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Optimization of methane production during mesophilic anaerobic digestion using response surface methodology

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ABSTRACT

Box–Behnken design based (BBD) on response surface methodology (RSM) adopted for evaluating and optimizing the performance of anaerobic digestion (AD) of vinasse in continuous mode concerning chemical oxygen demand (COD) removal and daily production of methane under mesophilic conditions. The process of AD is conducted in the digester with 4200 m^3 volume and continuous system operated for 1 y at industrial scale. The effect of three factors namely pH, hydraulic retention time (HRT) and organic loading rate (OLR) is investigated. RSM by the BBD verifies that the daily production of methane and COD removal mainly affected by operating condition: OLR, HRT and pH. For two responses % COD and production of methane, the analysis of variance results shows that all adequacy measures (R^2 values) is found to be close to 1 indicating a sufficient regression for model development. In addition, linear model values (A, B, C), quadratic model value (A^2, B^2, C^2), and interactive model values (AB, AC, BC) are found to be significant, with P-values <0.05. Thus, the error percentage between experimental and predicted values which is around 3.98% for methane production and 3.52% for COD degradation efficiency suggests the good predictability of the model.

Keywords: Vinasse; Anaerobic digestion; Methane production; Chemical oxygen demand removal; Response surface methodology

1. Introduction

Anaerobic digestion (AD) is one of the most promising technologies, breaking complex organic substrates into biogas [1] that is substantially composed of a mixture of methane and carbon dioxide. AD, being 100% renewable, is an effective and environmental-friendly waste and wastewater management technique and can be considered as one of the most important renewable energy sources, due to CH_4 generation during the digestion process [2]. In Morocco, AD can be a strategic choice for the promotion of green energy, thanks to the significant potential of biomass available in the Kingdom and due to its strong commitment to environmental protection and promotion of sustainable development (Law n° 13-09, 2010) [3]. Thus, the production of methane by AD of biomass is one the promising resources of renewable energy not yet supported in Morocco [4].

Thus, various factors affect the performance and design of an AD that can be identified as reactor design, feedstock characteristics, and operating conditions namely pH, temperature, hydraulic retention time (HRT), mixing, solid retention time (SRT), and organic loading rate (OLR).

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Although several authors have studied the effect of these several parameters on the methanogenic step of AD system. Elazhar et al. [5], evaluated over 1 y the AD performance of Sotrameg Company in industrial conditions. The evolution of OLR on the system stability is explored in terms of pH, chemical oxygen demand (COD) removal, biogas production, methane yield and HRT. The study showed that increasing OLR value improved the efficiency of total COD conversion into methane production. It is known that both HRT and OLR affect the stability and productivity of the process. With respect to HRT, the effect on the AD process is controversial; a high HRT favors volatile fatty acids (VFA) accumulation. Thus, there is a need to determine a suitable HRT to obtain stable and optimal AD [6–8]. The pH value could also influence the AD process and the appropriate range usually chosen is 6.5–8.2 [5–9,10].

Therefore, these parameters should be statistically optimized and the relationship between all these parameters must be investigated for effective methane production and COD degradation efficiency. response surface methodology (RSM) is one of the most effective approach for designing experiment, building model, and optimizing condition on responses which are influenced by several independent variables [11–13]. Compared to the traditional methods, RSM could define not only the influence of independent variables on the responses, but also the interaction between these variables to achieve best system performance [14]. The experiment designed by RSM requires fewer tests and shorter time consuming but could obtain a full-experimental design comparison [15].

In this study, the optimum conditions for maximum daily methane production and maximum COD degradation in 3 independent factors is performed using Box–Behnken experimental design (BBD) combining with response surface modeling and quadratic programming. For this, the individual and interactive effects of three factors such as pH, OLR and HRT on daily methane production and COD degradation efficiency during a mesophilic AD of vinasse are investigated. The models developed in this study allow providing guidance for future feedstock evaluation and process optimization in AD. Consequently, reducing the financial cost, this is the main argument for the lack of implementation or improvement of treatment systems.

2. Methods and materials

2.1. Treatment flow chart

Sotrameg Company, established in 1975, is the Moroccan leader of ethanol production by mesophilic fermentation of molasses with Saccharomyces cerevisiae for chemical, pharmaceutical, cosmetics and vinegar industry. The annual production reaches more than 50 million liters of alcohol with yields up to 250 L of alcohol/ton of molasses. In parallel, Sotrameg Company is facing a real problem, which is the generation of highly polluting effluents called vinasse (the main liquid stream from the first-generation ethanol production process), whit annual production between 400–750 million L. The company adopted AD as an interesting alternative for treatment of vinasse to promoting the stabilization of organic matter and for biogas production. Fig. 1 shows the schema of the wastewater treatment plant manufactured by Sotrameg Company in 2000. This full-scale bioprocess includes two anaerobic Complete Mix Bioreactors, followed by activated sludge treatment which is used as a tertiary treatment devoted to treat the water at the outlet of anaerobic reactors after the AD before their discharge in the receiving environment.

