time (DT) on weekly basis were optimized by RSM using rotatable central composite design (RCCD) to determine their optimum level. Considering various experimental data, the optimized result showed that enhanced biogas production (25.25 m^3/ton of RVW) was achieved with WAP (15%), h/D ratio (35), WAC (162 ml), and DT (18 week). A correlation coefficient (R^2) of 0.9976 indicated high degree of correlation between the variables

Keywords: Raw vegetable wastes, waste plastics, hydrolyzed material, biogas, response surface methodology.

Introduction

Rapid urbanization and increasing changes of the socio-economic condition would lead to the higher generation of municipal solid wastes (MSW) at a faster rate (Liu and Wu 2011). The overall characteristics of those MSW in general are different from place to place all over the world. Amongst all the wastes, RVW are the foremost carbonaceous biodegradable contributors available in MSW while WAP, another possible health hazardous material of various sizes and different categories are often found at random in MSW. However, most of the WAP found are non-biodegradable and impervious in nature and therefore, considered here to establish the study. Considering those two important characteristics, WAP inevitably restrain the easy gravity flow of hydrolyzed material (HYM) in the waste bed (WB) during the anaerobic biodigestion of MSW where, that HYM is the combined effect of water associated if any or initial moisture content of the waste materials and leachate generated in-situ in the WB.

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Hence, that intuitive property of impermeability of WAP was considered here as the principal basis of this study instead of the traditional cost induced mechanical or other artificial leachate recirculation practice to run the entire solid wastes treatment process efficiently. The contradictory situation implies that on one hand, more resources are being used to meet the increasing demand of plastics while on the other hand, more WAP are being generated concurrently. Most often, the WAP are neither collected properly nor disposed of systematically to avoid the negative impacts on the public health and the entire environment. Therefore, the increasing demand in plastic production along with its consumption has turned into a major challenge for the municipal authorities.

The overall biochemical reaction for anaerobic digestion represented as (Cossu et al. 1996):

$$C_aH_bO_cN_d + [(4a-b-2c+3d)/4]\, H_2O \rightarrow [(4a+b-2c-3d)/8]\, CH_4 + [(4a-b+2c+3d)/8]\, CO_2 + d\, NH_3$$

Considering the above biochemical reaction mechanism, the main product of that process is biogas, which can be the better alternative to the conventional fossil fuels and can reduce the greenhouse gases (Fergusen and Mah 2006; Madigan et al. 2003; Shahriari et al. 2012; Zehnder and Gujer 1983). In addition to the generation of biogas, residual solid material which, may be used as an amendment of soil also produced in due course of the reaction mechanism (Elango et al. 2007; Gomez-Lahoz et al. 2007; Kubler et al. 2000; Lawson 1992). Currently, the modern municipal waste management system follows the segregation and separation of the WAP as far as possible prior to suitable landfill operation, which is almost a difficult task. Hence, this is an additional cost for the entire operation which is another big question. Again, in the landfill site, sometimes the inferior, defective and inefficient treatment process lead to the groundwater contamination due to percolation of HYM through the WB and probable risk of explosion due to uncontrolled methane generation (Blakey 1996; Blight et al. 1996; Tchobanoglous et al. 1993).

Since a few decades, researchers are continuously trying to develop suitable models for such application which demands proper stability and consistency for an efficient operation. These analyses are also necessary from the aspect of process control of the landfill reactors ensuring consistent and trustworthy performances. However, no suitable and complete study has been found on the biodegradation of those waste materials in combination with WAP to draw the supportive conclusion because the spatially dependent process controlling factors cannot be properly characterized due to arbitrariness of the data and the possible complexities of the operating systems. Therefore, to make an affirmative conclusion and to establish the study, here an attempt was made to use the non-biodegradable and impervious WAP in the WB in contrast to the conventional non-usage of WAP and external recirculation of HYM to the WB (Barlaz et al. 1992; Pohland 1980). The necessary justification showed that the random presence of WAP in the WB would have an augmenting effect on the compactness of the WB during bio-consolidation and thus WB behaved as a packed bed. Consequently, this made a number of changes in the field capacity of the WB considering the porous areas (Tchobanoglous et al. 1993). Moreover, this condition improved the hydrolysis stage of the anaerobic bioprocess on holding the HYM for a longer period in the void spaces of the WB and thus enhanced the microbial enzymatic activities faster. Those void spaces were formed physically in the WB due to the random presence of WAP in it. Hence, that would be a better alternative to the additional cost involved water or leachate recirculation process by mechanical means and this in turn reduced the cost of whole operation. Therefore, application of this pioneering technique seems to be useful in other related bioprocess systems also.

