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Research article

Enhancing biogas production from vinasse through optimizing hydraulic retention time and added load using the response surface methodology

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A R T I C L E I N F O

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A B S T R A C T

Vinasse, a byproduct of ethanol production from sugarcane, is a rich organic matter and poses environmental challenges due to its high pollutant content. Effective biomethane production from vinasse can mitigate its environmental impact by converting organic matter into a useful energy source while reducing its pollutant load. The biomethane production by anaerobic digestion (AD) process of the vinasse byproduct was examined on a laboratory scale. In this regard, several loads from 0.5 to 7 g VS/L were investigated to assess AD performance and methane production. This study investigated how two separate factors, namely the load and hydraulic retention time (HRT), affect both cumulative methane production (CMP) and methane yield (Y_{CH4}). This investigation utilized a response surface methodology known as the central composite design (RSM-CCD). Statistical analysis of variance (ANOVA) was employed to evaluate the effectiveness of the model generated. Thus, the model's fit, Y_{CH4} has a maximum R^2 value of 0.9759. The results revealed an astounding level of agreement between the experimental data and the proposed model. The RSM results revealed maximum CMP and Y_{CH4} values of 409.82 ml and 178.95 ml/g VS respectively, obtained for optimum load values of 2.17 g VS/L and HRT of 15 h. The results emphasize the environmental and economic significance of AD, providing a sustainable waste management solution that helps reduce greenhouse gas emissions and organic pollution. Additionally, it generates valuable biogas and biofertilizers, presenting economic opportunities through renewable energy production and resource recovery. This approach not only alleviates the environmental burden of vinasse but also enhances the economic viability of ethanol production by creating additional revenue streams.

1. Introduction

Converting sugar cane and its by-products into bioethanol is one of the best-known applications for biorefineries [1,2]. In fact, in the coming years, bioethanol production is expected to increase and will be about 134 billion liters in 2024 [3]. These industries generate large quantities of liquid effluents called vinasse or stillage after ethanol production by fermentation of organic residues such as bagasse, sugarcane, and molasses [4,5]. The primary issue facing the ethanol production company is still the creation of this liquid residue, which requires 13L of vinasse for every 1L of ethanol produced. There are two feedstock types to produce bioethanol. The first

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uses the sugarcane crop as a substrate called first generation biofuel (1G biofuel) while the second uses the residue of the sugar industry called molasses to produce bioethanol (2G biofuel) [6]. Commonly, vinasse is a complex product containing several phenol compounds and defined by a high content of organic matter including a high chemical oxygen demand (COD) [6]. Its energy recovery would therefore in principle be economically viable if it is treated appropriately [7]. The properties of vinasse are determined by the raw material utilized to produce ethanol (1G or 2G) [6,8]. Furthermore, the high organic matter concentration, heavy metals and salt, vinasse could cause significant pollution of both surface water and soil, and also causing greenhouse gas emissions into the atmosphere [3]. Vinasse is used for soil fertilization, or "fert irrigation," to enhance sugarcane agriculture due to its high organic load and nutritional content. However, in some regions, the potassium content standard (400 Kg - $K_2O.ha^{-1}$. $Year^{-1}$) is imposed [9]. Despite the economic interest of Fert irrigation compared to inorganic fertilizers, the release of vinasse on the soil could, in the long term, ultimately harm the sole-water system [1,10]. Indeed, because of the continuous utilization of the vinasse on the soil, there are environmental effects on the cultivated area [11]. That is why it is necessary to recover this waste properly before being released into the environment [3].

In this context, AD is a biochemical treatment that enables most organic waste and effluents to be recovered, producing biogas rich in methane, a gas with high energy potential [12–16]. Several recent studies insist on the importance of AD as a treatment procedure of vinasse as we could obtain both significant organic depollution and energy production from the recovery of biogas using single-phase digesters [1,17–19]. Due to has incredible content of easily degradable organic components, vinasse is a suitable substrate for AD [20,21]. The efficiency of AD for vinasse varies depending on the characteristics of the substrate employed in the production of ethanol which could be either sugarcane or bagasse or molasses (1G or 2G ethanol production) [6].

Several factors impact the AD process, and methane production is hindered when these factors are not optimal. The methods that consist of fixing one parameter and varying the others very often fail to optimize biogas production because they do not take account of the interactions that exist between these parameters. Like all biological processes, the problem of the efficiency of the AD process is often posed. For this reason, scientists have been exploring techniques that could result in the best biogas production possible for long. Numerous optimization techniques and approaches have been developed over time. Response Surface Methodology (RSM) is one of these methods. This method was developed to investigate how various variables affect one another and to calculate the optimal situation [22,23].

The RSM can be defined as a group of statistical and mathematical methods which are helpful for analysis and modelling in situations where one's response is affected by a number of factors and where the goal is to maximize this solution [24,25]. There are many benefits to using the RSM approach for optimization research, including a decrease in the number of experimental trials, which makes it more efficient in terms of both time and money [23]. However, the disadvantage of RSM is that it can only test a single objective hypothesis and cannot test numerous objective hypotheses [26,27]. In the case of AD, the goal of RSM is to maximize a response, or Y_{CH4}, which is modified by inputs data variables and will be optimized by this method. The necessity for lengthy processes and high expenses, which is a drawback of traditional optimization techniques, can be eliminated using the RSM method [28]. In other terms, RSM is used to assess the link between the yield of biogas and the independent factors, as well as to optimize the proper setting for the factors to be able to forecast the design and determine the optimum values for responses [29]. The RSM is employed to look into the relationships and input-output interactions elements and the output variables. An analysis of variance (ANOVA) was carried out to confirm the reliability and precision of the model [24,30].

The economic and societal impacts of AD of vinasse using RSM are significant, ranging from cost savings and environmental benefits to rural development and technology transfer. Optimizing this process holds great potential for sustainable development and resource utilization.

The aim of this study was to determine the effect of added loading, hydraulic retention time (HRT) and their interactions on the mesophilic AD of vinasse. In order to determine the optimal substrate conditions and to statistically analyse the experimental digestion data, the central composite design (CCD) RSM method was employed.

The design of experiments procedure is used to create a test. This methodology ensures the acquisition of maximum data while reducing the number of experimental experiments by predicting the system's response. Since CCD tests in harsh environments as well, it offers better results for quadratic models. Using the Box-Behnken design (BBD) and the face CCD involves fewer tests, reduces costs, requires less effort, and is quicker compared to a full factorial design. Although CCD requires more experiments than BBD, it remains highly effective. Consequently, BBD necessitates at least three factors, while CCD can manage with just two factors. These factors play a significant role in determining the suitability of using the CCD technique [27].

The novelty of this study lies in the application of the RSM to optimize sugarcane vinasse treatment factors. To our knowledge, no literature has explored this approach before. The main objective of the study was to apply RSM to optimize conditions and assess the impact of process factors, with HRT and additional load as variables. The CCD of RSM was used to evaluate interactions between independent variables and selected responses, in order to identify optimal conditions. These results provide new insights into the modelling and optimization of AD processes using RSM, particularly for vinasse.