The volume of each anaerobic bioreactor is 4,200 m^3 based on the average OLR of 3 kg DCO/m^3 per day and 6.5 m

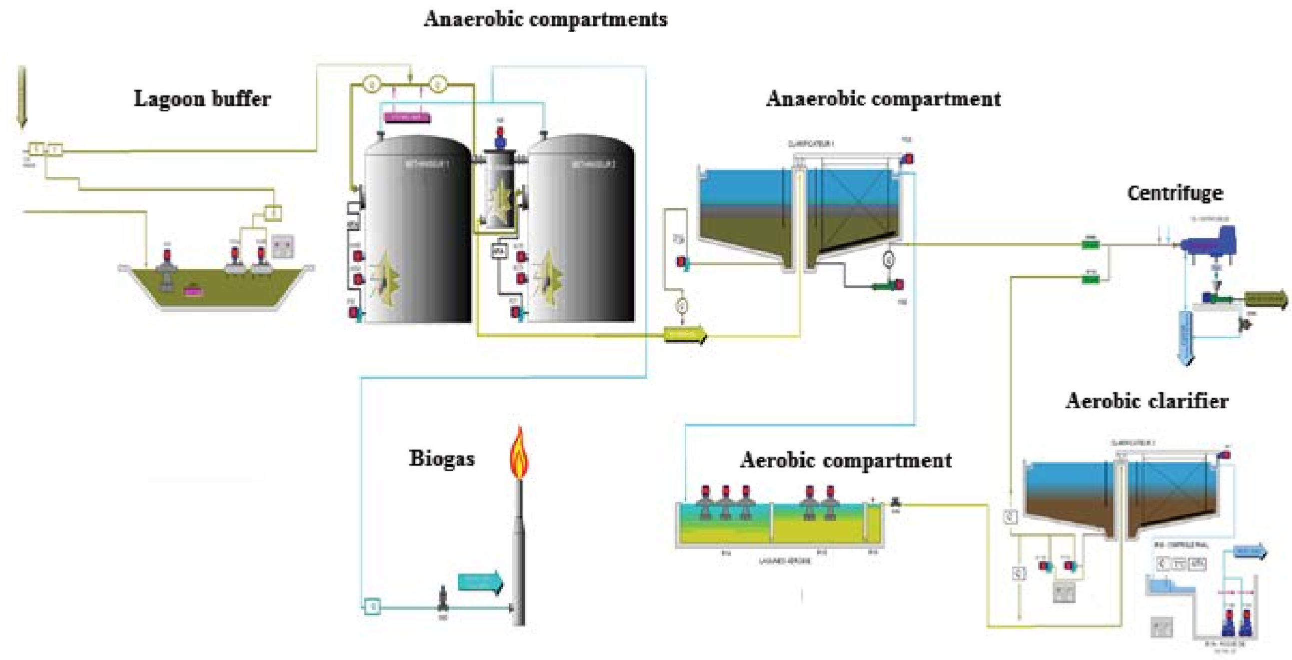

Fig. 1. Schematic of the full-scale plant of vinasse treatment of Sotrameg Company.

in height with an internal diameter of 30 m. The HRT of the bioreactors is 20 d. Peristaltic pumps controlled by programmed electronic timer are used to regulate the feeding, recirculation, and decanting operations in both the reactors. Four main parameters (pH, temperature, gas production and effluent flow) are recorded using an integrated on-line data recording system connected to the pH probe, temperature detector, gas flow meter, and effluent flow meter. pH of the digester is controlled by an automated pH controller, which pumped NaOH when pH dropped below 6.8. The pump stopped when the pH exceeds 7.8. Temperature of the digester is controlled and is recorded between 37°C and 39°C. A degassing post with a working volume of 15 m^3 is installed between the two bioreactors to collect the biogas. The collected biogas is sent to the boilers and the excess gas is sent to a flare to be burned. The treated wastewater is clarified in anaerobic clarifier before transferring to the aerobic basins.

The aerobic treatment is composed of three basins: degassing compartment of the water coming from the two digesters and two parallel aerobic compartments each with a capacity of 6,000 m^3. The aerobic compartments are inoculated with the biomass obtained from the anaerobic reactor. The necessary oxygen for degradation of organic matter is provided by five surface turbines. The generated sludges by the anaerobic and aerobic treatments are sent to a centrifuge to increase their dryness.

2.2. Vinasse characterization

Vinasse is characterized as an effluent with a high pollution potential, containing high levels of organic compounds and nutrients mainly COD, total phosphorus (TP) and total nitrogen (TN). The characterization of the raw vinasse is summarized in Table 1.

