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Influence of volatile fatty acids/alkalinity ratio on methane production during mesophilic anaerobic digestion: stability, efficiency and optimization

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ABSTRACT

Biogas production from sugarcane vinasse has enormous economic, energy, and environmental management potential. However, methane production stability remains key issue, hindered by the presence of inhibitory compounds and other characteristics of the vinasse such as volatile fatty acids (VFA), alkalinity and pH. These parameters are identified as potential auxiliary indicators for diagnosing imbalances in the anaerobic digestion (AD) reactor. This study investigates the influence of VFA/alkalinity ratio on reactor stability, efficiency and optimization of continuous AD of vinasse during mesophilic conditions in terms of methane yield production. To this end, a combined experimental and simulation approach is adopted using the response surface methodology (RSM). Results reveal that for VFA/alkalinity ratio in the range of 0.23–0.3, a higher methane yield is obtained reaching up to 4.0 L CH_4/L with an organic loading rate of 4.8 kg/m^3 d and a VFA less than 30 meq/L. The result of RSM verifies that the methane yield are affected mainly by operating conditions: VFA/alkalinity ratio (*A*), VFA (*B*) and pH (*C*).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 average maximum value of the produced methane is obtained for the following optimum conditions: pH of 7.2, VFA of 10 d, and the VFA/alkalinity ratio of 0.3. These findings provide a scientific foundation for predicting the behavior of anaerobic systems and optimizing the digestion process.

Keywords: Vinasse; Anaerobic digestion; Methane yield; Volatile fatty acid; Stability; Optimization; Response surface methodology (RSM)

1. Introduction

The anaerobic digestion (AD) of organic wastewater is a sustainable management strategy that is gaining significance due to the increasing costs of fossil fuels, the need to mitigate anthropogenic global warming. Biogas production from various types of raw materials (e.g., vinasse, manure, sewage sludge, food waste) has been shown to be a source of renewable energy that can occur sustainably in many different countries throughout the world [1,2]. The main products of AD are biogas, which is a mixture of gases, mainly methane (CH_4) and carbon dioxide (CO_2), and a nutrient rich effluent that can be used as a fertilizer [3]. AD has been used for decades as a waste stabilization

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process, but more recently, there has been increased interest on its potential for bioenergy production [4], as the biogas, composed of 40%–70% CH_4, can be either burn directly in a clean way for heat and power generation, or upgraded to be used as a transportation fuel [5].

AD involves a series of metabolic reactions (hydrolysis, acidogenesis, acetogenesis and methanogenesis) [6,7]. Among these intermediate products of AD, acetic acid, butyric acid, isobutyric acid, isovaleric acid, and propionic acid are known as good indicators for monitoring performance of AD process, especially in the activity of acetogenic and methanogenic bacteria [8–10]. Additionally, various physico-chemical parameters such as pH, temperature, alkalinity, volatile fatty acids (VFA), hydraulic retention time (HRT), organic loading rate (OLR), etc. influence these reactions [11,12]. The complexity of the process makes it difficult to interpret stability and performance of the process; therefore, a combination of these parameters was suggested as a better way and procedure for monitoring the performance of the process [13].

Indeed, accumulation of VFA decreases pH resulting in acidification [14]. In this case, alkalinity forms the ultimate medium to neutralize the VFA generated in order to offset pH changes. For this reason, offsetting high VFA production during AD requires that total alkalinity (TA) be maintained slightly above 1.5 g $CaCO_3$/L. Moreover, current process indicators of early warning signs of AD include the VFA/TA ratio and the rate of constant production of biogas [15]. Therefore, VFA/TA preference during AD to pH is borne out of the fact that AD rarely returns to normalcy in the event of a drastic drop in pH, thus rendering pH less significant [16]. In this case, alkalinity must be monitored alongside VFA to ascertain proper overview of anaerobic digester stability [17]. Against this background a VFA/TA ratio of 0.23–0.3 constitutes a stable range for AD, and a quotient <0.23 is an indicator of an under-fed digester that requires more feeding whereas a quotient >0.3 is an indicator of indigestion and poor stability [18]. Similarly, VFA/TA ≤ 0.4 is a safe range for AD with less acidification risk whereas VFA/TA ratio ≥ 1.0 produces carbon dioxide and hydrogen as the main decomposition products [19].

