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Performance analysis of operating parameters during mesophilic anaerobic digestion of vinasse using principal component analysis

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ABSTRACT

Principal component analysis (PCA) is the statistical tool used in this paper to investigated the synergistic correlations of systematic parameters and their correlations based on the effects of input parameters such as: chemical oxygen demand (COD), initial pH, volatile fatty acids (VFA), hydraulic retention time (HRT), organic loading rate (OLR) and temperature (T) on the anaerobic digestion (AD) performance of vinasse such as methane yield and removal COD in order to improve their monitoring and control. In addition, the variations of these parameters are followed during more than one year taking into account the OLR and their effect on behavior of methane yield, pH and VFA. The results of the PCA indicate that the percentage of variance is 59.74% and 22.21%, respectively for the axes F_1 and F_2 and the total information is estimated at a percentage of 81.95%. The correlation circle shows that methane yield is positively correlated with pH and T, and inversely correlated with HRT, while VFA is directly correlated as well as alkalinity. Moreover, PCA reveals that the stabilization of pH in the digester has a positive effect on specific biogas yield and methane content. Thus, results indicate a good performance of the mesophilic AD thanks to the stability of physicochemical parameters with biogas production containing 71% of proper methane. The models developed in this study can provide guidance for future feedstock evaluation and process optimization in AD.

Keywords: Anaerobic digestion; Principal component analysis; Methane yield; Biogas; Performances

1. Introduction

In Morocco, agricultural activity related to sugar beets generates, from the harvest to industrial processing about 50 million L of alcohol per year with yields up to 250 L of alcohol/ton of molasses. On the other hand, industrial processing generates up to 10–15 m^3/h of vinasse for each one liter of produced ethanol [1,2]. Presently in Morocco 400–750 million L of vinasse per year are generated, they are a very pollutant residue, which has a chemical oxygen demand (COD) around 100 g/L.

To reduce the environmental impact, anaerobic digestion (AD) of organic substrates for biomethane production has become one of the most mature technologies to

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produce renewable energy from biomass [3]. Elazhar et al. [4] analysed over one year the life cycle assessment of the AD process of the Gharb Molasses Processing Company (Sotrameg) in industrial mesophilic conditions. The study showed an interesting environmental performance and allowed the valorization of biogas production especially the amount of methane (CH_4). This methane energy generation may not be economically viable due to low production rates of methane, but these rates can potentially be increased via process modifications and optimization informed by modeling [5]. It is well known that AD processes are sensitive to environmental conditions and are easily influenced by operational parameters. To improve the AD efficiency, the influence of temperature, pH, hydraulic retention time (HRT), volatile fatty acids (VFA), organic loading rate (OLR) probably present a certain degree of correlation. An efficient tool for AD process monitoring should therefore benefit from integrating the optimization of the information by statistical modeling on how the measured parameters interact when the process is in control [6,7]. It is important to note that models are often limited by the availability of monitored parameters that can be used for modelling purposes. A chemometrics methods have been applied to environmental studies as a statistic tool [8–10] to satisfy this condition monitoring the reactors using statistical process control. In addition, the consideration of pre-processing of input data to reduce input parameters (e.g., principal component analysis (PCA)) has not been well developed. This pre-processing can be useful in limiting model input data that are highly correlated with the resulting model processing time being significantly reduced and, thus, results in a more efficient model. However, the multivariate methods such as PCA can provide further interpretation in environmental studies. Indeed, by plotting the principal component, the interrelationships between different variables may be visualized, and sample patterns, grouping similarities or differences could easily be interpreted [11]. Few environmental studies used the PCA for results interpretation. Mao et al. [12] used the PCA method for studying the effects of total solids (TS) content and initial pH on biogas production, process parameters and kinetic parameters of anaerobic mono-digestion of corn straw and the synergistic correlations of these mentioned parameters and the effects of each other. The results obtained indicate a significant correlation between operational parameters, process parameters and kinetic parameters. In addition, three different synergistic pathways were found: inoculum/substrate (I/S), pH, VFA, hydrolysis rate (k), biogas production potential (P), maximum biogas production rate (R_{max}) and latency phase (λ) were found with values of coefficient $R^2 > 0.95$. In another study, Enitan et al. [13] studied the correlation between operational parameters and methane production using multivariate analysis. The results of PCA showed the effect of pH and other measured physico-chemical parameters on methane production with high positive correlation coefficient ($R^2 > 0.6$). Lemaigre et al. [14] used a static multivariate statistical process control model (MSPC) based on principal component analysis (PCA-MSPC) for an anaerobic reactor maintained in steady-state, joining the biogas composition (CH_4, CO_2, H_2) to the total solids (TS), volatile solids (VS), total inorganic carbon (TIC) and total ammonia nitrogen (TAN) contents of the slurry. The result is very interesting since these parameters are nowadays poorly exploited for AD process monitoring and their online sensing can be easily implemented at the real-scale level, the PCA-MSPC model was successfully transferred to the overfed reactor.