2.3. Experiment design by Box–Behnken of RSM

For RSM, the BBD is used. All experiments carried out in a randomized order to minimize the error in the response due to miscellaneous factors. The experiment consists of 15 experimental runs, with all possible combination of values for each experimental factor, namely pH (*A*), HRT (*B*), and OLR (*C*). The independent input variables at low, medium, and high levels are shown in Table 2.

Table 1
Characteristics of the vinasse

Parameters	Vinasse	Moroccan standard discharge values [16]
pH	4–5	6.5–8.5
TSS (mg/L)	1,500–2,500	50
T (°C)	58.2	<30°C
COD (mg/L)	6,000–70,000	500–800
BOD_5[a] (mg/L)	35,000–40,000	100–200
TP (mg/L)	270	10
TN (mg/L)	31–1,250	30

[a]BOD5: 5′day biochemical oxygen demand.

In RSM experiments, the responses studied the removal COD (y_1) and daily methane production (y_2). A quadratic polynomial regression model is used to predict both responses as follows [17]:

$$Y = \beta_0 + \sum_{i=1}^{2}\beta_i X_i + \sum_{i=1}^{2}\sum_{j=1}^{2}\beta_{ij}X_iX_j + \sum_{i=1}^{2}\beta_{ii}X_i^2 + \xi \tag{1}$$

where *Y* is the predicted response, β_0 is the constant coefficient, β_i is the linear coefficients, β_{ij} is the interaction coefficients, β_{ii} is the quadratic coefficients, and X_i and X_j are the coded values of the AD variables and ξ is the residual term. The goodness of fit of the model is evaluated by the analysis of variance (ANOVA). 3D and contour surface response are constructed to evaluate the interaction that has significant effects.

3. Results and discussion

3.1. Box–Behnken experimental design and responses

The experimental results of COD removal and daily production of methane by the Box–Behnken design and the predicted values are presented in Table 3. The results obtained from the model show that the offered model is not much different from the actual results obtained, and the R^2 values gained from the correlation values of the results obtained and the foreseen values for CH_4 and % COD are 0.989. In this study, the ANOVA test is applied for appraising the regression; its details are given in Table 4.

3.2. ANOVA and model fitting

The ANOVA examines the significance of three types of variables based on *F*-value, *P*-value, and sum of the squares (SS). According to results of ANOVA, *F*-value for % COD and CH_4 is 4,945.22 and 5,985,292, respectively, which specifies that the model used is significant and is able to show a good correlation between response and independent variables. For each response, if the *P*-values is less than 0.05, the items are significant and if the *P*-values are greater than 0.05 it means that the parameter is not significant [18,19]. As shown in Table 4, the significant terms of the model for % COD and CH_4 are linear model values (*A*, *B*, *C*), quadratic model values (A^2, B^2, C^2), and interactive model values (*AB*, *AC*, *BC*) found to be significant, with *P*-values <0.05.

By applying multiple regression analysis on the experimental data, a second-order polynomial equation, for COD removal and daily production of methane fitted in terms of coded factors obtained as follows:

Table 2
Independent input variables range in terms of coded levels

Factors	Variables	Coded level		
		–1	0	+1
pH	*A*	7	7.5	8
HRT (d)	*B*	10	27	36
OLR (kg/m^3 d)	*C*	0.1	2.45	4.5

Table 3
Box–Behnken design matrix measured and predicted response of COD degradation and daily production of methane

Std	Run	Factors levels			Response			
		A	*B*	*C*	% COD_{exp}	% COD_{pred}	CH_{4exp} (m^3/d)	CH_{4pred} (m^3/d)
1	10	0	1	–1	74	74.00	1,367	1,429.25
2	13	0	0	0	64	63.75	7,578	7,475.00
3	1	–1	–1	0	58	58.12	1,344	1,494.75
4	3	–1	1	0	54	53.87	532	579.75
5	14	0	0	0	46	45.87	2,345	2,345.00
6	4	1	1	0	79	79.12	1,276	1,125.25
7	12	0	1	1	74	73.89	3,346	3,283.75
8	8	1	0	1	74	74.000	2,345	2,345.00
9	7	–1	0	1	48	48.125	567	670.00
10	15	0	0	0	78	78.06	432	266.75
11	5	–1	0	–1	75	75.25	3,763	3,748.50
12	9	0	–1	–1	85	84,87	153	167.50
13	11	0	–1	1	76	75.87	6,537	6,702.25
14	2	1	–1	0	59	59.06	2,345	2,345.00
15	6	1	0	–1	52	52.12	4,578	4,530.25

$$\begin{aligned}\text{COD} = {} & 496.7 - 122.51A + 20.431B + 2.602C \\ & + 8.000A \times A - 0.04142B \times B + 2.2011C \times C \\ & - 0.0769A \times B + 0.244A \times C - 0.32833B \times C\end{aligned} \quad (2)$$

$$\begin{aligned}\text{CH}_4 = {} & -41{,}681 + 13{,}074A - 511.1B + 2{,}719C \\ & - 1{,}014A \times A + 161.4C \times C - 1.527B \times B \\ & - 209.5A \times C + 80.5A \times B - 30.29B \times C\end{aligned} \quad (3)$$