Therefore, the objective of this study is to investigate the effect of various factors such as VFA, pH, and the ratio VFA/alkalinity on the production of methane from vinasse under mesophilic anaerobic conditions. Thereby the digester performance and stability are optimized using response surface methodology (RSM) to achieve the maximum methane yield and by reducing as little as possible inhibition. A Box–Behnken design for three variables, each one at three levels, is used to modeling the AD process. The RSM is applied to identify the ideal levels of parameters that result in the best upgrading methane process.

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 L of alcohol with yields up to 250 L of alcohol/ton of molasses. In parallel, Sotrameg Company (Located in SOUK EL HAD, Commune Benmansour, Allal Tazi, Morocco) 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

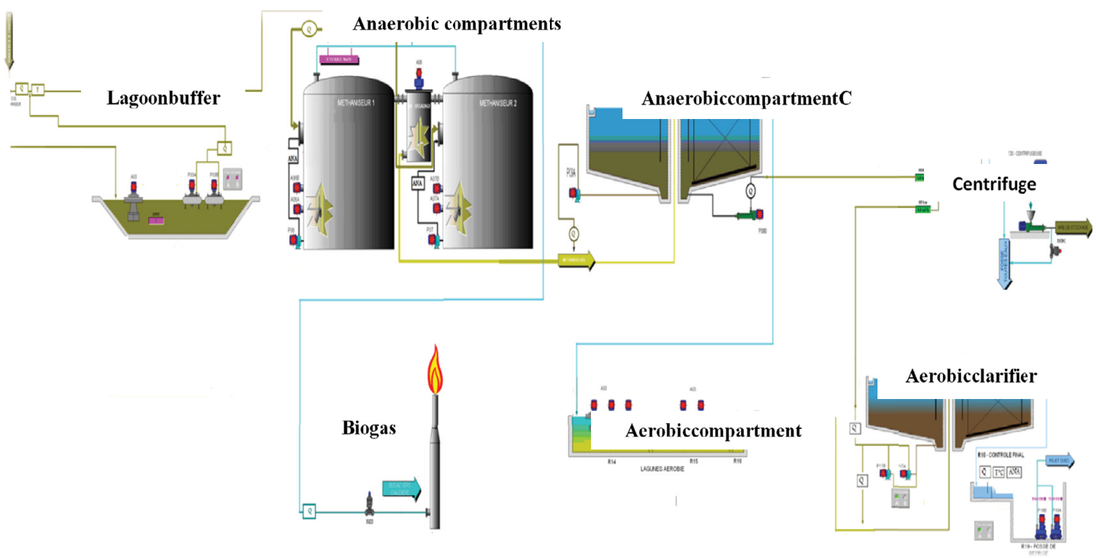

Fig. 1. Schematic of the full-scale plant of vinasse treatment of Sotrameg Company.

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 COD/m^3 d and 6.5 m 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 online 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 chemical oxygen demand (COD), total phosphorus (TP) and total nitrogen (TN) and relatively high level of total suspended solids (TSS). The characterization of the raw vinasse is summarized in Table 1.

2.3. Chemicals analysis

Routine analyses including total 5 d biochemical oxygen demand (BOD_5) and COD, alkalinity, nitrogen, pH, TSS, TP and TN are performed using procedures outlined in standard methods [21,22]. Alkalinity and VFA are then calculated using Eqs. (1)–(3).

Table 1
Characteristics of the vinasse

Parameters	Vinasse	Moroccan Standard Discharge Values [20]
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 (mg/L)	35,000–40,000	100–200
TP (mg/L)	270	10
TN (mg/L)	31–1,250	30

BOD_5: 5 d biochemical oxygen demand

$$\text{Alkanity}(\text{mg/L}) = \frac{\text{Normality of } H_2SO_4 \times \text{mL } H_2SO_4 \times 50.000}{\text{mL of sample used}} \quad (1)$$

$$\text{Volatile fatty acids}(\text{mg/L}) = \frac{\text{Normality of NaOH} \times \text{mL NaOH} \times 50.000}{\text{mL of sample used}} \quad (2)$$

Volumetric methane production rate (L CH_4/(L d)):

$$rCH_4 = \frac{Q_{CH_4}}{V} COD_{int} \quad (3)$$

where Q: effluent flow rate (m^3/h); V: effective volume of reactor bed (m^3); COD_{int} concentrations (mg/L) in the effluent stream; Q_{CH_4}: volume of biogas produced per day (m^3/d).