2. Materials and methods

2.1. Substrate and inoculum used

Vinasse from the distillery was processed in the digester, which was infused with biomass from an industrial anaerobic reactor. The chosen inoculum has a high methanogenic activity, which the laboratory staffs have seen from earlier investigations. This biomass has a methanogenic flora that can break down the effluent; these microbes can hasten the AD's beginning [31]. The inoculum is kept at

Table 1
Physico-chemical parameters of inoculums and second generation ethanol vinasse.

Parameter	Inoculum	Substrate
pH	7.1 ± 0.1	4.1 ± 0.2
Total Solid TS (g/L)	25.08 ± 1.50	65.3 ± 2.1
Mineral Solid MS (g/L)	14.58 ± 1.22	21.65 ± 0.75
Volatile Solid VS (g/L)	10.5 ± 0.4	45.97 ± 2.13
VS (% TS)	42	68
%TS	2.2	5.82

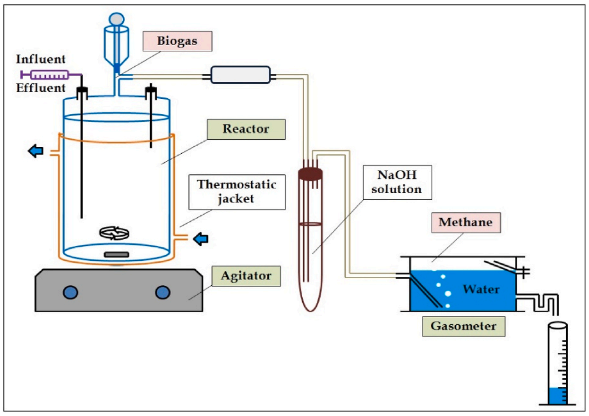


Fig. 1. Schematic diagram of experimental laboratory set up.

4 °C for a relatively brief period of time which might aid to prolong the methanogenic activity. Table 1 displays the properties of this inoculum. The inoculum's pH showed a value of 7.1 ± 0.1, which is close to neutral and provides insight into the handling's acidity and basicity. Mineral solid, overall solid and volatile solid all had concentrations of 10.5 g/L, 25.08 g/L, and 14.58 g/L, respectively. The 2G ethanol vinasse utilized in this investigation comes from an ethanol distillery where ethanol is made from sugar cane molasses. Table 1 displays the physico-chemical properties of this substrate.

2.2. Chemical analysis

Effluent parameters were determined for each load, including pH, moisture, volatile solids (VS) (g/L), total solids (TS) (g/L), and mineral solids (MS) (g/L) and alkalinity (Alk, mg $CaCO_3$/L). These analyses were carried out in accordance with the ''Standard Methods for the Examination of Water and Wastewater'' [32].

2.3. Experimental design

The lab-scale apparatus used for AD of vinasse is a Continuous Stirred-Tank Reactor (CSTR), specifically a Pyrex reactor that is continuously stirred and has a total volume of 1.5 L, including a 0.5-liter extra capacity for vacuum purposes. It is equipped with essential components such as a jacket for efficient hot water circulation, a sealing system, and a lid, enabling it to function as a fully stirred continuous reactor. The system includes a magnetic stirrer and a thermostat device controlling water temperature precisely at 37 °C, maintaining optimal mesophilic conditions throughout the experiment (Fig. 1).

Biogas produced passes through a bubbler containing a 6 N NaOH solution, purifying it by capturing CO_2 and allowing only methane to pass through. Subsequently, methane content is measured using a gasometer and the water displacement method. The volume of methane generated displaces water from the gasometer into a test tube, providing a visible indication of the methane volume produced. Taking into account the effect of atmospheric pressure, temperature, and water vapor pressure at ambient temperature during measurement, the cumulative methane results are normalized, with results expressed under standard conditions (0 °C and 760 mmHg). The temperature is determined, respectively by the thermometer, while the pressure is considered constant throughout the process, equal to atmospheric pressure. Using the law of perfect gases, the volume of methane produced under experimental conditions can be corrected and converted to normal temperature and pressure conditions.

2.4. Experimental procedure

The inoculum used in this study is from a mesophilic anaerobic digester in Kenitra City, Morocco, which handles sludge from wastewater treatment plants. Digesters are filled with this inoculum using a 1-liter. For duration of one week, inoculation takes place in

Table 2
The corresponding responses and the experimental design matrix.

Independent variables			Levels			
			−1	0		1
X1: Load (g VS/L)			0.5	3		5.5
X2: HRT (h)			1.5	16.75		32
			Y_{CH4} (Nml/g VS)		CMP (Nml)	
Run	X1	X2	Experimental value	Predicted value	Experimental value	Predicted value
1	3	1.5	83.33	72.14	125	172.52
2	5.5	32	185.81	178.07	1022	1015.95
3	5.5	16.75	129.09	144.33	710	722.12
4	0.5	16.75	198	183.55	99	161.72
5	3	32	202.85	217.17	608.559	634.75
6	0.5	1.5	59.184	72.27	29.5919	6.67
7	3	16.75	182.66	184.19	548	531.54
8	3	16.75	182.66	184.19	548	531.54
9	3	16.75	182.66	184.19	548	531.54
10	0.5	32	208.04	215.77	104.02	74.3
11	3	16.75	182.66	184.19	548	531.54
12	3	16.75	182.66	184.19	548	531.54
13	5.5	1.5	33.4	31.52	183.718	172.47

mesophilic AD conditions until complete depletion. After this time, the inoculum is ready to start breaking down the organic materials in the food waste that is the subject of this investigation.

A solution containing sodium acetate, Lactic acid, and glucose at 25 g/l, 25 g/l, and 50 ml/l, respectively, was used to ensure the bio-activation of the biomass. Over the course of 15 days, an organic load was gradually introduced to the reactor (in steps of 0.25 g/L to 1 g/L). Therefore, a process of acclimatizing the biomass is undertaken. The synthetic solution and vinasse effluent are then fed into the reactor, eventually raising the vinasse content to 100 %. During this stage, the amount of methane generated as a function of time is calculated. Until the generation of methane is stopped, each consignment's maximum time for the VS test at this stage is 23 h; this value is required for the degradation of organic matter and the biogas production. A series of batch mesophilic studies were conducted in the last phase, known as the treatment phase, loading the digester with 100 % of the substrate and gradually increasing the additional loads from 0.5 to 7 g VS/L. It should be emphasized that the reactor was fed in triplicate for each load during the acclimation and treatment phases. As soon as a phase was finished, methane production came to an end. Regarding the biodegradation of the vinasse substrate, only the methane produced during the treatment phase was taken into account [33]. Each substrate requires time to degrade, and the amount of time depends on the BOD/COD ratio and the biodegradability of the feedstock. With sample collection and analysis for the parameters: pH, alkalinity, TS, and VS, methane volume is computed as a function of the amount fed and the amount of time before and after.