In the same way, low *P*-value and high *F*-value show that the terms are significant [18, 20]. Moreover, the sum of squares (SS) values of the variables is high, which displays the importance of the variables. As shown in Table 4, linear coefficients compared to the second-order and interactive coefficients are important for both responses.

The other two parameters, which should be considered and employed for indicating the quality of the designated polynomial model for data fit are the coefficient of determination R^2 and adjusted-R^2. Researchers have established that good statistical models of best fit should have an R^2 value between 0.75 and 1 [21, 22]. In this study, all adequacy measures (R^2 values) are found to be close to 1 indicating a sufficient regression for model development for two responses. The values of R^2 and adjusted-R^2 for % COD are 0.998 and 0.995 and for CH_4 are 0.997 and 0.993, respectively. As it shown in Table 4, the value of adjusted-R^2 is lower than R^2, reflecting the good quality of the model.

Figs. 2a, b and 3a, b show the predicted values vs. actual and normal probability plot of residuals for the two studies responses, COD removal and daily production of methane. In the scatter diagram a homogeneous distribution of the design points close to the diagonal line seen indicating that model is adequate. There is no significant difference between the experimental data and the predicted data in any case and the developed models validated within 95% level of confidence. In addition, normal distribution of residuals (the difference between actual and predicted data) is seen in COD removal and daily production of methane. The normal distribution plot proves the assumption of normal data distribution. Therefore, no abnormality in the experimentation works for the two responses. The model is successful in predicting COD removal and daily methane production.

3.3. Response surface plot

The 3D response surfaces and 2D contour plots which applied to describe the interaction of different variables on COD removal and daily production of methane are shown respectively in Figs. 4 and 5.

The RSM analysis shown in Fig. 4a reveals that the COD removal increases with increasing OLR from 0.1 to 4.2 kg/m^3.d and then decreases gradually with increasing TRH at fixed value of pH = 7.2. The maximum response values of COD removal (84%) obtained at OLR 4.2 kg/m^3.d and HRT 10 d. Also, the decrease in pH favors the reduction rate of COD, more pH tends towards neutrality, more the (organic degradation is better (Fig. 4b). The COD removal remarkably low with increasing HRT and pH (Fig. 4c). At fixed value of OLR, the optimum HRT and pH obtained for COD removal 10 d and 7.2 respectively. Under this condition, the response surface suggests that OLR, pH and HRT have a significant positive effect on the degradation of COD.

The interaction of OLR and TRH on the response of daily production of methane at every pH level within the experimental range (pH = 7.2) is illustrated in Fig. 5a. This figure indicates that the production of methane is favored for high values of OLR and low values of HRT. Thus, the maximum value of CH_4 is obtained for OLR = 4.2 kg/m^3.d and for a HRT value of 10 d, when the CH_4 value reaches

Table 4
ANOVA test for removal COD and daily production of methane

COD removal	DF	Sum of squares	Mean square	*F*-value	*P*-value
Model	9	2,225.35	247.261	4,945.22	<0.0001
A	1	28.12	28.125	562.50	<0.0001
B	1	924.50	924.500	18,490.00	<0.0001
C	1	465.12	465.125	9,302.50	0.0200
A^2	1	14.77	14.769	295.38	0.0020
B^2	1	315.92	315.923	6,318.46	<0.0001
C^2	1	180.92	180.923	3,618.46	0.001
AC	1	1.00	1.000	20.00	0.007
AB	1	0.25	0.250	5.00	0.076
BC	1	306.25	306.250	6,125.00	<0.0001
Lack of fit	3	0.25	0.083	*	*
Pure error	2	0.00	0.000		
Total	14	2,225.60			
S	R^2		R^2 (adjusted)		R^2 (predicted)
0.223607	0.998		0.995		0.998
Daily production of methane					
Model	9	53,867,625	5,985,292	223.31	<0.0001
A	1	1,102,613	1,102,613	41.14	0.0010
B	1	2,473,088	2,473,088	92.27	<0.0001
C	1	44,048,498	44,048,498	1,643.42	<0.0001
A^2	1	237,510	237,510	8.86	0.0310
B^2	1	246,013	246,013	9.18	0.0290
C^2	1	1,699,173	1,699,173	63.39	0.0010
AC	1	184,470	184,470	6.88	0.0470
AB	1	1,095,162	1,095,162	40.86	0.0010
BC	1	2,606,610	2,606,610	97.25	<0.0001
Lack of fit	3	134,015	44,672	*	*
Pure error	2	0	0		
Total	14	53,867,625	5,985,292		
S	R^2		R^2 (adjusted)		R^2 (predicted)
163.716	0.997		0.993		0.960

