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Industrial-scale anaerobic digestion of vinasse in Morocco: performances and statistical models

Maryem Elazhar[a], Abdelmajid Bouchabchoub[b], Fatima Elazhar[a,c], Azzedine Elmidaoui[a], Mohamed Taky[a,d,*]

[a]*Laboratory of Advanced Materials and Process Engineering, Faculty of Sciences, Ibn Tofail University, BP 1246, Kenitra – Morocco, emails: mohamed.taky@uit.ac.ma (M. Taky), Maryem.elazhar@uit.ac.ma (M. Elazhar), fatima.elazhar@uit.ac.ma (F. Elazhar), elmidaoui@uit.ac.ma (A. Elmidaoui)*
[b]*Gharb Molasses Processing Company (Sotrameg), Kenitra – Morocco, email: dg@sotrameg.ma*
[c]*National Higher School of Chemistry (NHSC), IbnTofail University, Kenitra – Morocco*
[d]*International Water Research Institute, Mohammed VI Polytechnic University, Lot 660, Hay Moulay Rachid, Ben Guerir, 43150 – Morocco*

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ABSTRACT

The vinasse causes a serious environmental problem when it is not previously treated before being discharged directly into the environment. Organic loading rate (OLR) is an important operating variable for the anaerobic digestion (AD) process related to system stability, waste treatment capacity and biogas production. For this reason, the main aim of this paper is to evaluate the performances in the mesophilic methanogenic AD of a sustainable alternative for the treatment of vinasse over nearly 1 y on an industrial-scale (Sotrameg Company, Souk EL HAD, Municipality Benmansour, Province of Kenitra, Morocco). The evolution of OLR on the stability of the AD of vinasse is evaluated through an experimental study and statistical methods using (ANOVA, Modified Gompertz, Richards, and Logistic). The energy potential of biogas generated from the anaerobic process is also evaluated. Experimental results show that the increase in OLR favors the production of biogas. Thus, the chemical oxygen demand (COD) removal efficiency is 85% at an OLR of 4.2 kg COD m^3/d and hydraulic retention time of 12 d achieving a methane yield of 70%. The daily energy generation achieved is 6.95 kWh/m^3 and approximately 50% of this energy is reused to satisfy the needs of Sotrameg facilities. The proposed statistical models are used to describe the dynamic behavior of methane production and its dependence on OLR. A good fit between the variables estimated by the statistical model and experimental data is obtained, reaching determination coefficients (R^2) greater than 0.9.

Keywords: Vinasse; Anaerobic digestion; Methane yield; energy; Organic loading rate; Kinetic; statistical models

1. Introduction

Anaerobic digestion (AD) is a process in which microorganisms break down the biodegradable material waste in absence of oxygen. This process is widely used in the treatment of wastewater and organic waste [1–3]. AD has plenty of advantages that can be summarized in two points: on the one hand, AD allows the depollution of organic effluents by considerably reducing their chemical oxygen demand (COD). On the other hand, it allows the production of biogas, which contains more than 60% of methane [4,5]. The latter has very high energy potential and is considered renewable energy [6].

In Morocco, AD can be a strategic choice for the promotion of green energy, thanks to the significant potential

* Corresponding author.

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of biomass available in the Kingdom and due to its strong commitment to environmental protection and promotion of sustainable development (Law n° 13-09). Thus, the production of biogas by AD of biomass is one of the promising resources of renewable energy not yet supported in Morocco [7]. The potential for development of this technology is promising in agriculture, industry or agri-food sectors.

Sotrameg Company, Souk EL HAD, Municipality Benmansour, Province of Kenitra, Morocco, 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 company has a long historical tradition and today, Sotrameg is one of the most important agro-industries in the country, which contributes largely to providing the local market. The annual production reaches more than 50 million L of alcohol with yields up to 250 L of alcohol/ton of molasses. Nowadays, Sotrameg Company, Souk EL HAD, Municipality Benmansour, Province of Kenitra, Morocco is facing a real problem, which is the generation of highly polluting effluents, also called vinasse (the main liquid stream from the first-generation ethanol production process). This major environmental problem is related to the sugarcane processing plants, which generally generate up to 10–15 m^3/h of vinasse for each liter of produced ethanol [8–10]. So presently, in Morocco 400–750 million L of vinasse per year are generated. The nature and the quantities of this waste constitute a real danger and threat to the environment and public health. Moreover, with the paradigm shift brought recently by the water-energy nexus, this effluent very rich in organic matter is now viewed not as a waste but rather as a resource with high potential.