2.5. Response surface methodology

For determining the links between the answer and the independent variables, RSM combines mathematical and statistical methodologies. The impact of either singularly or collectively on the processes is defined by RSM. This experimental setup creates a mathematical model and investigates the effects of variables. The mathematical model's graphical perspective inspired the creation of the response surface approach [34].

RSM allows for the establishment of three phases for an optimization research investigation. The preliminary work required to identify the independent criteria and their levels constitutes the first phase. The next step entails selecting an experimental design as well as predicting and validating the model equation. The last phases involve locating the optimum areas and creating a response surface plot and contour plot as a function of the independent factors. The information for each stage is provided below. The parameters for the study are the load (X_1), 0.5–5.5 g VS/L, and the HRT (X_2), 1.5–32 h. Table 2 shows the three levels of coding for each independent variable in the central composite design (CCD): lowest (−1), center (0), and highest (1).

The polynomial equation shown below was fit to the experimental results using a multiple regression technique (Eq. (1)):

$$Y = b_0 + b_1 X_1 + b_2 X_2 + b_{12} X_1 X_2 + b_{11} X_1^2 + b_{22} X_2^2 \quad \text{(Eq. 1)}$$

2.6. Analysis of variance (ANOVA) for quadratic model

A lack of fit test that is iterative, regression analysis (using measures such as R^2, adequacy precision ratio, and adjusted R^2), and (ANOVA) were utilized to evaluate the model's suitability. It was only judged acceptable within the confidence interval when the model's and each term's p values were less than the significance level ($p = 0.05$). Paired t-tests were used to statistically analyse the discrepancies between the projected results and the validation experiment data, and independent validation experiments further validated the proposed models [35].

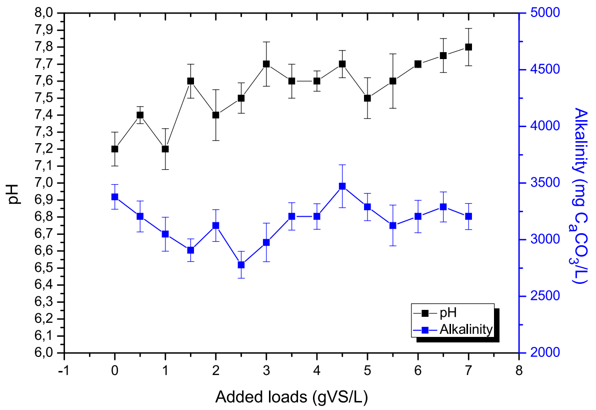


Fig. 2. pH and Alkalinity variation during the AD of vinasse as a function of the added loads.

Table 3
Comparison between methane yield obtained from the experience and bibliography.

References	*Methane yield* (mL_{STP} CH_4/g VS)
In this study	205
[9]	102 to 203
[46]	369

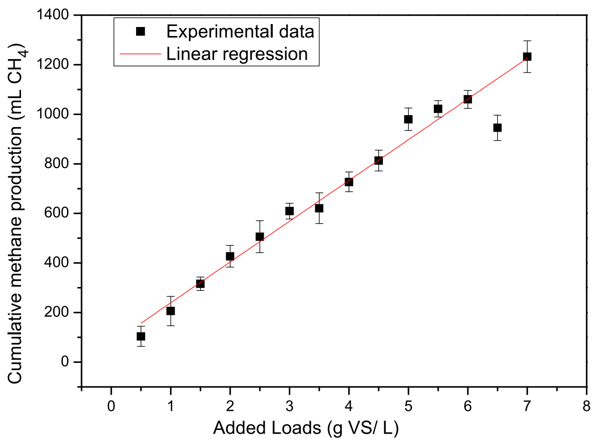


Fig. 3. Cumulative methane volume versus added loads (g SV/L).

3. Results and discussion

3.1. Process stability

As well as bacteria are sensitive to variations in pH and alkalinity, this can lead to unstable AD process. As a result, pH and alkalinity measurements were made during the addition of loads in order to assess the process' stability. Fig. 2 shows the evolution of pH and alkalinity for each added load of the vinasse. These values are in line with AD conditions [33].

The pH evolution as a function of the added loads (Fig. 2) shows that the pH varied between 7.1 and 8 with a mean value of 7.58. This variation of the pH is very close to the range of 7 and 7.8 which is stated in the literature as the optimum for AD [36]. This stability in pH was probably due to the formation of the Total Inorganic Carbone [37]. Beside the pH, the alkalinity is another stability parameter for AD. It is generally related to the buffer capacity of the anaerobic process. Some research considers alkalinity to be a more accurate indicator than pH for assessing an imbalance in the AD process [38,39]. According to the fluctuation of alkalinity as a function of added load (Fig. 2), the process functioned favourably without risk of acidification, which can result in unstable AD. The alkalinity was higher in the loads between (7 g VS/L), exhibiting values in the range of 3000 and 3450 mg CaCO3/L. These results were in the line with literature in which it indicated that the alkalinity should not be lower than 1000 mg $CaCO_3$/L [39,40].

Table 4
ANOVA for the fitted quadratic model for CMP.

Source	Sum of squares	df	Mean square	F value	P value	Remark
Model	1.049E+06	5	2.099E+05	137.29	<0.0001	Significant
X1-Load	4.721E+05	1	4.721E+05	308.84	<0.0001	
X2- HRT	3.249E+05	1	3.249E+05	212.55	<0.0001	
X1X2	1.459E+05	1	1.459E+05	95.42	<0.0001	
X^21	22196.20	1	22196.20	14.52	0.0066	
X^22	44804.76	1	44804.76	29.31	0.0010	
Residual	10701.15	7	1528.74			
Lack of fit	10701.15	3	3567.05			
Pure error	0.000	4	0.000			
Cor total	1.060E+06	12				

R^2: 0.9899 R^2_{adj}: 0.9827 Adeq precision: 38.64 CV (%): 9.04 SD: 39.10 Mean: 432.45.

Table 5
ANOVA for the fitted quadratic model for YCH4.

Source	Sum of squares	df	Mean square	F value	P value	Remark
Model	40502.75	5	8100.55	56.78	<0.0001	Significant
X1-Load	2278.54	1	2278.54	15.97	0.0052	
X2- HRT	29510.14	1	29510.14	206.85	<0.0001	
X1X2	3.16	1	3.16	0.0221	0.8859	
X^21	1133.39	1	1133.39	7.94	0.0258	
X^22	4577.87	1	4577.87	32.09	0.0008	
Residual	998.63	7	142.66			
Lack of fit	998.63	3	332.88			
Pure error	0.000	4	0.000			
Cor total	41501.37	12				

R^2: 0.9759 R^2_{adj}: 0.9587 Adeq precision: 22.29 CV (%): 7.71 SD: 11.94 Mean: 154.85.

The Y_{CH4} of vinasse in this study was important and similar results were found in the bibliography (Table 3). The study by Jiménez et al., the maximum Y_{CH4} of beet molasses alcoholic fermentation wastewater using anaerobic biodegradation is about 203 mLCH_4/g VS) [9]. Also the study by Siles et al., observed that the best value of Y_{CH4} is about 369 mL CH_4/g VS using biomethanization of vinasse derived from ethanol manufacturing [41].