3. Results and discussion

3.1. Process stability and efficiency

Alkalinity, VFA, and final pH are the major indicators parameters of stability of AD. All these parameters should be stayed in appropriate range, otherwise the AD efficiency will be influenced and disrupted. In Sotrameg Company and during almost 1 y of work, process stability experienced a disruption whenever either as a result of increasing VFA or due to fluctuations in the OLR. Additionally, fluctuation of pH induces unsteady state of the process, especially when they are concomitant with organic overloading. In these cases, the immediate response of the system results in a decline up to 40%–50% in methane content. This decline in the produced biogas is attributed to the decline and disruption in methanogenic activity. The yearly evolution of VFA/alkalinity ratio and alkalinity are presented in Fig. 2.

As shown in Fig. 2, initially, the alkalinity, decreases slightly, then starts to increase again and stabilizes. However, alkalinity values in reactor are ranged between 180 and 298 mg/L corresponding to a low value of VFA/alkalinity ratio (<0.4). Therefore, during the last month higher value (0.7) of this ratio is detected, indicating an unstable digester. This behaviour is a consequence of high VFA content in the reactor, and the methane content in the biogas is suddenly dropping (Fig. 2). Conversely, alkalinity suddenly increases during the same period. Fig. 3 presents the evolution of pH and VFA/alkalinity ratio during AD process.

As shown in Fig. 3, at the beginning, the pH drops rapidly as the easily digestible fraction of organic matter is hydrolyzed and converted to fatty acids. After the initial drop, pH starts to rise gradually as the fatty acids are transferred to the methane phase reactors, consumed by methanogens, and joined the gas phase. Generally, the pH value is approximately stable between 7.6 and 8.0 throughout the study period without any adjustment, even that the VFA/alkalinity ratio in the reactor is higher than the

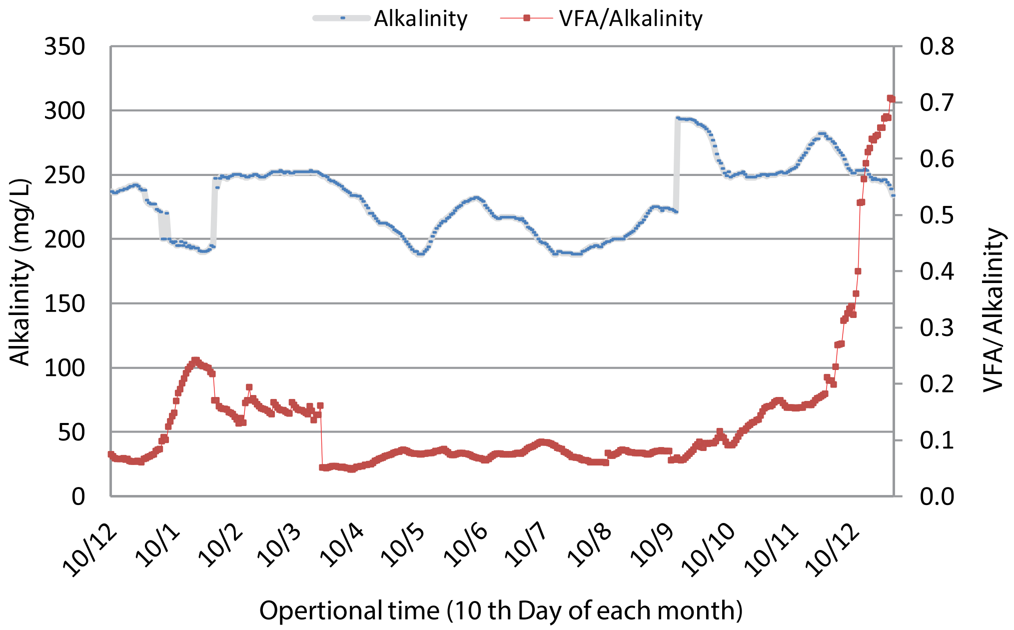

Fig. 2. Yearly evolution of VFA/alkalinity ratio and alkalinity during AD of vinasse.