There are several treatment methods of vinasse, physic-chemical treatment such as (sedimentation and flotation, coagulation and precipitation, filtration, reverse osmosis, adsorption, wet oxidation, ozonation and other advanced oxidation processes) and biological treatment, such as AD, aerobic treatment, activated sludge and fungal treatment [11,12]. It is generally accepted that anaerobic processes offer several advantages over aerobic ones for the treatment of vinasse because the anaerobic processes are considered energy, environmental and economically viable. Anaerobic and aerobic digestions of vinasse have been the subject of a number of studies using laboratory or pilot scale digesters [13].

Among various environmental conditions, the organic loading rate (OLR) is an important parameter that has an essential effect on AD performance. Indeed, the suitable increase in OLR can favor cell activity, which allows amelioration of the methane (CH_4) production and degradation of the substrate, that is, increasing OLR can improve the overall performance of the AD process. In this way, Wijekoon et al. [14] studied the effect of OLR in the production of volatile fatty acids (VFA) from wastewater, identifying that the moderate increase in OLR improves the efficiency of organic load degradation.

Blasius et al. [15] focused on the determination of the ideal conditions for treating household waste by AD, to evaluate the potential of methane generation under mesophilic and thermophilic conditions and to determine the maximum OLR not to be exceeded in the digesters. Barros et al. [16] evaluated startup and stabilization of the AD of vinasse to methane with a gradual increase of OLR in UASB reactors. The study showed that increasing OLR value improved the efficiency of total COD conversion into methane. In another work, Arreola-Vargas et al. [17] studied the effect of OLR on AD of tequila vinasse, concluding that methane production can be enhanced by increasing OLR.

Recently, Moguel-Castañeda et al. [18] investigated the effect of OLR on methane production on AD of raw whey experimentally and theoretically. They evaluated through an experimental study and numerical simulations based on an applied mathematical model (Levenberg–Marquardt algorithm), the operating conditions that promote methane production.

Thus, this work aims at evaluating over 1 y the AD performance of Sotrameg Company, Souk EL HAD, Municipality Benmansour, Province of Kenitra, Morocco in industrial conditions. The evolution of OLR on the system stability is explored in terms of pH, COD removal, biogas production, methane yield and hydraulic retention time (HRT). Furthermore, based on the laboratory data, kinetic results of the methane production from the AD of vinasse using statistical models (ANOVA, Modified Gompertz, Richards, and Logistic kinetic) are compared to determine the best-fit method. Lastly, the technical study of the energy potential of biogas generated from the anaerobic process is evaluated.

2. Methods and materials

2.1. Description of Sotrameg Company, Souk EL HAD, Municipality Benmansour, Province of Kenitra, Morocco

Sotrameg Company, Souk EL HAD, Municipality Benmansour, Province of Kenitra, Morocco 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 entire processing plant of the vinasse adopted by the Company. Table 1 gives the criteria of dimensioning for the anaerobic bioreactor process.

2.2. Vinasse characterization

Vinasse is characterized as an effluent with a high pollution potential, containing high levels of organic compounds and nutrients (mainly total phosphorus (TP) and total nitrogen (TN)). The characterization of the raw vinasse is summarized in Table 2.

2.3. Chemicals analysis

Routine analyses including total biochemical oxygen demand (BOD_5) and COD, alkalinity, nitrogen, pH, total suspended solids (TSS), TP and TN are performed using procedures outlined in standard methods [20]. The AD performances of this study are followed by HRT, OLR, COD removal efficiency and volumetric methane production rate. Most of the parameters are monitored daily during the startup phase and every other day during the normal operation.