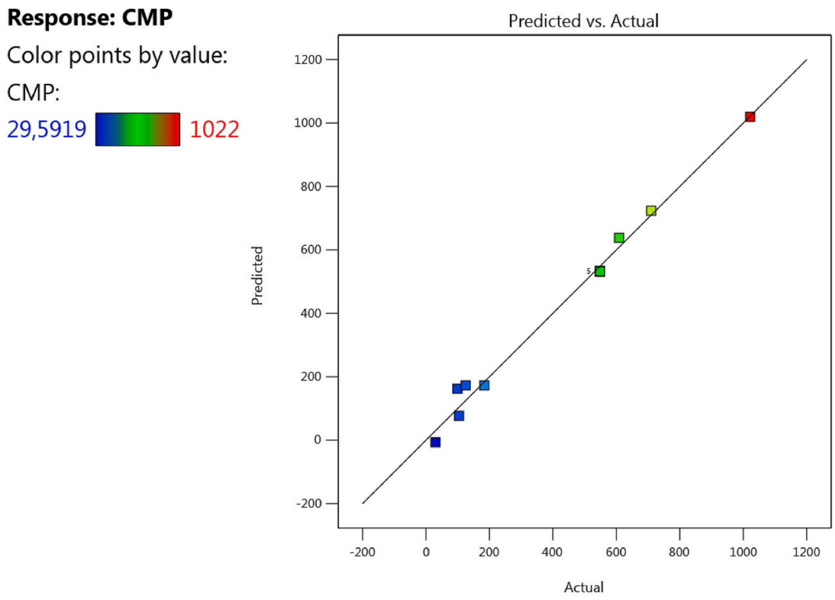
3.2. Methane yield and biodegradability

The CMP and the additional VS, which were known for all loads, were used to calculate the Y_{CH4} [41]. As shown in Fig. 3, by fitting the pairs of values (CMP, VS added) to a linear regression, the Y_{CH4} coefficient is found by determining the slope of this straight line. The Y_{CH4} was found to be 205 mLCH_4/g VS added (at 1atm, 0 °C).

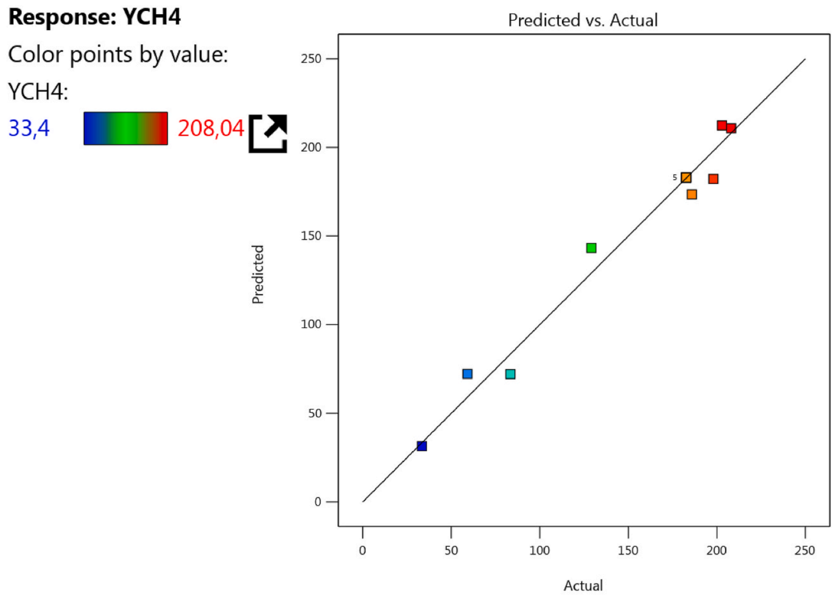
According to Fig. 3, the volume of methane production rose as the added load increased (from 0.5g to 7g VS added). Methane, which is produced as a result of the decomposition of the additional accessible organic material, is the cause of these phenomena. Due to its caloric power, which is used to produce the useful product methane (Lower Caloric Power: 35,793 kJ/m^3, equivalent to 9.96 kWh/m^3), the degraded VS is used [41], The primary goal of microbial metabolism is to produce gas. No suppression of the methane generation was observed, and the biodegradability of the vinasse under the investigated conditions was identified as another crucial operational variable. The definition of biodegradability was based on the interaction between the VS removed and the VS supplied to the digesters. The vinasse showed an average biodegradability value of 94 % (in VS) along the AD process.

3.3. Analysis of variance

To test hypotheses regarding the model's variable, the analysis of variance (ANOVA) is used [42]. The significant ratio of the mean square (MS) variance generated from the regression to the residual error of the mean square was examined using an ANOVA. The results showed in Tables 4 and 5 are for the Y_{CH4} and CMP ANOVAs, respectively. The lacks of fit for Y_{CH4} and CMP are 998.63 and 10701.15 respectively. Notable F-values for Y_{CH4} and CMP are 56.78 and 137.29. Kainthola et al. uses the model's F-value of 33.04, which shows that it is significant, to demonstrate how useful it is. However, the model can only forecast 0.01 % of that big a number with accuracy. An F-value of this magnitude can come from both the signal and the noise. This F value indicates the lack of fit, and it's about 1427.02, indicates that it is most likely far larger than the pure error [43]. The coefficients of determination R^2 for Y_{CH4} and CMP are 0.9759 and 0.9899 respectively, indicating the model's ability to match the data. For the study by Habchi el al. the R^2 is about 0.9710 using also RSM-CCD for AD of poultry slaughterhouse waste [44]. The ANOVA for each pertinent response anticipated that the models were significant based on the p values ($p < 0.05$) of the models and the insignificant p values ($p > 0.05$) of the lack of fit test.



(a)



(b)

Fig. 4. Scatter diagram of predicted response versus actual for (a) CMP (b) Y_{CH4}.

3.4. Regression model development

In this work, a quadratic model was constructed utilizing information from all CCD trials, and the response were fitted using Design-Expert software, Version 13 (DX13). In this part, the results of the fitting are displayed as an ANOVA with an Y_{CH4} and CMP. Eq. (2) and Eq. (3), for Y_{CH4} and CMP respectively, indicate the results of the quadratic modeling. Regression analysis enables us to calculate the coefficient b for the response based on the observed data using statistical methods. It proves that the second-order polynomial model's Eq. (2) and Eq. (3) were crucial and correctly predicted the experiment's outcomes.

$$Y_{CH_4} = 52.23 + 11.26*[Load] + 10.39*[HRT] + 0.02*[Load*HRT] - 3.24*[Load]^2 - 0.17*[HRT]^2 \quad \text{(Eq. 2)}$$