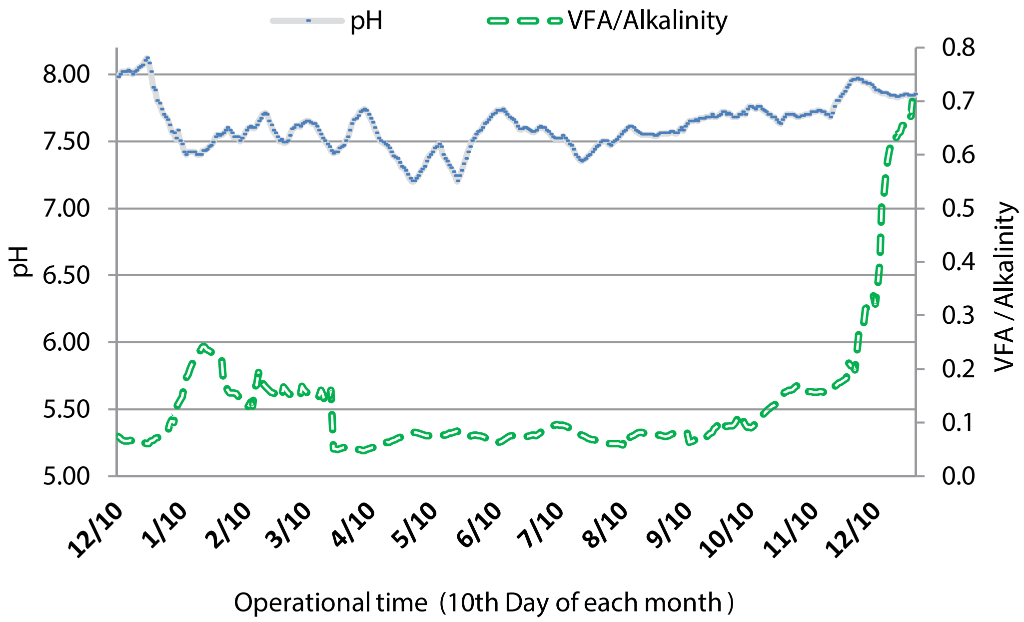

Fig. 3. Evolution of VFA/alkalinity ratio and pH during AD of vinasse.

recommended values >0.4 in certain occasion. The reason for this is that the alkalinity of the system is also the highest which ensures, through its buffer capacity of the system pH drop resulting from VFA accumulation on the one hand. On the other hand, the proper final pH values for all the groups in this study might be stable because the AD system could adjust pH gradually to some extent and an appropriate initial pH is easier for microorganism to adjust it into proper range. When digester is unstable or in sour condition, the activity of methanogens is reduced, causing accumulation of VFA. Fig. 4 indicates the variation of individual VFA and methane yield during the start-up period.

The methane production during AD of vinasse is extremely important to make feasible the technology application. With regards to the methane content in biogas during mesophilic conditions of AD of vinasse, the analysis of the Fig. 4 shows that the percent of methane yield in biogas vary between 49% and 71%. A higher range of methane yield is obtained reaching up to 4.0 L CH_4/L d with an OLR of 4.8 kg/m^3 d and a VFA less than 30 meq/L. Whereas, for a value of VFA higher than 20 meq/L d, the methane yield decreases progressively, drops to 1.5 L CH_4/L for VFA higher than 50 meg/L d, along with the risk of acidification or reactor failure. According to Paritosh et al. [23], in acidic conditions high VFA occurs, carbonic acid predominates, and more CO_2 can be released in the biogas, which could contribute to the lower methane concentrations in reactor. Consequently, the profile of the VFA/alkalinity ratio is indeed very similar to that of total VFA (Figs. 2 and 3). In previous studies on anaerobic treatment of sugarcane vinasse with UASB reactors operating under mesophilic conditions, de Barros et al. [24] reported volumetric methane production up to 0.9 L CH_4/L d with an OLR of 6.0 g COD/L d. In another work, Janke et al. [25] obtained values up to 1.4 L CH_4/L d with OLR of 5.9 g COD/L d.

Table 2 summarizes mean values of operating and efficiency parameters during stable periods under mesophilic AD of vinasse. It should be noted that the period of experiment is subdivided into four phases.