- Hydraulic retention time (d):

$$\mathrm{HRT} = \frac{V}{Q} \tag{1}$$

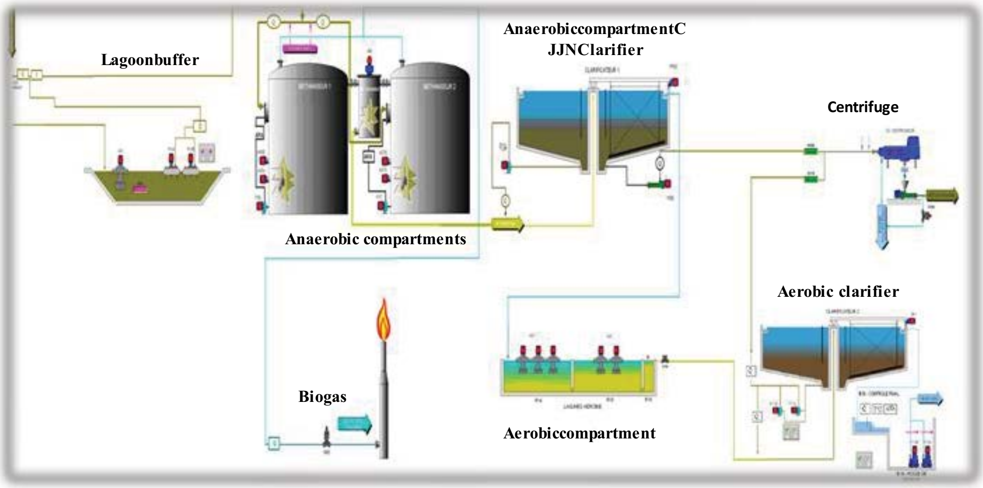


Fig. 1. Schematic of the full-scale plant of vinasse treatment of Sotrameg Company, Souk EL HAD, Municipality Benmansour, Province of Kenitra, Morocco.

Table 1
Criteria of dimensioning for the anaerobic bioreactor process

Average volume load	3 kg DCO/m^{3}d
Liquid volume	8,539 m^3
Hydraulic retention time	20 d
Diameter	30 m
Total height	6.5 m
Digester volume	4,200 m^3
Organic load	23 tons COD/d

- Organic loading rate (g/(Ld)):

$$OLR = \frac{Q}{V} COD_{int} \quad (2)$$

- COD removal efficiency (%):

$$\%COD = \frac{COD_{INT} - COD_{OUT}}{COD_{INT}} \times 100 \quad (3)$$

- Volumetric methane production rate (L_{CH4}/(Ld)):

$$rCH_4 = \frac{Q_{CH_4}}{V} COD_{int} \quad (4)$$

where Q: effluent flow rate (m^3/h); V: effective volume of reactor bed (m^3); COD_{int} and COD_{out}: concentrations (mg/L) in the influent and effluent stream respectively; Q_{CH4}: volume of biogas produced per day (m^3/d).

3. Results and discussion

3.1. Evolution of OLR, COD abatement efficiency and HRT during the company experiments

The industrial AD performance is evaluated over the long-term on the basis of results obtained over a year. This study compares the performance of an anaerobic digester at fluctuating and constant daily OLR. Fig. 2 shows the change in OLR imputed to HRT and COD removal efficiency during AD of vinasse. Fig. 2a shows that at the startup of the AD process, the OLR value is close to 0.2 kg m^3/d while the HRT appears to take too long, more than 40 d. It takes the time required for the reactor to completely stabilize the biomass. However, this long period before stabilizing is likely due to the shock created by the nutriments, which consequently change of state of the OLR. After a short adaptation period of the ecosystem, the OLR stepwise increases from 0.7 to 4.2 kg m^3/d and the HRT is shortened progressively up to 10 d. Indeed, this observation indicates a good activity of biomass formation and organic matter degradation. The slight drop in OLR (Fig. 2a) has a value of 2.3 kg m^3/d causes a slight increase of the HRT up to 13 d, this is mainly due to a technical electricity problem.

Fig. 2b shows that the efficiency of COD abatement is between 56%–60% at OLR 0.2 kg m^3/d, that is, at the startup

Table 2
Characteristics of the vinasse

Parameters	Vinasse	Moroccan Standard discharge values[a]
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[b] (mg/L)	35,000–40,000	100–200
TP (mg/L)	270	10
TN (mg/L)	31–1,250	30

[a]Moroccan Pollution Standards: specific limits for industrial discharge [19].
[b]BOD_5: 5′d biochemical oxygen demand.