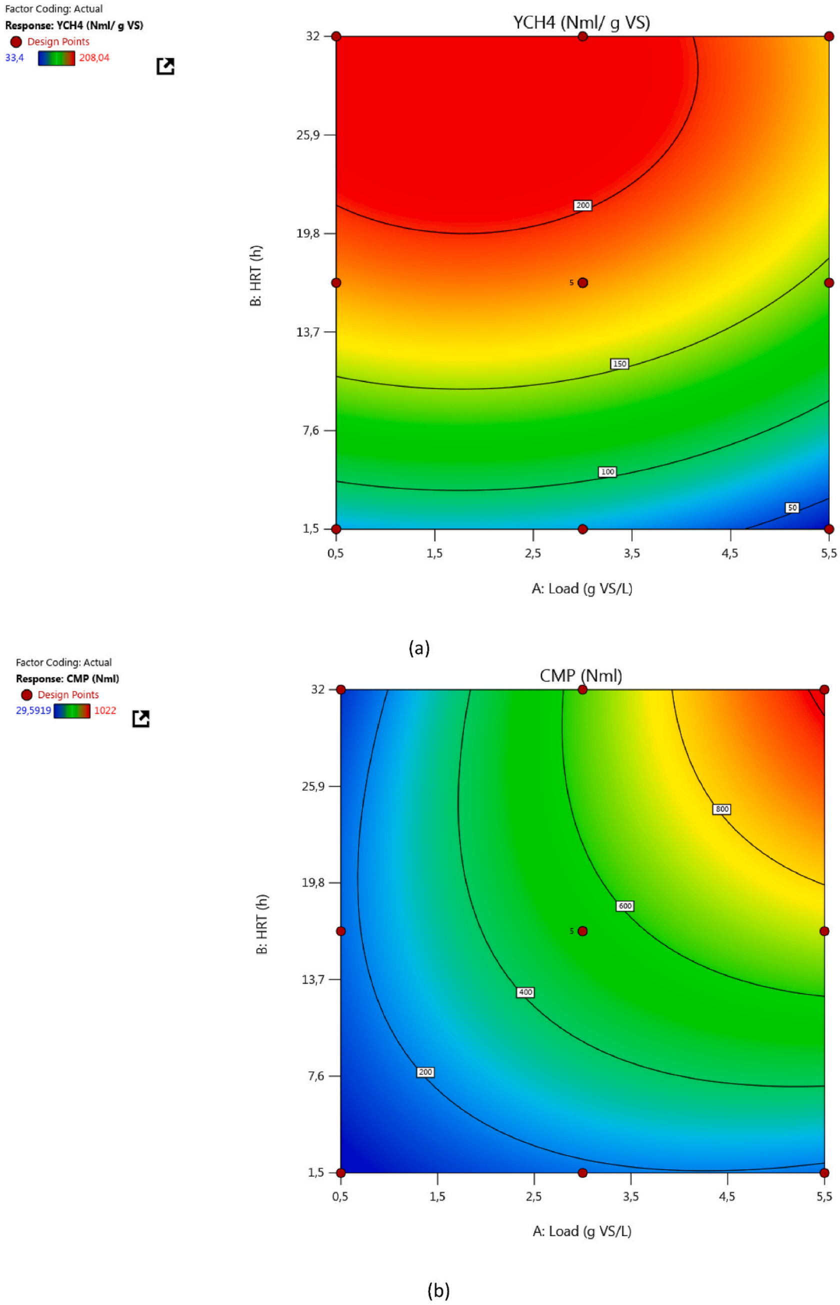
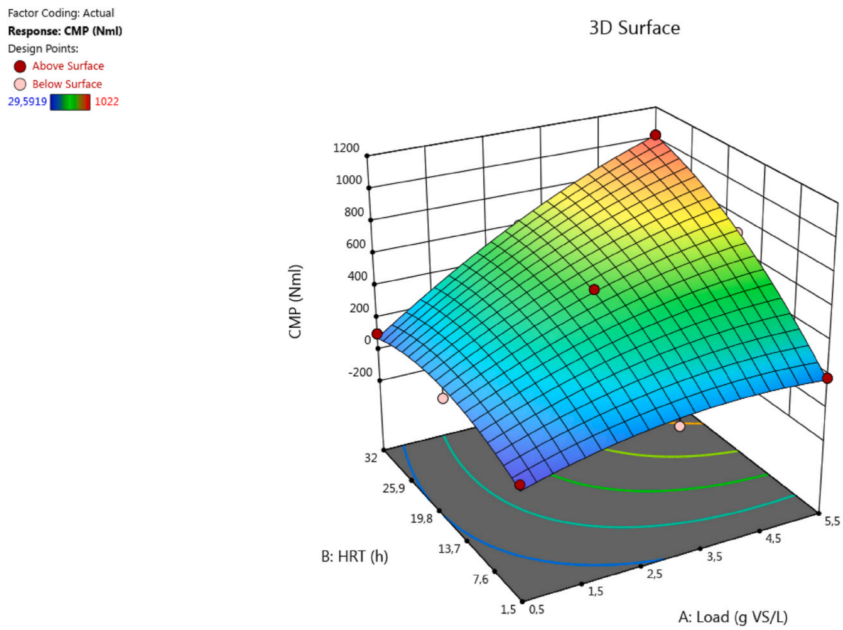


Fig. 5. Contour plot showing the influence of Load and HRT on (a) Y_{CH4} (b) CMP.

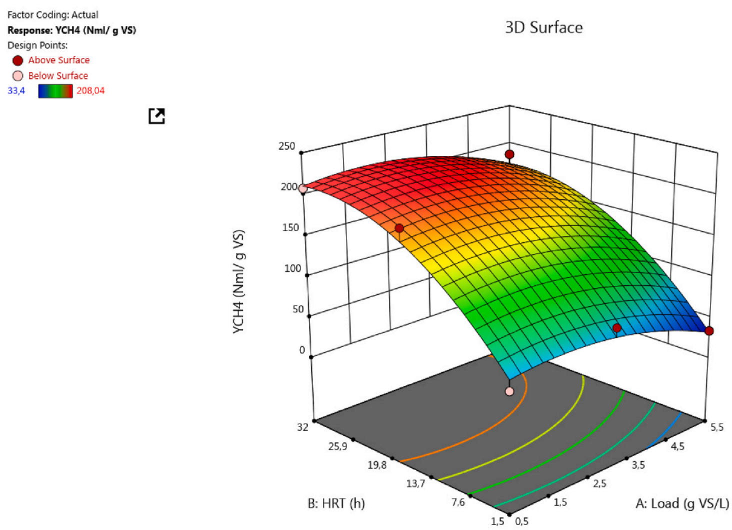
$$CMP = -90.66 + 114.37*[Load] + 18.58*[HRT] + 5*[Load*HRT] - 14.34*[Load]^2 - 0.55*[HRT]^2 \quad \text{(Eq. 3)}$$

3.5. Model accuracy assessment

Validating the proposed model's accuracy is an essential step in the analytical process. Using an inadequate approximation model when simulating the real system can result in inaccurate or deceptive outcomes (Fig. 4) [45]. As illustrated in Fig. 4a, the experimental data points exhibited greater dispersion within the consistent range of residuals compared to those in Fig. 4b.