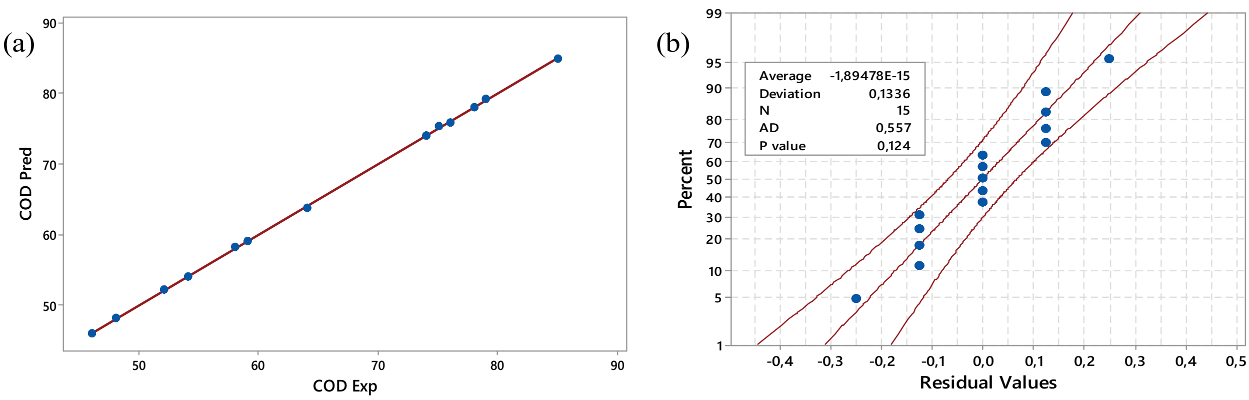

Fig. 2. (a) Plot of actual vs. predicted values and (b) plot of residuals and normal probability for COD removal.

6,500 Nm^3/d. Results show also a significant interaction between HRT and OLR for daily production of methane. All the 3D surface plots depict the effect of HRT and OLR on COD degradation and daily methane production at alkaline medium. This agrees with previous studies, in which a similar trend is observed [23–24].

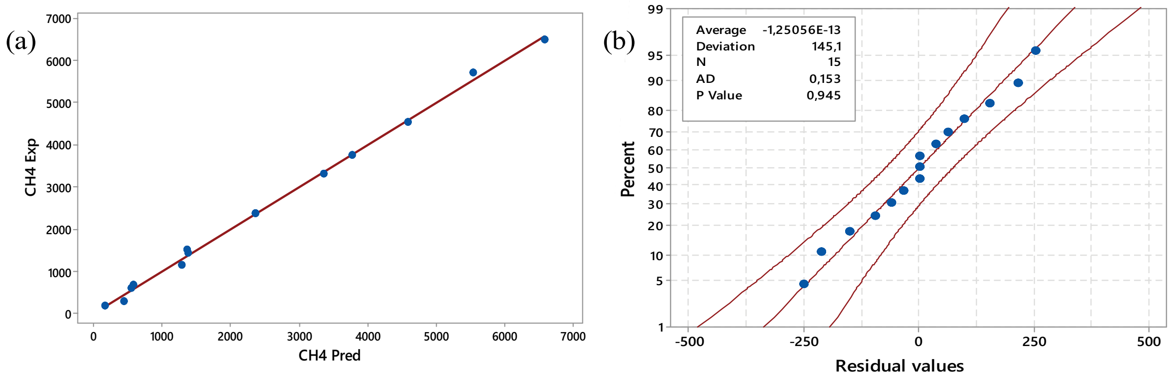

Fig. 3. (a) Plot of actual vs. predicted values and (b) plot of residuals and normal probability for daily production of methane.