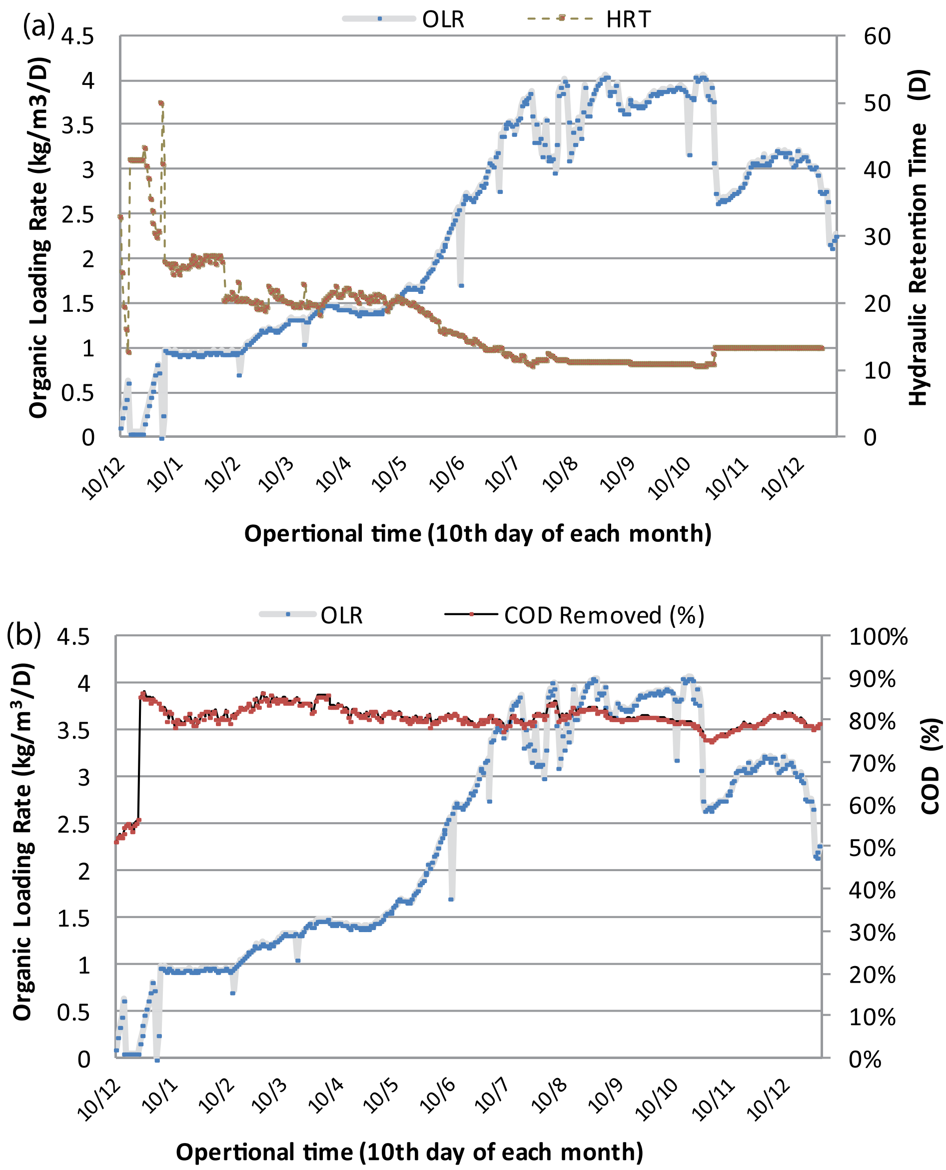


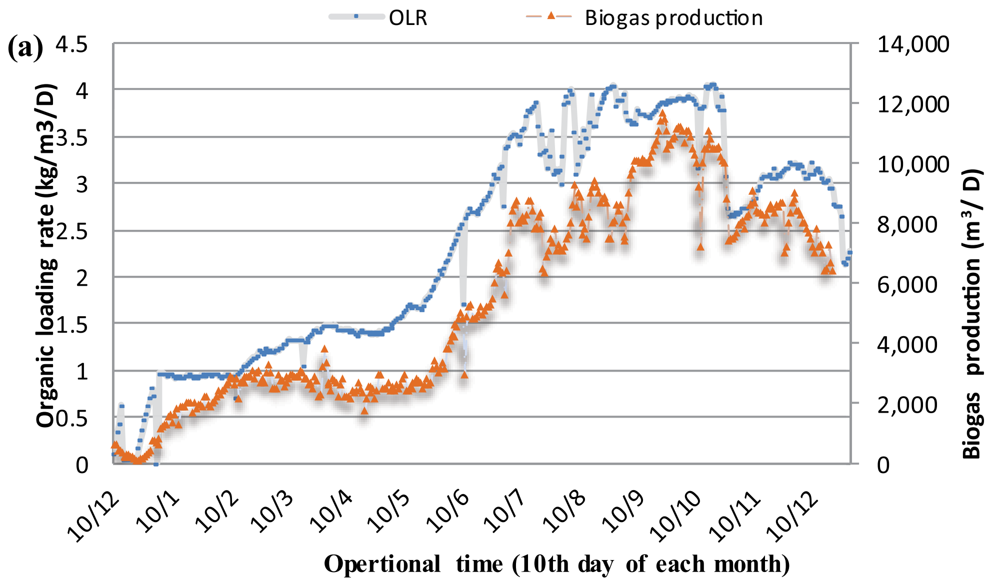
Fig. 2. (a) Evolution of OLR and HRT during the company experiments and (b) evolution of OLR and COD abatement during the company experiments.