(a)

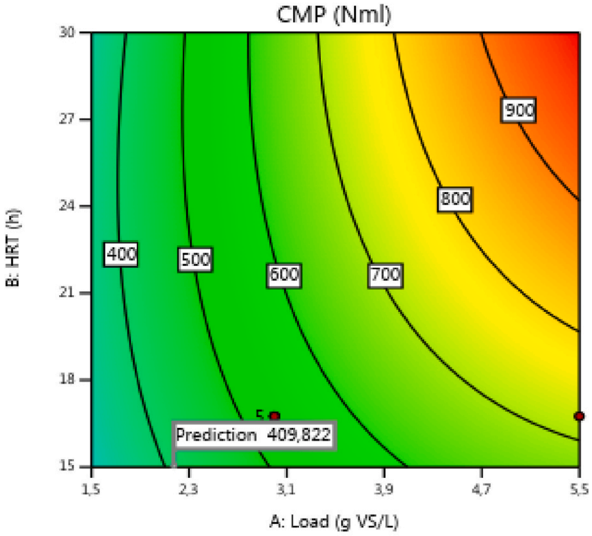


(b)

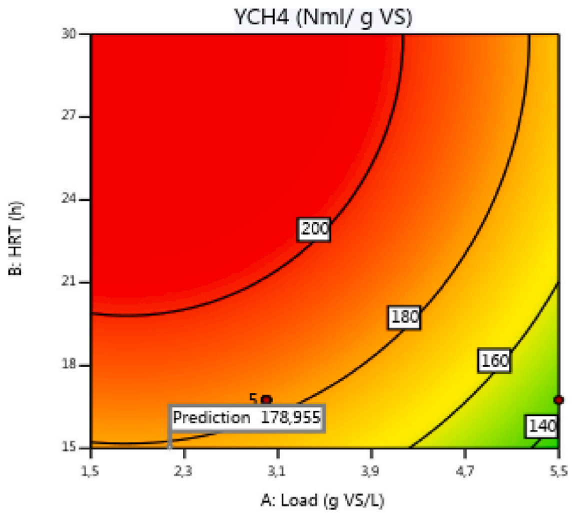
Fig. 6. Response surface plot showing the influence of Load and HRT on CMP (a) Y_{CH4} (b).

3.6. *Interactive effect of variables on CMP and Y_{CH4}*

The effect of vinasse loading and HRT interaction on Y_{CH4} and CMP is seen in Fig. 5. Increased vinasse load and HRT led to an increase in Y_{CH4} and CMP, but these concentrations fell as vinasse load continued to rise. The contour plot revealed that the interaction between load and HRT had a significant ($P < 0.0001$) impact on CMP (Fig. 5a) but no discernible ($P > 0.05$) impact on Y_{CH4} (Fig. 5b). However, each subject was significantly ($P < 0.0001$) impacted by the HRT. The response surface 3D plot is showed in Fig. 6. The study by Kainthola et al. demonstrates that the interaction between factors (pH and feedstock/microorganisms ration) was insignificant while the individual parameters are significant ($P < 0.05$) for AD of rice straw and hydrilla verticillata [43].



(a)



(b)

Fig. 7. Numerical optimization of process variables for CMP (a) and Y_{CH4} (b).

3.7. *Numerical modeling of variables used*

The ideal conditions for the variables were determined using a numerical optimization study using the developed models, which represent the entire design area. As a result, the ideal ranges for the elements that will result in the greatest output of methane are determined. The Design Expert model, the AD of vinasse, and other techniques were used in the study to determine the Y_{CH4} and CMP. To select the optimal optimization, the upper limit of the process load and the lower limit of the HRT were carefully chosen. Fig. 7 shows the best maximal response parameters with a load of 2.17 g VS/L and an HRT of 15.06 h, with values of 409.82 ml for CMP (Figs. 7a) and 178.95 ml/g VS for Y_{CH4} (Fig. 7b), respectively. The study findings indicated that achieving optimal methane production conditions requires approximately one day of experimentation, providing sufficient time to achieve satisfactory results. These outcomes are applicable in industrial settings to ensure the efficient operation of AD processes for vinasse. Also the study by Habchi et al. found that the optimal CMP and Y_{CH4} are observed for 1.6 g VS/L added load and 108 h with values of 343 mL and 224 mL/g VS, respectively [44].

4. Study limitations

AD of vinasse, while promising for biogas production, faces several limitations influenced by the varied compositions of vinasse from different sources. Vinasse, a byproduct of ethanol production from sugarcane, can vary significantly in its chemical composition depending on factors such as the type of raw material used, fermentation processes, and post-fermentation treatments. These variations present challenges in consistently optimizing AD processes.

One significant limitation is the high organic loading and nutrient content in vinasse. While this can initially seem beneficial for biogas production, excessive nutrient levels, particularly potassium, can inhibit microbial activity in anaerobic digesters. This inhibition can lead to process instability, decreased methane yield, and prolonged digestion times. Conversely, vinasse with lower nutrient levels may require additional supplementation to support microbial growth and methane production, adding complexity and cost to the process.

Moreover, the acidic nature of vinasse, often characterized by low pH values, poses another challenge. Anaerobic bacteria are sensitive to pH fluctuations, and acidic conditions can suppress their activity, thereby reducing biogas production efficiency. Adjusting pH levels through neutralization or buffering agents is often necessary but adds to operational costs and requires careful monitoring to maintain optimal conditions.

Another critical factor is the presence of recalcitrant compounds in vinasse, such as lignin and certain phenolic compounds. These compounds are resistant to microbial degradation under anaerobic conditions, leading to incomplete digestion and the accumulation of organic matter in digesters. Over time, this accumulation can result in process inhibition and reduced biogas yield, necessitating periodic cleaning and maintenance of digesters.

Variability in vinasse composition, including differences in organic matter, pollutant levels, and pH, can significantly impact the efficiency and stability of AD at an industrial scale. This variability may affect methane production, process stability, and operational costs. To manage these challenges, regular monitoring and analysis of vinasse composition are essential, allowing for adjustments in process parameters and feeding strategies. Implementing automated systems, advanced control strategies, blending, and conditioning of vinasse can help maintain process stability. Additionally, adaptive feeding strategies and microbial community management further ensure consistent performance and optimize both environmental and economic outcomes.

5. Conclusion

This study effectively utilized RSM to model and optimize key parameters for AD of vinasse, focusing on enhancing biogas production. The optimized conditions substrate load of 2.17 g VS/L and HRT of 15 h resulted in substantial improvements, with methane yield reaching 178.95 ml/g VS and cumulative methane production at 409.82 Nml. The high R^2 values from ANOVA validate the model's predictive accuracy, demonstrating RSM's robustness in optimizing biogas production. These findings have significant practical implications for industrial-scale applications as RSM reduces the need for extensive experimental trials, saves time, and cuts costs, making it an invaluable tool for improving the efficiency of biogas production systems. By streamlining the optimization process, RSM supports the advancement of bioenergy research and its practical deployment in sustainable energy solutions.

Data availability

Data will be made available on request.