On the basis of stoichiometric approach, at the onset of the digestion process both RVW and water (the initial moisture content) might be considered as limiting reactants (Cossu et al. 1996) . However, due to the random presence of WAP in the WB, the HYM generated and available in-situ in the pockets for a longer time could be considered as excess reactant for the anaerobic biochemical reaction mechanisms. This would lead to the higher and efficient generation of biogas by regulating the hydrolysis stage followed to entire anaerobic biodigestion process (Laines Canepa et al. 2011; Tchobanoglous et al. 1993). Again, considering stoichiometry, the changes in the chemical oxygen demand values during the anaerobic biodigestion process are often used to make an account for the methane production (Tchobanoglous et al. 2003). Furthermore, on investigation, height to diameter ratio (h/D) of the WB reactors, WAC and DT were compared and verified along with the WAP as important measureable controlling factors to establish the optimized model using RSM.

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 (Reddy et al. 2008). 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) using rotatable central composite design (RCCD) for the enhanced and efficient biogas generation. The interaction effects of the screened variables were also determined to establish the study. Recently, this method has been successfully used to improve many other bioprocess applications (Li et al. 2007; Puri et al. 2002; Xiao et al. 2007).

Materials and methods

Chemicals and Analysis

In the current study, two types of raw materials were used as feedstock. The first one was RVM which was collected from different local markets and the second one is various categories of WAP (nonbiodegradable and impervious), collected from different open dumping yards in local municipal areas. After collection, both of those raw materials were cut into smaller pieces. This technique was employed here to maintain the innumerable void spaces and low surface areas for effective surface contact of those waste materials for a better and sustainable digestion process. The statistical software package, Design Expert® 7.0.0, Stat-Ease Inc., Minneapolis, USA was used to analyze the experimental design and carry out the regression analysis of the experimental data.

Biogas Production

At the beginning of the experiment, preferred numbers of nonmetallic containers of different known dimensions with equal volumetric capacities of two liters to each of them were made ready as bioreactors. Then the physically processed and pre-calculated raw materials were added to the respective reactors and sealed consequently to maintain the strict anaerobic condition. Basically, RVW (fixed amount of 400g added to each container) considered here as the key ingredient for this experiment because of its enormous possible carbon source which actually accountable for the effective generation of biogas. However, to establish the catalytic impact of WAP in the process during anaerobic biodigestion of RVW, different combinations of WAP, WAC, h/D ratio and DT (week) were considered here according to design matrix (Table 1). The entire study was carried out at ambient temperature. The bed-heights for all those reactors were carefully noted all through the experimentation time. The leachate samples were collected separately from the respective reactors after stipulated DT. The said samples were then tested for the estimation of TOC (total organic carbon) content using standard methods & instruments and noted separately (Bartlett et al. 1994). Hence the biogas generation was estimated on the basis of TOC values of those leachate samples by theoretical stoichiometric calculations. Such estimation was done with the help of Ehrig's equation:

$$Ge = 1.868C(0.014T + 0.28) \tag{1}$$

where, Ge = total gas quantity (cubic meter per ton of RVW), C = TOC (kg per ton of RVW) and T = temperature (^{0}C) (Ehrig 1996).

Table 1. Experimental range of the four numerical variables studied using rotatable CCD in terms of actual and coded factors.

Factor	Name	Range of variables				
		-α (-1.68)	Low (-1)	Mid (0)	High (+1)	+α (+1.68)
A	WAP (%)	5	10	15	20	25
B	h/D	20	25	30	35	40
C	WAC (ml)	100	125	150	175	200
D	DT (week)	15	16	17	18	19

Optimization of biogas production

Selection of process factors

WAP, h/D ratio, WAC and DT (week) were considered as process factors for the production of biogas from RVW.