of the AD process, after stabilization, the increase of OLR leads to a clear improvement of COD abatement efficiency to achieve values ranging from 85% to 90%. These values are higher by more than 50% compared with those observed by Santos et al. [21]. In spite of the OLR disturbances, the COD efficiency is maintained constant throughout the experiment. It is remarkable that the COD abatement efficiency is not affected even though for higher OLR.

3.2. Evolution of OLR, daily volume of produced biogas and methane yield during the company experiments

The production and composition of the biogas are monitored throughout the AD reactor with the objective to assess the effect of OLR on the production of methane. Fig. 3a shows the total amount of biogas produced during the reactor-operating period. After acclimatization and subsequent batch studies, the initial OLR of 0.2 kg m^3/d is gradually raised up to 4.2 kg m^3/d, leading to a gradual biogas production. This production peaks at a maximum value of 11,660 m^3/d for OLR equal to 4.2 kg m^3/d of COD. Fig. 3a shows clearly that the behavior of biogas evolution is identical to that of the OLR. Generally, papers that reported methane production show some tendency towards an increased influent concentration and a concomitant rise in stability and methane production if not inhibited by substrate concentration [14].

However, the biogas composition of methane yield is maintained constant during the three first months of operation of the digester at 60% ± 5% (Fig. 3b). As shown in Fig. 3b, the percentage of methane yield peaks in the range of 75%–88%. Subsequently, the OLR is then increasing to



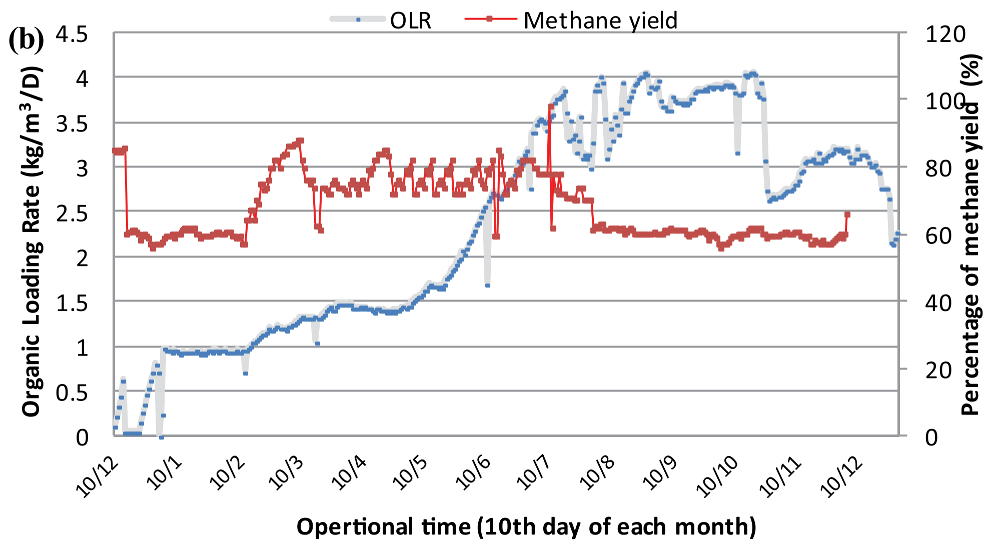


Fig. 3. Evolution of (a) OLR and biogas production and (b) OLR and methane yield during the company experiments.

4.2 kg m^3/d and the percentage of methane yield remains at 60%. It is clear that a drop in methane production occurs with the increase in OLR at some points [22]. It seems that this is attributed to the shock received by the system due to changes in OLR that affect the methanogenic metabolic activities in the reactor.