CRediT authorship contribution statement

Hassan El Bari: Supervision, Methodology. **Sanae Habchi:** Writing – original draft, Visualization, Supervision, Formal analysis.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

References

[1] B.S. Moraes, T.L. Junqueira, L.G. Pavanello, O. Cavalett, P.E. Mantelatto, A. Bonomi, M. Zaiat, Anaerobic digestion of vinasse from sugarcane biorefineries in Brazil from energy, environmental, and economic perspectives: profit or expense? Appl. Energy 113 (2014) 825–835, https://doi.org/10.1016/j.apenergy.2013.07.018.

[2] J.A. Siles, I. García-García, A. Martín, M.A. Martín, Integrated ozonation and biomethanization treatments of vinasse derived from ethanol manufacturing, J. Hazard Mater. 188 (2011) 247–253, https://doi.org/10.1016/j.jhazmat.2011.01.096.

[3] Sugar Processing Research Institute, in: Frontiers in sugar processing: proceedings of the 2006 Sugar Processing Research Conference ; September 17 - 20, 2006, Águas de São Pedro, Brazil ; [SPRI 2006], Sugar Processing Research Inst, 2006. New Orleans, La.

[4] H. Caillet, D.M. Madyira, L. Adelard, Study of the performances of a vinasse mesophilic anaerobic digester behavior submitted to intermittent mixing: monitoring of the physicochemical properties of the digestate and local samples of the digester, Bioresour. Technol. Rep. 16 (2021) 100837, https://doi.org/10.1016/j.biteb.2021.100837.

[5] V.R. Moreira, T.G. Carpanez, F.S. dos Santos, L.S. Santos, D. dos S. Fernandes, L.S. França-Neta, L.C. Lange, M.C.S. Amaral, Circular economy in biorefineries: scale-up of anaerobic/aerobic membrane bioreactors for vinasse recycling, J. Clean. Prod. 377 (2022) 134448, https://doi.org/10.1016/j.jclepro.2022.134448.