The industrial AD performance is evaluated over the long-term on the basis of results obtained over a year. The vinasse flow applied in this study is increased in a stepwise fashion in order to minimize the transient impact on the reactors. During the first phase, vinasse flow in the reactor equal to 3.73 m³/d, corresponding to 0.1 of VFA/alkalinity ratio indicating that the system is an under-fed digester that requires more feeding. A slightly higher vinasse flow of 5 m³/d is applied in the second phase corresponding to VFA/alkalinity ratio of 0.22. In the third phase, vinasse flow reaches 7.12 m³/d corresponding to VFA/alkalinity ratio of 0.23–0.3, indicating that the system constitutes a stable range for AD process. In this phase, the production of biogas, reaches 11,600 m³/d containing 71% of proper methane, and the degradation of COD is close to 85%. Whereas, in the fourth phase the VFA/alkalinity ratio higher than 0.3 indicates indigestion and poor stability of system. Consequently the biogas flow production is dropped from 11,000 to 5,000 m³/d. This decline in the biogas flow production can be attributed to the inhibition activity of methanogenic bacteria inducted by the increase of VFA production.

3.2. Process optimization

An anaerobic reactor may fail if syntrophic relation between acetogens and methanogens is not stable [27]. For a better syntrophic relation, pH of the anaerobic reactor plays dynamic role as accumulation of VFA may cause dramatic change in pH which may affect methanogenesis. Apart

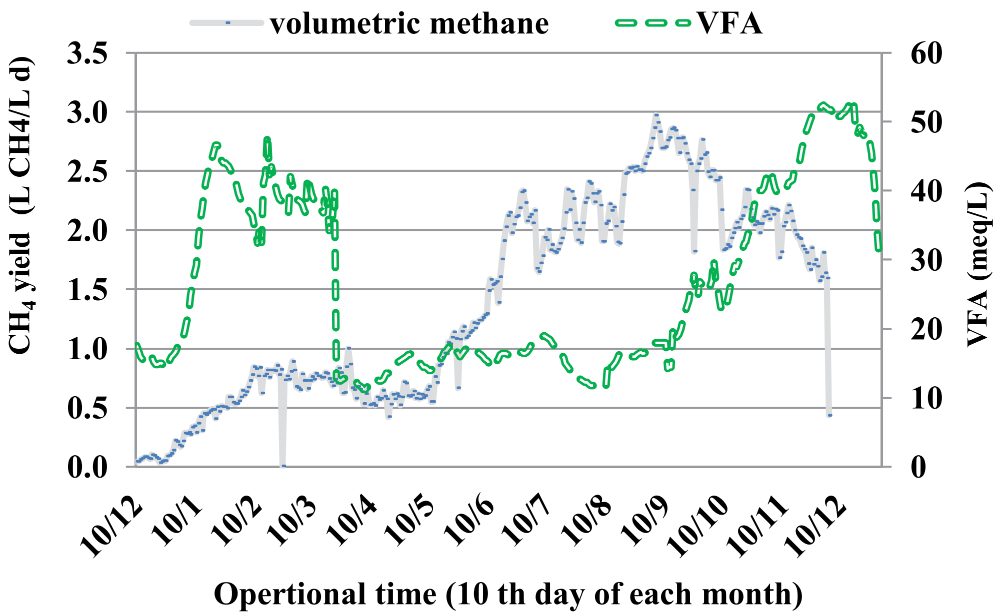

Fig. 4. Evolution of VFA and methane production yield.

Table 2
Operating and efficiency parameters during stable periods under mesophilic AD of vinasse

Reactor conditions			Reactor performances				
Periods	Vinasse flow (m³/d)	OLR (kg/m³ d)	pH	HRT (d)	VFA/alkalinity ratio	% COD	Biogas (m³/d)
I	3.73	0.7	8.0–8.5	40	0.1	54	2,800
II	5.02	1.4	7.4–7.8	20	0.22	65	5,100
III	7.12	4.2	7.5	10	0.3	85	11,600
IV	9.35	4.8	>7.8	15	0.72	55	5,160

from pH and VFA, alkalinity also affects the smooth operation of an anaerobic digester. Koch et al. [27], reported that for inspecting the stability of an anaerobic digester, VFA/alkalinity ratio could be used. In this part, optimization of the interactive effect of three factors on daily methane production during a mesophilic AD of vinasse is investigated through response surface methodology (RSM). Fig. 5 shows the effect of pH, VFA, VFA/alkalinity ratio on average of daily methane production.