3.3. Evolution of OLR and effluent pH during the company experiments

The pH is the most sensitive parameter in the AD process. The pH of liquid effluent in the digester indicates the stability of the system and its variation depends on the buffering capacity of the system. Fig. 4 shows that the pH at various OLR is comparatively stable in the range between 7.6–8.01 throughout the study period. Hence, the effluent in this reactor exhibits high buffering capacity and, consequently, does not require pH correction. In fact, several studies reported that most of the anaerobic bacteria, especially methanogens, enhance biogas production at a pH range of 7.8–8.2 [23,24]. Thus, the slight decrease of the pH is attributed to the accumulation of the fatty acids in the digester.

3.4. Statistical models

3.4.1. Correlation between OLR and cumulative biogas production

Fig. 5 confirms a linear trend and a strong relationship between cumulative biogas production and OLR. The cumulative biogas production tends to increase when vinasse residue is treated at high OLR, this result agrees with previous studies [25]. The obtained value of the coefficient (R^2) is estimated at 0.99, which indicates a very good fit.

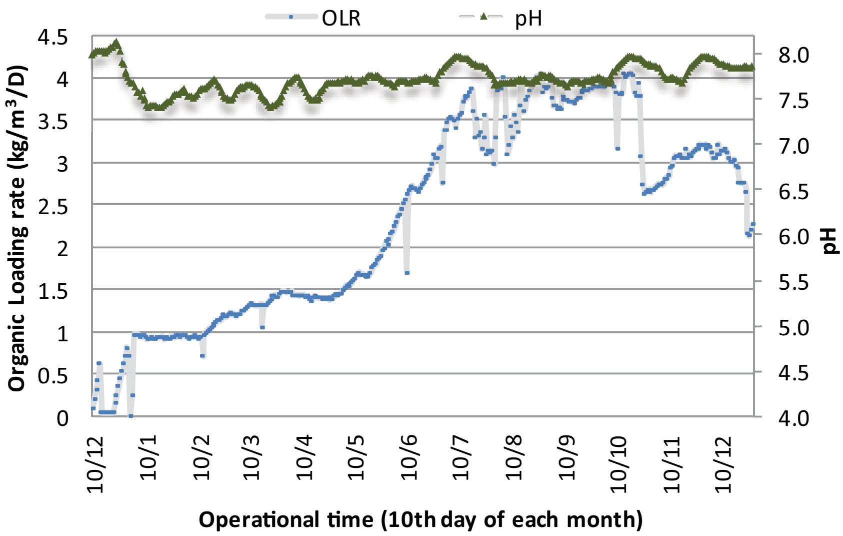


Fig. 4. Evolution of OLR and pH during the company experiments.

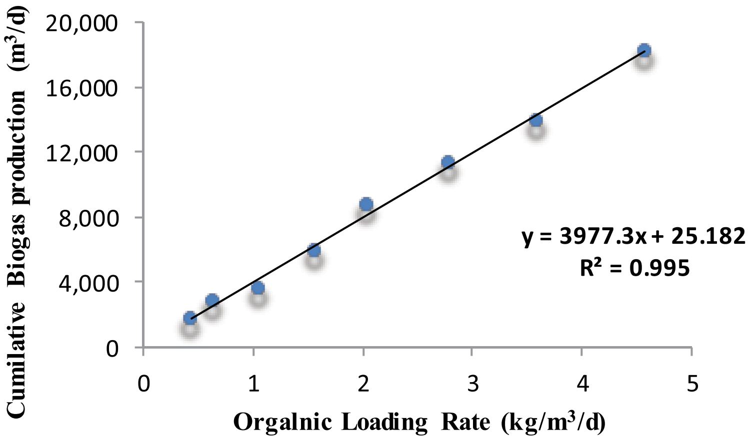


Fig. 5. Relationship between OLR and cumulative methane production.

3.4.2. ANOVA test

The statistic regression on the performance data is performed to predict the cause and effect relationship of biogas production and OLR. Fig. 6 shows the summary of a multiple regression plot of biogas production in the anaerobic reactor during the period of the operation. The obtained value of coefficient (R^2) 0.86 indicates a very good fit. 86% of the variation of biogas production is explained by OLR variation in an anaerobic digester. The value of significance F is obtained as 0.0035, which is less than 0.05. The p-value of the hypothesis test, that is, $H0$: $b1 = 0$ is 0.0031, which is less than 0.05 for intercept, indicates an existing good relationship between biogas volume and OLR. The regression summary output with ANOVA results for biogas production is presented in Table 3.