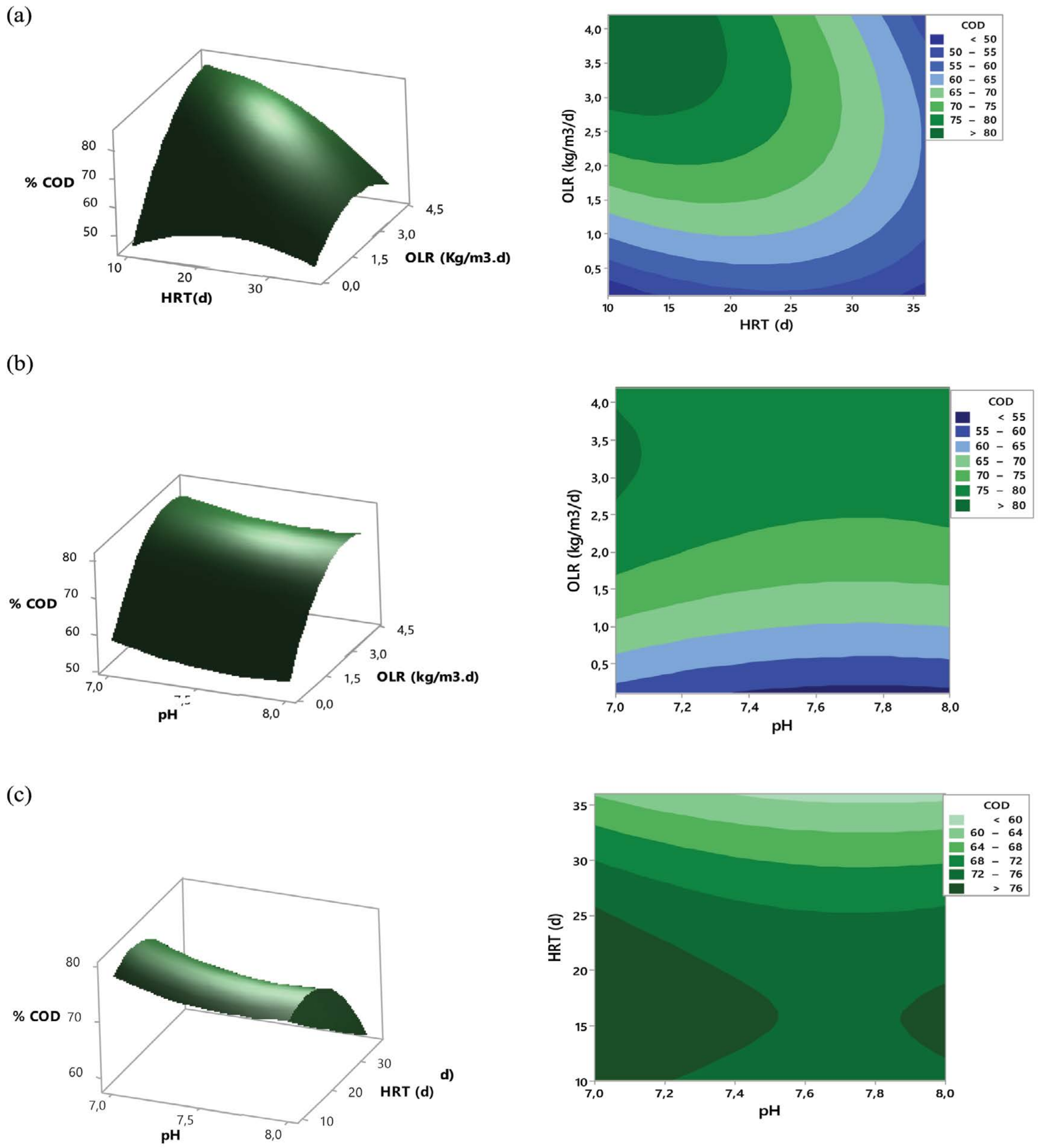

Fig. 4. 3D response surfaces and 2D contour plots for COD degradation.