Optimization of key determinants by response surface methodology

Four key determinants viz. WAP (A), h/D ratio (B), WAC (C) and DT (D) were selected to study their effect on biogas production. All the variables were investigated at four widely spaced levels shown in Table 1 decided from previous unreported work. The response surface approach involving a rotatable central composite design (RCCD) was adopted to optimize the process controlling factors for biogas production from RVW (Bezerra et al. 2008; Ferreira et al. 2007). A set of thirty experiments including six center points was carried out to establish the study. Each numeric factor was varied over 5 levels (-2, -1, 0, +1, +2) i.e. plus and minus alpha (axial point), plus and minus one (factorial points) and zero (center point). The full experimental plan with respect to their actual and coded forms is listed in Table 1&2. The response values (Y) in each trial were the average of the triplicates.

Statistical analysis and modelling

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:

$$Y = \beta o + \sum_{i} \beta_i Xi + \sum_{ii} \beta_{ii} Xi^2 + \sum_{ij} \beta_{ij} XiXj$$

In this polynomial equation, Y is the predicted response, X_i X_j are independent variables, β_o is the intercept term, β_i is the linear coefficient, β_{ii} is the quadratic coefficient, and β_{ij} is the interaction coefficient.

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

$$Y = \beta_o + \beta_1 A + \beta_2 A + \beta_3 A + \beta_4 A + \beta_{11} A^2 + \beta_{22} B^2 + \beta_{33} C^2 + \beta_{44} D^2$$
$$+ \beta_{12} AB + \beta_{23} BC + \beta_{34} CD + \beta_{41} DA$$

Diagnostics Plots and model graphs were obtained using the Design Expert software to analyze the effects of variables individually and their interaction**s** to determine their optimum level. The point prediction method was used for optimization of the levels of each variable for maximum response.

Validation of the experimental model

The statistical model was validated with respect to all the three variables within the design space. A random set of five experimental combinations were used to study the biogas production. The experimental results were verified and compared with the optimized values.

Results and Discussion

Present days rapidly increasing energy demand and considering the higher calorific value of the fuels used, the large scale biogas production is often encountered as a better alternative to the limited resources of fossil fuels. RVW, the essential biodegradable carbonaceous parts of MSW are the principle contributor for the biogas generation which can be managed through systematic landfill management system. This in turn reduces the green house gases also. Here in this research work, an attempt was made to ascertain the above fact by using WAP for the availability of HYM as an essential excess reactant in the WB instead of additional cost involved mechanically managed water or leachate recirculation. Therefore, in this study to establish the new dimension of use of WAP along with other necessary controlling process factors, a systematic and robust optimization strategy was adopted for the enhanced generation of biogas under optimized conditions.

Optimization of biogas production

The full experimental plan of RCCD design for studying the effects of four independent variables, viz. WAP (A), h/D ratio (B), WAC (C) and DT (D) are listed in Table 2.

Table 2. Rotatable CCD matrix for four variables with actual biogas production.

.Sl. No.	A	B	C	D	Biogas production m^3/ton of RVW
1	10	25	125	16	12.71
2	20	25	125	16	13.55
3	10	35	125	16	15.85
4	20	35	125	16	16.47
5	10	25	175	16	13.94
6	20	25	175	16	14.75
7	10	35	175	16	16.54
8	20	35	175	16	17.43
9	10	25	125	18	17.82
10	20	25	125	18	17.97
11	10	35	125	18	19.82
12	20	35	125	18	20.07
13	10	25	175	18	18.17
14	20	25	175	18	19.21
15	10	35	175	18	19.84
16	20	35	175	18	20.46
17	5	30	150	17	5.77
18	25	30	150	17	6.53
19	15	20	150	17	19.2
20	15	40	150	17	23.93
21	15	30	100	17	20.72
22	15	30	200	17	22.73
23	15	30	150	15	15.46
24	15	30	150	19	24
25	15	30	150	17	22.37
26	15	30	150	17	22.71
27	15	30	150	17	21.89
28	15	30	150	17	22.61
29	15	30	150	17	22.8
30	15	30	150	17	21.77

The statistical significance of the second-order polynomial equation was checked by an F-test (ANOVA). The corresponding all the data are shown in Table 3. For biogas production, the correlation coefficient (R^2) of polynomial equation was found as 0.9976. 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 99.76% of the total variation could be explained by the model and only 0.24% of it was not explained by the model. So, quadratic model was chosen for this analytical work. "Adeq Precision" measures the signal to noise ratio. A ratio greater than 4 is desirable. The ratio found here 205.418 which indicated an adequate signal for this study. This model was used to navigate the design space. The adjusted R^2 (0.9954) was also very high, which indicated the higher significance of the model. The "Pred R-Squared" value of 0.9931

Table 3. Regression analysis for the production of biogas for quadratic response surface model fitting (ANOVA).