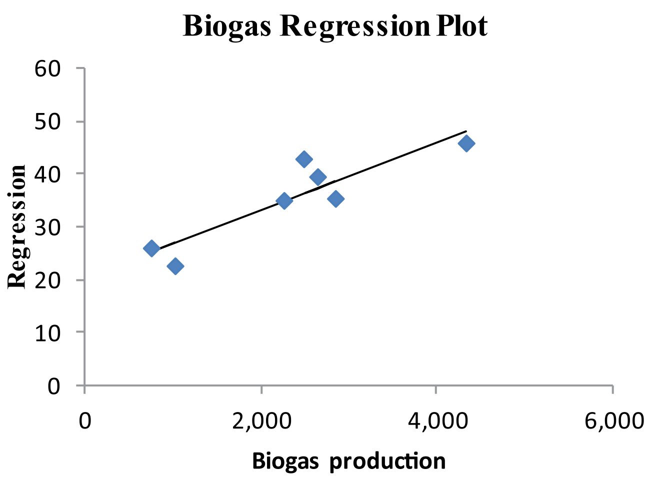


Fig. 6. Summary of multiple regression plot of biogas production.

3.4.3. Kinetic performances

Based on industrial data above, the accuracy of biogas yield prediction using three statistical models is analyzed

[26]: Modified Gompertz model [Eq. (1)], Logistic model [Eq. (2)] and Richards model. The aim of the kinetic study is to adjust the experimental results in terms of the volume of produced methane during the experiment for the different added loads. In addition, this information is important to describe the kinetic parameters of methane production and to select the most useful model(s) for the AD of vinasse. Fig. 7 shows that the model fits with the experimental data. The estimation of the kinetic parameters of three statistical models, Gompertz, Logistics and Richard, is summarized in Table 4.

$$y = P\exp\left\{\exp\left[\frac{R_m e}{P}\left(a-t\right)+1\right]\right\} \tag{5}$$

$$y = \frac{P}{1+\exp\left\{\exp\left[\frac{4R_m}{P}\left(a+t\right)+2\right]\right\}} \tag{6}$$

Table 3
Summary of regression

Regression statistics					
Multiple R					0.855
R-square					0.947
Adjusted R-square					0.813
Standard error					1.788
Observations					7
ANOVA results					
	df	ss	Ms	F	Significance F
Regression	1	86.660	86.660	27.090	0.0035
Residual	5	15.995	3.199		
Total	6	102.654			

$$y = P\left\{1+n\exp\left(1+n\right)\exp\left[\frac{R_m}{P}\left(1+n\right)\left(1+\frac{1}{n}\right)\left(a-t\right)\right]\right\}^{1/n} \tag{7}$$

where Y is the biogas accumulation (m^3/g VS) at time t (d), "t" is the time (d) over the digestion period. P is the biogas production potential (m^3/g VS), R_m is the maximal biogas production rate (m^3/g VS/d) while "a" is the lag phase (d) or minimum time to produce biogas and "e" is a mathematical constant (2.718282).

Fig. 7 indicates that the volume of methane production values predicted is almost equal for all three models when all values are in the range of 11,660 m^3/d. Furthermore, the difference between the predicted and measured biogas yield is higher with the Logistic kinetic model 3.55% than with the Richards model 0.65% and the Modified Gompertz model 0.56%. Consequently, the small difference between the theoretical values and the experimental values (less than 10%) indicates that all the proposed models accurately predict the behavior of the AD reactors. The coefficient correlation (R^2) indicates how best the predictive model curve fits the experimental observations. Although the three models have desirably high values of R^2 which are higher than 0.99. Therefore, the lag phase required to producing

Table 4
Kinetic parameters by applying the three statistical models

	Logistic	Gompertz	Richards
Measured CH_4 (m^3/d)	11,660	11,660	11,660
Predicted CH_4 (m^3/d)	11,245.23	11,594.23	11,548.5
Difference (%)	3.55	0.56	0.65
Rm	9,534.95	9,320.064	11,548.5
P	2.64×10^6	2.89×10^6	2.31×10^6
a (d)	156.5	146.7	164
n	–	–	1.8
R^2	0.92827	0.99854	0.98854