[6] P.S. Santos, M. Zaiat, C.A. Oller do Nascimento, L.T. Fuess, Does sugarcane vinasse composition variability affect the bioenergy yield in anaerobic systems? A dual kinetic-energetic assessment, J. Clean. Prod. 240 (2019) 118005, https://doi.org/10.1016/j.jclepro.2019.118005.
[7] I.Z. Pereira, I.F.S. dos Santos, R.M. Barros, H.L. de Castro e Silva, G.L. Tiago Filho, A.P. Moni e Silva, Vinasse biogas energy and economic analysis in the state of São Paulo, Brazil, J. Clean. Prod. 260 (2020) 121018, https://doi.org/10.1016/j.jclepro.2020.121018.
[8] J.M. Sonawane, R. Mahadevan, A. Pandey, J. Greener, Recent progress in microbial fuel cells using substrates from diverse sources, Heliyon 8 (2022) e12353, https://doi.org/10.1016/j.heliyon.2022.e12353.
[9] A.M. Jiménez, R. Borja, A. Martın, Aerobic–anaerobic biodegradation of beet molasses alcoholic fermentation wastewater, Process Biochem. 38 (2003) 1275–1284, https://doi.org/10.1016/S0032-9592(02)00325-4.
[10] J. Ariunbaatar, A. Panico, G. Esposito, F. Pirozzi, P.N.L. Lens, Pretreatment methods to enhance anaerobic digestion of organic solid waste, Appl. Energy 123 (2014) 143–156, https://doi.org/10.1016/j.apenergy.2014.02.035.
[11] A.M. Jiménez, R. Borja, A. Martın, Aerobic–anaerobic biodegradation of beet molasses alcoholic fermentation wastewater, Process Biochem. 38 (2003) 1275–1284, https://doi.org/10.1016/S0032-9592(02)00325-4.
[12] K. Hamraoui, A. Gil, H. El Bari, J. Siles Lopez, A. Chica, M. Martín, Evaluation of hydrothermal pretreatment for biological treatment of lignocellulosic feedstock (pepper plant and eggplant), Waste Manag. 102 (2020) 76–84, https://doi.org/10.1016/j.wasman.2019.10.020.
[13] S. Habchi, L. Nabila, K. Fadoua, N. Ikram, L. Yahya, B. Mohammed, S. Brahim, E.B. Hassan, Effect of thermal pretreatment on the kinetic parameters of anaerobic digestion from recycled pulp and paper sludge, Ecol. Eng. Environ. Technol. 23 (2022) 192–201, https://doi.org/10.12912/27197050/143568.
[14] M. Bakraoui, F. Karouach, B. Ouhammou, N. Lahboubi, Y.E. Gnaoui, O. Kerrou, M. Aggour, H. El Bari, Kinetics study of methane production from anaerobic digestion of sludge and wastewater recycled pulp and paper, IOP Conf. Ser. Mater. Sci. Eng. 946 (2020) 012009, https://doi.org/10.1088/1757-899X/946/1/012009.
[15] S. Habchi, N. Lahboubi, H. El Bari, Valorization of Moroccan poultry slaughterhouse waste using anaerobic digestion: kinetic study, Waste Biomass Valorization (2024), https://doi.org/10.1007/s12649-024-02450-2.
[16] N. Lahboubi, S. Habchi, K. Hamraoui, H.E. Bari, New insight of combined hydrothermal and ultrasonic pretreatment for methane production from anaerobic digestion of food waste, Int. J. Green Energy. 21 (2024) 3101–3111. https://www.tandfonline.com/doi/abs/10.1080/15435075.2024.2352498.
[17] H. El Bari, S. Habchi, Toward biomass energy development in africa: opportunities and obstacles, Eur. Biomass Conf. Exhib. Proc. 31st EUBCE-Bologna 2023 (2023) 1149–1154, https://doi.org/10.5071/31stEUBCE2023-ICO.4.3.
[18] L.T. Fuess, M.A. Piffer, M. Zaiat, C.A.O. do Nascimento, Phase separation enhances bioenergy recovery in sugarcane vinasse biodigestion: absolute or relative truth? Bioresour. Technol. Rep. 18 (2022) 101026 https://doi.org/10.1016/j.biteb.2022.101026.
[19] S. Mohana, B.K. Acharya, D. Madamwar, Distillery spent wash: treatment technologies and potential applications, J. Hazard Mater. 163 (2009) 12–25, https://doi.org/10.1016/j.jhazmat.2008.06.079.
[20] A.D. Nunes Ferraz Júnior, P.G. Machado, F. Jalil-Vega, S.T. Coelho, J. Woods, Liquefied biomethane from sugarcane vinasse and municipal solid waste: sustainable fuel for a green-gas heavy duty road freight transport corridor in Sao Paulo state, J. Clean. Prod. 335 (2022) 130281, https://doi.org/10.1016/j.jclepro.2021.130281.
[21] L. Sillero, R. Solera, M. Perez, Effect of temperature on biohydrogen and biomethane production using a biochemical potential test with different mixtures of sewage sludge, vinasse and poultry manure, J. Clean. Prod. 382 (2023) 135237, https://doi.org/10.1016/j.jclepro.2022.135237.
[22] D.M. Brown, A.M. Grunden, J.J. Pawlak, Statistical optimization of black liquor-containing media for growth and lactic acid production by Paenibacillus glucanolyticus SLM1, Bioresour. Technol. Rep. 13 (2021) 100629, https://doi.org/10.1016/j.biteb.2021.100629.
[23] S.S. Djimtoingar, N.S.A. Derkyi, F.A. Kuranchie, J.K. Yankyera, A review of response surface methodology for biogas process optimization, Cogent Eng 9 (2022) 2115283, https://doi.org/10.1080/23311916.2022.2115283.
[24] E.K. Armah, M. Chetty, N. Deenadayalu, Biogas production from sugarcane bagasse with South African industrial wastewater and novel kinetic study using response surface methodology, Sci. Afr. 10 (2020) e00556, https://doi.org/10.1016/j.sciaf.2020.e00556.
[25] N. Thi Thuy Van, P. Gaspillo, H.G.T. Thanh, N.H.T. Nhi, H.N. Long, N. Tri, N. Thi Truc Van, T.-T. Nguyen, H. Ky Phuong Ha, Cellulose from the banana stem: optimization of extraction by response surface methodology (RSM) and charaterization, Heliyon 8 (2022) e11845, https://doi.org/10.1016/j.heliyon.2022.e11845.
[26] J. Kainthola, A.S. Kalamdhad, V.V. Goud, Optimization of methane production during anaerobic co-digestion of rice straw and hydrilla verticillata using response surface methodology, Fuel 235 (2019) 92–99, https://doi.org/10.1016/j.fuel.2018.07.094.
[27] S. Habchi, N. Lahboubi, M. Asbik, H.E. Bari, Enhancing biomethane production from food waste using olive pomace hydrochar: an optimization study, Environ. Adv. 15 (2024) 100477, https://doi.org/10.1016/j.envadv.2023.100477.
[28] Y. Mortezaei, T. Amani, Sh Elyasi, F. Akhlaghian, High-rate anaerobic digestion of yogurt wastewater in a hybrid EGSB and fixed-bed reactor: optimizing through response surface methodology, Process Saf. Environ. Protect. 113 (2018) 255–263, https://doi.org/10.1016/j.psep.2017.10.012.
[29] S. Sathish, S. Vivekanandan, Parametric optimization for floating drum anaerobic bio-digester using response surface methodology and artificial neural network, Alex. Eng. J. 55 (2016) 3297–3307, https://doi.org/10.1016/j.aej.2016.08.010.
[30] R. Sirisangsawang, N. Phetyim, Optimization of tannin extraction from coconut coir through response surface methodology, Heliyon 9 (2023) e13377, https://doi.org/10.1016/j.heliyon.2023.e13377.
[31] S. Belhadj, F. Karouach, H. El Bari, Y. Joute, The biogas production from mesophilic anaerobic digestion of vinasse, IOSR-JESTFT 5 (2013) 72–77.
[32] Standard methods: for the examination of water and wastewater. https://law.resource.org/pub/us/cfr/ibr/002/apha.method.3111.1992.html. (Accessed 5 September 2023).
[33] N. Lahboubi, O. Kerrou, F. Karouach, M. Bakraoui, A. Schüch, K. Schmedemann, W. Stinner, H. El Bari, A. Essamri, Methane production from mesophilic fed-batch anaerobic digestion of empty fruit bunch of palm tree, Biomass Convers. Biorefinery (2020), https://doi.org/10.1007/s13399-020-00864-1.
[34] D. Baş, İ.H. Boyacı, Modeling and optimization I: usability of response surface methodology, J. Food Eng. 78 (2007) 836–845, https://doi.org/10.1016/j.jfoodeng.2005.11.024.
[35] B. Gunes, J. Stokes, P. Davis, C. Connolly, J. Lawler, Optimisation of anaerobic digestion of pot ale after thermochemical pre-treatment through Response Surface Methodology, Biomass Bioenergy 144 (2021) 105902, https://doi.org/10.1016/j.biombioe.2020.105902.
[36] M.C. Sterling, R.E. Lacey, C.R. Engler, S.C. Ricke, Effects of ammonia nitrogen of H2 and CH4 production during anaerobic digestion of dairy cattle manure, Bioresour. Technol. 77 (2001) 9–18, https://doi.org/10.1016/s0960-8524(00)00138-3.
[37] M.A. Martın, F. Raposo, R. Borja, A. Martın, Kinetic study of the anaerobic digestion of vinasse pretreated with ozone, ozone plus ultraviolet light, and ozone plus ultraviolet light in the presence of titanium dioxide, Process Biochem. 37 (2002) 699–706, https://doi.org/10.1016/S0032-9592(01)00260-6.
[38] M. Bakraoui, F. Karouach, B. Ouhammou, M. Aggour, A. Essamri, H. El Bari, Kinetics study of the methane production from experimental recycled pulp and paper sludge by CSTR technology, J. Mater. Cycles Waste Manag. 21 (2019) 1426–1436, https://doi.org/10.1007/s10163-019-00894-6.
[39] M. Bakraoui, F. Karouach, B. Ouhammou, H. El Bari, Experimental biogas production from recycled pulp and paper wastewater by biofilm technology, Biotechnol. Lett. 41 (2019) 1299–1307, https://doi.org/10.1007/s10529-019-02735-w.
[40] Y. Joute, Semi-continuous anaerobic co-digestion of cow manure and banana waste: effects of mixture ratio, Appl. Ecol. Environ. Res. 14 (2016) 337–349, https://doi.org/10.15666/aeer/1402_337349.
[41] J.A. Siles, I. García-García, A. Martín, M.A. Martín, Integrated ozonation and biomethanization treatments of vinasse derived from ethanol manufacturing, J. Hazard Mater. 188 (2011) 247–253, https://doi.org/10.1016/j.jhazmat.2011.01.096.
[42] L. Huiping, Z. Guoqun, N. Shanting, L. Yiguo, Technologic parameter optimization of gas quenching process using response surface method, Comput. Mater. Sci. 4 (2007) 561–570, https://doi.org/10.1016/j.commatsci.2006.03.014.

[43] J. Kainthola, A.S. Kalamdhad, V.V. Goud, Optimization of methane production during anaerobic co-digestion of rice straw and hydrilla verticillata using response surface methodology, Fuel 235 (2019) 92–99, https://doi.org/10.1016/j.fuel.2018.07.094.

[44] S. Habchi, N. Lahboubi, B. Sallek, H. El Bari, Response surface methodology for anaerobic digestion of waste from poultry slaughterhouse: optimization of load and hydraulic retention time, Results Eng (2023) 101215, https://doi.org/10.1016/j.rineng.2023.101215.

[45] B.K. Körbahti, M.A. Rauf, Application of response surface analysis to the photolytic degradation of Basic Red 2 dye, Chem. Eng. J. 138 (2008) 166–171, https://doi.org/10.1016/j.cej.2007.06.016.