.Source	Sum of Squares	Degree of freedom	Mean Square	Coefficient estimate	Standard Error	F Value	p-value Prob > F	
Model*	604.59	14	43.18	-	-	452.29	< 0.0001	Significant
Intercept	-	-	-	22.36	0.13			
A	1.89	1	1.89	0.28	0.06	8.25	0.0005	
B	32.25	1	32.25	1.16	0.06	140.53	< 0.0001	
C	4.25	1	4.25	0.42	0.06	18.52	< 0.0001	
D	100.86	1	100.86	2.05	0.06	439.52	< 0.0001	
AB	0.01	1	0.01	-0.029	0.08	0.06	0.7150	
AC	0.14	1	0.14	0.094	0.08	0.61	0.2437	
AD	0.08	1	0.08	-0.069	0.08	0.33	0.3875	
BC	0.24	1	0.24	-0.12	0.08	1.05	0.1336	
BD	1.17	1	1.17	-0.27	0.08	5.08	0.0033	
CD	0.27	1	0.27	-0.13	0.08	1.18	0.1131	
A^2	455.19	1	455.19	-4.07	0.06	1943.20	< 0.0001	
B^2	1.33	1	1.33	-0.22	0.06	3.80	0.0020	
C^2	0.89	1	0.89	-0.18	0.06	2.29	0.0081	
D^2	12.64	1	12.64	-0.68	0.06	48.51	< 0.0001	
Residual	1.43	15	0.10					
Lack of Fit	0.48	10	0.05			0.08	0.9683	Not significant
Pure Error	0.95	5	0.19					
Cor Total	606.02	29						

*SD, 0.31; Mean, 18.24; R-Squared, 0.9976; Adj R-Squared, 0.9954; C.V. %, 1.69; PRESS, 4.16.

showed the reasonable agreement with the "Adj R-Squared" value of 0.9954. 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 (1.69 %) 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 4.16. The Model F-value of 452.29 implied that the model was significant. It must be mentioned hereof that due to the creation of noise, the probability of obtaining high model F-value was only 0.01%. Values of 'prob > F' less than 0.05 indicated that the model terms were significant. In this case A, B, C, D, BD, A^2, B^2, C^2, D^2 were significant model terms. Moreover "Lack of Fit F-value" of 0.26 implied that it was not significant relative to the pure error. Non-significant lack of fit indicated a good fitness of the model. There was only 96.83% chance that this magnitude of "Lack of Fit F-value" could occur due to noise. The model showed standard deviation and mean values of 0.31 and 18.24, respectively.

The normal probability plot given in Figure 1a shows some scatter along the line indicating that the residuals follow a normal distribution. Residuals vs. Predicted plot in Figure 1b indicates the residuals versus the ascending predicted response values. The plot shows a random scatter (constant range of residuals across the graph). Actual vs. Predicted plot (Figure 1c) also represents a high degree of similarity that is observed between the predicted and the experimental values. From the three diagnostic plots (Figures 1a-c), it can be concluded that the model has satisfied the assumptions of the analysis of variance and also reflected the accuracy and applicability of RSM to optimize the process factors for the efficient generation of biogas. Perturbation plot in Figure 2 represents the comparison of the effect of process parameters at the midpoint (coded 0) in the design space. A steep slope or curvature was found for the plot with WAP, h/D ratio, WAC and DT showing the response sensitivity to these factors.