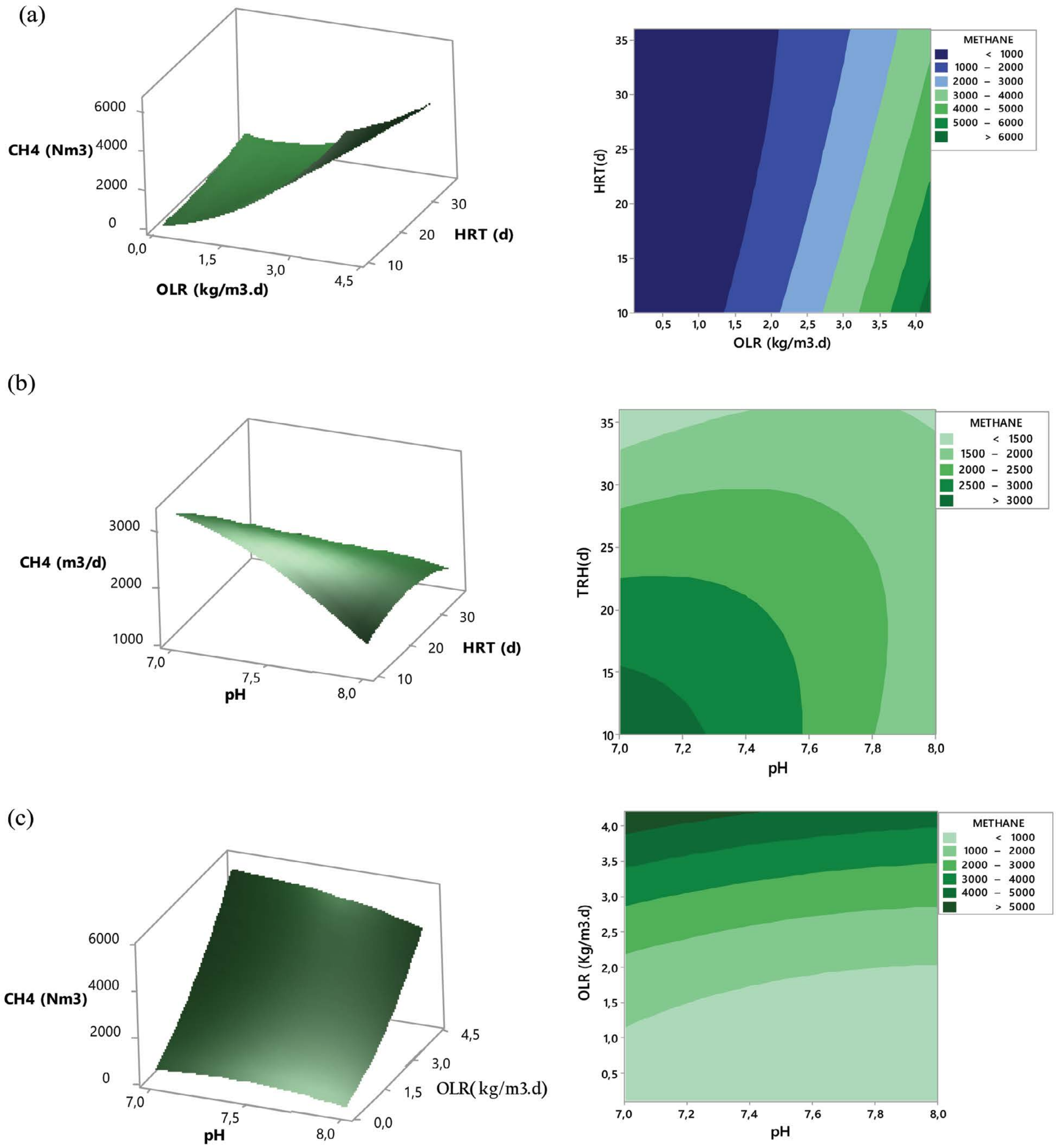

Fig. 5. 3D response surfaces and 2D contour plots for daily production of methane.

Similar trend is observed for pH and HRT interaction on daily production of methane (Fig. 5b). The production of methane is favored in a practically neutral pH 7.2 and lower HRT (10 d).

Fig. 5c shows the relationship between pH and OLR on the evolution of daily production of methane for a TRH of 10 d. The production of methane is optimal for OLR of 4.2 kg/m^3 d and a pH of 7.2. Then, the methane production gradually decreases for high pH and low OLR values. This result means that no interaction is generated between initial pH and OLR [25–26]. In general, interaction is observed only between HRT and OLR and these two factors influence the methane production more substantially.

3.4. Optimization using desirability analysis

In RSM, for performing multi-objective optimization, all the responses are considered simultaneously using desirability analysis, a useful approach for optimization more than one response. The parameter setting achieving higher desirability value close enough to 1 is considered to be the optimum conditions, and the simultaneous objective function is a geometric mean of all responses. In this experimental investigation, the optimum input parameter setting is evaluated with the both objectives formulated of maximizing production of methane and COD degradation.

The plot given in Fig. 6 shows the desirability analysis of COD degradation and daily production of methane which indicates the optimum input values and the predicted output responses. pH 7, OLR 4.2 kg/m^3.d and HRT 10 d, lead to the production of 7,880 Nm^3 of methane, which correspond to COD degradation of 88%. The desirability value of methane and % COD is equal to 1 which corresponds in to perfect desirability. It is found that the best pH range for an anaerobic co-digestion healthy environment for bacteria needs to be between 6.5 and 8.0 [13]. Whilst below 6.5, the efficiency of the system is inhibited. This agrees with the report by Torkian et al. [27].

Table 5
Predicted and observed optimum values of responses

Variables	Low level	High level	Optimum level	
A: pH	7	8	7	
B: HRT (d)	10	36	10	
C: OLR (kg/m^3 d)	0.1	4.2	4.2	
Response	Goals	Exp.	Pred.	Error
% COD	Maximize	85	88	3.52%
CH_4 (Nm^3/d)	Maximize	7,578	7,880	3.98%

Table 5 shows the RSM predicted and experimentally optimum response values and the corresponding percentage error during experimental validation of the developed models. The maximum error is 3.98% for daily production of methane and it equal to 3.52% for COD removal. Hence, a close relationship is identified between the predicted and the observed values.