Fig. 5 shows that the average daily methane production is significantly impacted by operating conditions: VFA/alkalinity ratio, VFA and pH. However, the average value of daily methane production response decreases strongly with the increase VFA going from a level below –1 to a level above +1 and when the VFA/alkalinity ratio is ranged in 0.1–0.4. Whereas, when the ratio is higher than 0.4, the methane production drops drastically. In terms of pH, the trend is practically stable when pH varies between –1 and 1. Based on the analysis of these results, the average maximum value of the produced methane is obtained for a pH of 7.2 and VFA of 10 as well as a VFA/alkalinity ratio less than 0.4. Therefore, VFA close to 10 meq/L seems to be preferable to avoid accumulation of VFA and subsequently inhibition of the management environment. By applying multiple regression analysis on the experimental data, a second-order polynomial equation, for daily production of methane fitted in terms of coded factors obtained as follows:

$$\text{Methane}\left(\text{m}^3/\text{d}\right) = 166{,}810 - 119A - 43{,}240B - 5{,}334C + 2.57AA + 2{,}844BB - 11{,}983CC + 2.3AB - 172AC + 2{,}724BC \quad (4)$$

The significant terms of the model for 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. Fig. 6 presents the surface plot showing effect of pH, VFA, and VFA/alkalinity ratio on methane yield.

It could be deducted from Fig. 6a that the interaction is significant between VFA/alkalinity ratio and pH for methane production at different VFA/alkalinity ratio level. The same variable trend for methane production at different initial VFA and VFA/alkalinity ratio level is observed in Fig. 6b. This behaviour means that interaction is not significant between initial pH and VFA. In general, Fig. 6 indicates that the production of methane is favored for values of VFA/alkalinity ratio less than 0.4, with a maximum value of CH_4 obtained for VFA/alkalinity ratio of 0.3, VFA level within the experimental range (VFA = 10 meq/L) and pH range in 7.2–8.0. The pH behaviour for all the groups in this study might be stable because the AD system could adjust pH gradually to some extent and an appropriate initial pH is easier for microorganism to adjust into proper range.

4. Conclusion

The AD of Sotrameg vinasse was conducted under mesophilic conditions. The study sought to objectively assess the influence of VFA/alkalinity ratio on digester stability and biogas production, as a means for managing biowaste. A process disturbance caused by over loading with industrial waste was reflected by a significant increase in all VFA concentrations. In the light of this, biogas production in relation to influencing indicators such as VFA/alkalinity and pH were strictly monitored.

The study can conclude that the stability of digester was assured by VFA/alkalinity ratio of 0.23–0.3, and for a ratio <0.1 is an indication of an under-fed digester that requires more feeding whereas a ratio >0.3 is an indication of indigestion and poor stability. In case at which the digester maintained stability, the production of biogas, reaches 11,600 m^3/d containing 71% of proper methane, the degradation of COD is close to 85%. Although, the pH value is approximately stable between 7.6 and 8.0 throughout the study period without any adjustment, even that the VFA/alkalinity ratio in the reactor is higher than the recommended values >0.4 in certain occasion.

Based on the analysis of RSM, the average maximum value of the produced methane is obtained for a pH of 7.2 and VFA of 10 as well as a VFA/alkalinity ratio less than 0.4. Therefore, VFA close to 10 meq/L seems to be preferable to

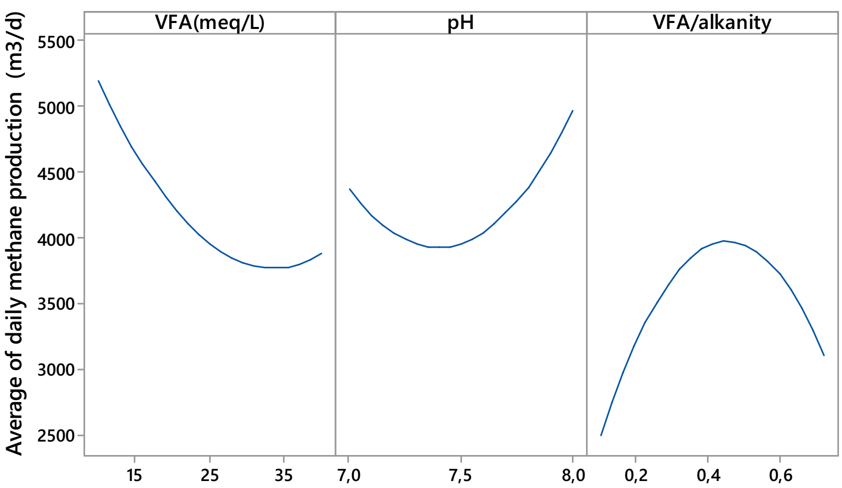

Fig. 5. Effect of pH, VFA, VFA/alkalinity ratio on average of daily methane production.