The contour plots (Figures 2a-c) determined the interaction of the process parameters and optimum value of each component for maximum response. Those plots were obtained from the pair-wise

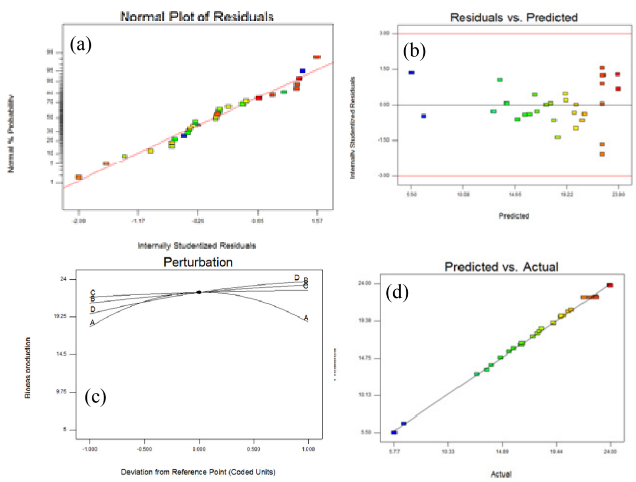

Figure 1. Diagnostic plots of the quadratic model used for biogas production (a) Normal plot of Residuals, (b) Residuals vs. Predicted plot and (c) Perturbation plot of production of biogas as a function of WAP (A), h/d ratio (B), WAC (C) and DT (D). and (d) Predicted vs. Actual Response plot

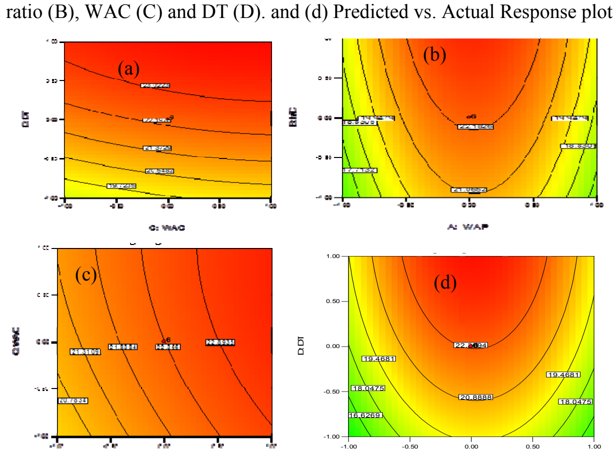

Figure 2. Contour plot of biogas production as a function of (a) WAC (C) and DT (D) (B), (b) WAP (A) and h/d ratio, (c) h/d ratio (B) and WAC (C)and (d) WAP (A) and DT (D).

combination of the independent factors, while keeping other factors at its centre point level. The elliptical contour plots (shown in Figures 2a-d) clearly indicate that the mutual interaction is prominent among the factors.

Table 4. Validation of quadratic model within the design space.

Sl. No.	**WAP (%)**	**h/D**	**WAC (ml)**	**DT (week)**	Predicted biogas	Actual Biogas
1	20	25	150	16	13.34	12.94
2	17.5	35	140	17	22.24	21.91
3	12.5	30	125	18	22.18	22.98
4	10	25	170	16	13.88	13.02
5	15	35	162	18	24.44	25.25

The optimum values for the four components were found as 15% WAP, 35 of h/D ratio, 162 ml of WAC and 18 DT (week) for biogas production from point prediction method. The maximum predictable response was calculated using regression equation employing substituted level of factors and was experimentally verified.

Validation of the experimental model

The model was validated for all three variables within the design space. A random set of five combinations of variables were prepared and tested for biogas production (given in Table 4). The experimentally determined production values were in close agreement with the statistically predicted ones, confirming the model's authenticity and applicability of the statistical model (RSM) for the optimization of process variables.

Conclusions

The present study focuses on the optimization of process parameters for the maximal biogas production using RVW under the strong influence of WAP. This is important for obtaining the higher biogas production as well as for the reduction of process operating cost by omitting the cost induced artificial leachate or watery material recirculation. Four variables namely, WAP, h/D ratio, WAC and DT (week) were found to have significant effects on the efficient generation of biogas during anaerobic biodigestion of RVW. Optimization of those four variables was carried out by Response Surface Methodology using Rotatable Central Composite Design. The maximum generation of biogas found experimentally using the optimized condition is 25.25 m^3/ton of RVW, which is in correlation with the predicted values of 24.44 m^3/ton of RVW.

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