4. Conclusion

Experimental investigation on COD removal and daily production of methane through AD of vinasse are successfully optimized using RSM through the BBD statistical experimental design, and a satisfactory second-order polynomial equation is derived.

For two responses % COD and production of methane, the analysis of variance shows that all adequacy measures (R^2 values) are found to be close to 1 indicating a sufficient regression for model development. In addition, linear model values (*A*, *B*, *C*), quadratic model value (A^2, B^2, C^2) and interactive model values (*AB*, *AC*, *BC*) are found to be significant, with *P*-values <0.05.

The desirability approach leads to the following combined optimum condition: pH 7, OLR 4.2 kg/m^3.d and HRT of 10 d. In these conditions, the production of methane

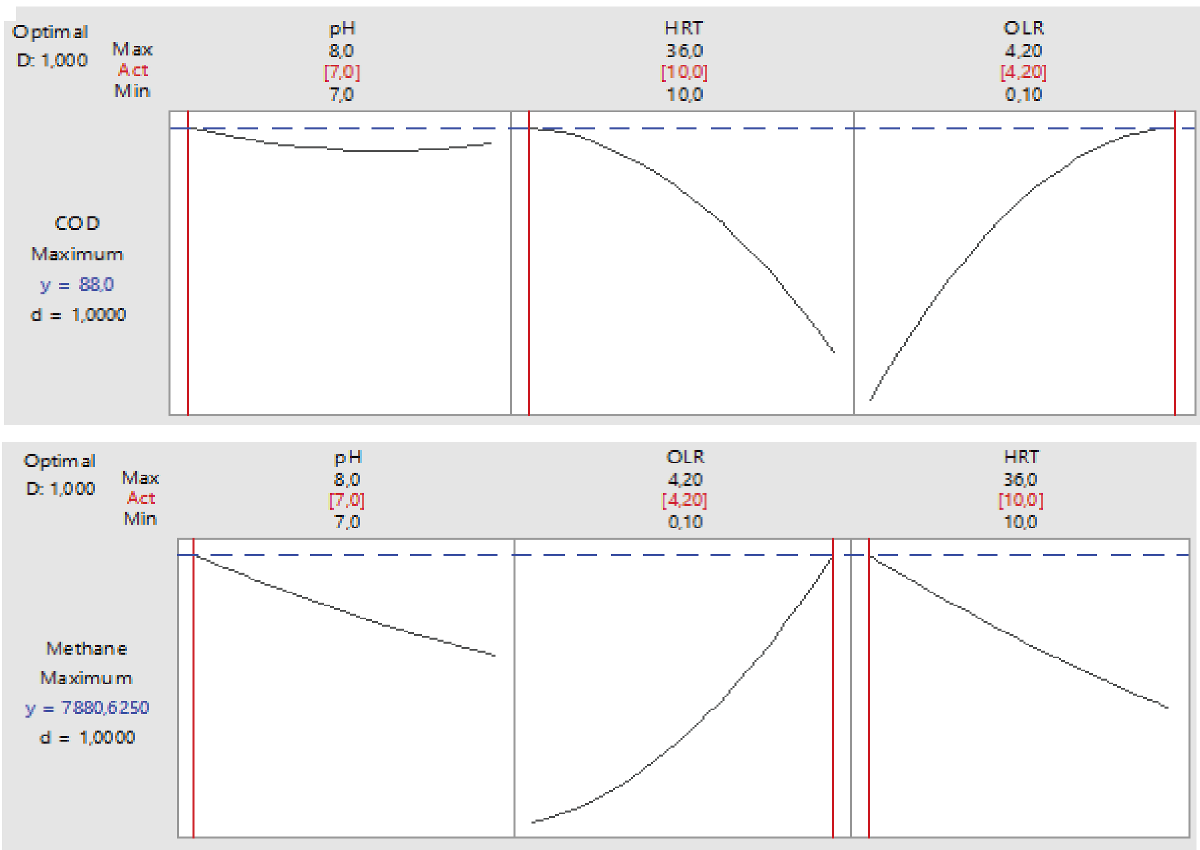

Fig. 6. Desirability plot of COD degradation and daily methane production.

peaks at 7,880 Nm^3 and degradation of COD reaches 88%. The maximum error % between RSM predicted and experimentally observed optimum response is 3.98% for daily production of methane and it equal to 3.52% for COD removal. These values confirm the efficiency of the optimization procedure. RSM is an effective and economically viable alternative technique that can be adapted for optimizing the parameters of AD process and favorably maximizing the output responses.

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