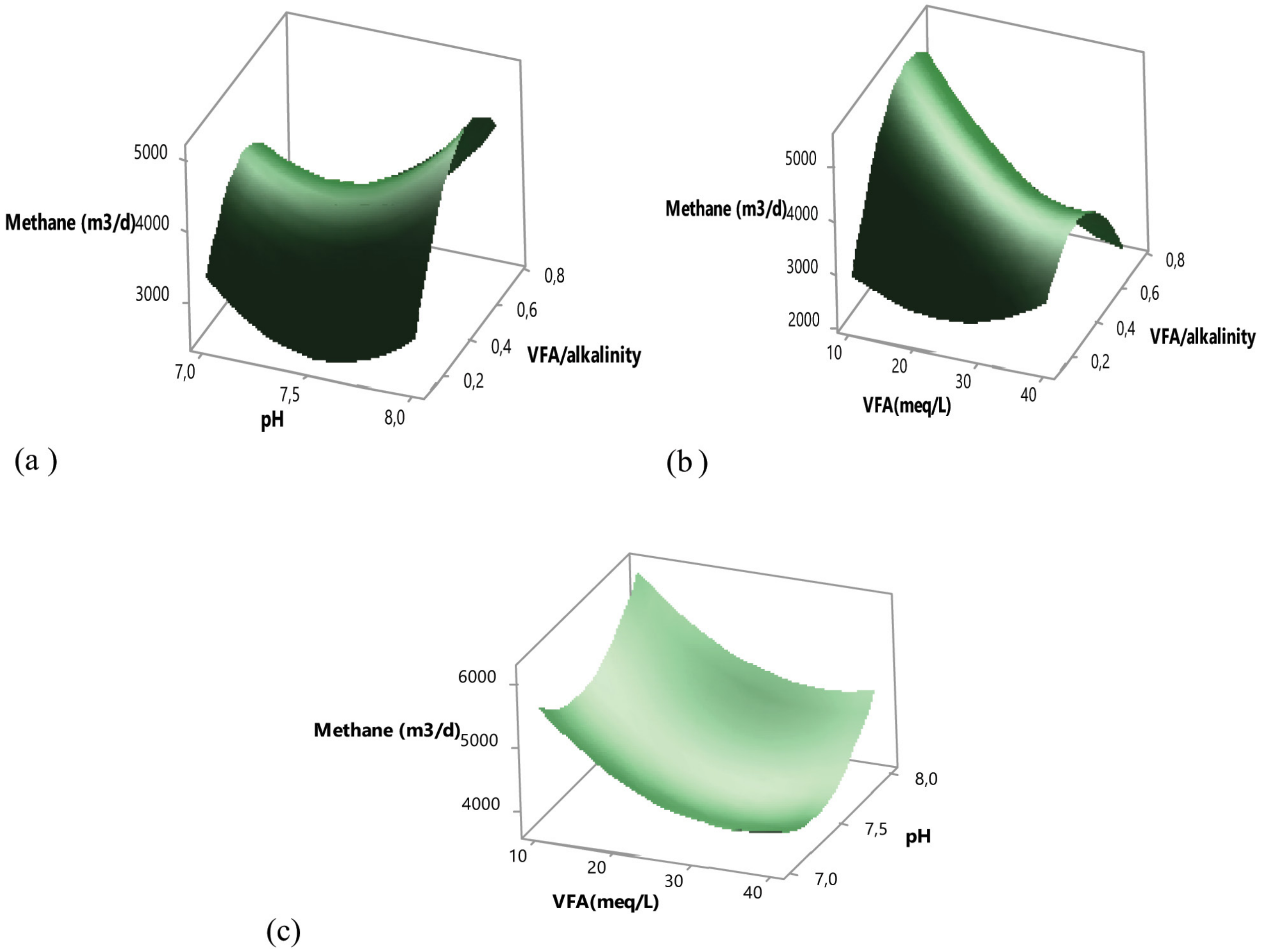

Fig. 6. Surface plot showing effect of pH (a), VFA (b) and VFA/alkalinity ratio (c) on methane yield.

avoid accumulation of VFA and subsequently inhibition of the management environment.

Thus, these results indicate a good performance of the mesophilic AD thanks to the stability of physico-chemical parameters with biogas production containing 71% of proper methane. The models developed using RSM methodology can provide guidance for future feed stock evaluation and process optimization in AD.

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