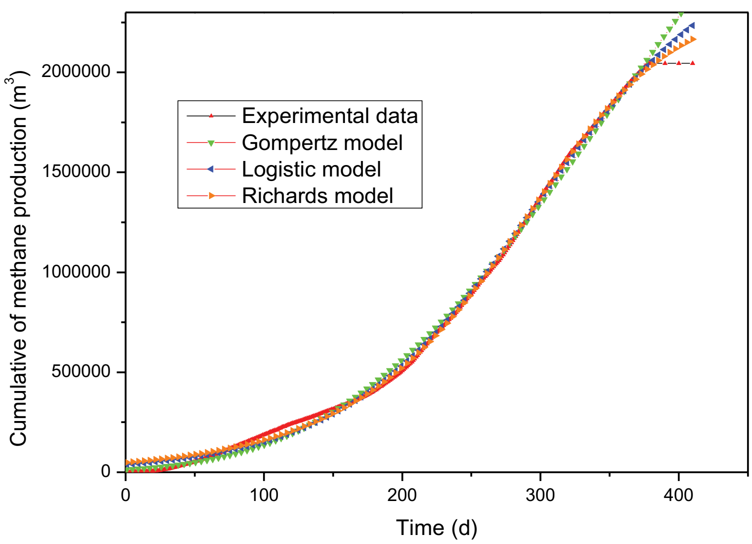


Fig. 7. Simulation by three models and experimental production of cumulative methane for AD of vinasse.

Table 5
Evaluation of energy potential generated by AD of vinasse

Maximum COD abatement (%)	85
Abatement kg COD	9,299
Methane production coefficient (m^3/kg COD)	0.7
Energy generation (kWh/m^3)	7.95

biogas is short with the Modified Gompertz model. From the obtained results by the kinetic study with nonlinear regression, it appears that the Modified Gompertz model has a better fit for vinasse substrate.

3.4.4. Energy potential of AD of vinasse

The result shows that the Sotrameg distillery plant with a capacity of 4,200 m^3 could generate 7.95 kWh/m^3 of daily energy potential, with a capacity biogas production reaches 11,660 m^3/d and the percentage of methane yield is 70%. During this process, approximately 50% of the recovered energy is contributed to satisfy Sotrameg Company, Souk EL HAD, Municipality Benmansour, Province of Kenitra, Morocco needs in the plant. From an overall balance, the adoption of AD for vinasse treatment in biorefineries could potentially lead to profit from energy, environmental and economic perspectives. These performances will improve and optimize the plant in terms of sustainability [27]. The energy generated by the AD process is calculated as [28]:

$$E = \text{LHVCH}_4 \times \text{ECOD} \times \text{CCOD} \times \alpha\text{CH}_4 \quad (8)$$

where E is the AD energy production (kWh/m^3), LHVCH_4 is the low heating value of methane which is 38 MJ/m^3 (10.55 kWh/m^3), ECOD is the COD removal efficiency, CCOD is the total COD fed to the reactor (kg COD) and αCH_4 is the methane production coefficient (m^3/kg COD removed). Table 5 shows the evaluation of the energy potential generated from the AD reactor.

4. Conclusion

This study is dedicated to the AD of vinasse on an industrial-scale of Sotrameg Company. AD of vinasse is an effective and sustainable solution to convert organic matter into biogas rich in methane. The results reveal that OLR affects many aspects concerning the performance of an anaerobic reactor. The reactor appears to support high organic loads. However, the industrial AD process works without inhibition problems. The COD reduction reaches 80%–90% and the biogas production reaches 11,600 m^3/d with an OLR of 4.2 g COD/L. According to the data of the biogas composition, the average percentage value of methane is equal to 70% and the daily energy generated is 7.95 kWh/m^3. A good fit between the variables estimated by the statistical model and experimental data is obtained, reaching determination coefficients (R^2) greater than 0.9 of each applied model. Therefore, the obtained results of the kinetic study with nonlinear regression, reveal that the Modified Gompertz model is found to have a better fit for vinasse substrate. The lag phase required to producing biogas is short than the other models. This study indicates that AD is a sustainable alternative to simultaneously solve the environmental pollution issue engendered by the vinasse and to offer an excellent opportunity to exploit locally and internally the economic benefits of the produced energy.

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