The objective of this research is to study how chemometry contributes to the knowledge of the performance of AD in the treatment of the Sotrameg vinasse. Accordingly, a multivariate static statistical process control based on PCA is developed linking biogas composition (CH_4), and COD removal to OLR, HRT, T, VFA and pH, in order to obtain better information on the feasibility of AD of vinasse on methane production and to highlight the correlation between the individual process variables measured in the vinasse and the methane production. The progress over time of the process performances indicators for reactor during the experiment is also described in terms of stability and methane yield production. Thus, this study has a dual objective with reducing the number of variables analyzed and, consequently, financial cost, which is the main argument for the lack of implementation or improvement of treatment systems.

2. Materials and methods

2.1. 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 1.

2.2. Operating conditions of the digesters

Table 2 summarizes the operating conditions of the digesters. Each value in this table is the average of one month.

2.3. Analytical methods

The main parameters monitored in this study are presented in Table 1. Most of the parameters are monitored daily during the startup phase and every other day during the normal

Table 1
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 (mg/L)[b]	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.
[b]BOD_5: 5'day biochemical oxygen demand.

Table 2
Operating conditions of the digesters

Days	OLR (kg/m³ d)	HRT (d)	*T* (°C)	pH
30	0.61	33	35.1	7.8
60	1.166	25	36.1	7.4
90	1.32	20	37.2	7.5
120	1.63	20	36.7	7.5
150	1.74	20	37.9	7.4
180	2.33	18	38.0	7.4
210	3.58	13	38.7	7.6
240	4.3	11	39.4	7.4
270	4.55	11	39.1	7.5
300	4.71	10	39.5	7.6
330	4.45	11	39.1	7.7
360	3.85	13	36.9	7.7
382	3.5	13	36.3	7.8

operations. Routine analyses including COD, alkalinity, pH, VFA, acidity are evaluated by a titrimetric method [15,16]. The biogas production of reactor is measured by a drum-type wet gas meter (TG-0.5, Ritter, Germany) and is normalized to normal temperature and pressure conditions (0°C; 1025 hPa). For gas composition analysis, the head-space gas is recirculated every hour for 3 min through CH_4 and CO_2 non-dispersive infrared sensors (Dynament, UK).

2.4. Statistical analysis

PCA is a mathematical technique used to reduce the dimensions needed to accurately portray the characteristics of data matrices. By means of this method, the original matrix is represented by a set of new variables, called principal components (PC). Each PC is constructed as a linear combination of variables:

$$P_i = \sum_{k=1}^{n} C_{ij} X_j \tag{1}$$

where P_i is the *i*th principal component and $C_{i,j}$ the coefficient of the variable X_j. The first principal component PC1 is chosen in such a way that the new axis *p*1 has the direction which maximizes the variance of data along that axis. The second and subsequent ones are chosen to be orthogonal to each other and account for the maximum variance in the data not yet accounted for by previous principal components.

PCA has been used to reduce the dimensionality of the dataset, while preserving the valuable information as much as possible. It transforms a large number of correlated variables to a new set of independent factors (groups of variables) called principal components (PC) that could explain most of the total variation in the data set. A principal component's Eigen value greater or equal to one is a criterion for statistical significance. All the statistical calculation for PCA is performed on XLSAT version 2015. PCA is performed on auto-escalated data organized in a matrix. The variables used are pH, COD, VFA, OLR, T, alkalinity, HRT, CH_4, and biogas. Efficiencies for each point of sampling are also added at variables columns and the samples are divided in different classes according to the degree of efficiency.

Pearson correlation analysis is performed to determine the relationship between kinetic parameters and operational conditions (SPSS.20). Additionally, to test for differences in the methane production and those parameters of each sample in relation to operational conditions, one-way ANOVA, analysis of variance of means.

3. Results and discussion

3.1. Anaerobic digestion: performances and stability

The process dynamic is evaluated from the stability and methane yield production. To do this, Fig. 1 shows the progress over time of the three process stability indicators (pH, CH_4 yield and VFA) as function of OLR for reactors during the one-year experiment.

In terms of stability, Fig. 1a gives the best information on the state of the process, since the pH value remains permanently between 7.2 and 8.2 throughout the study period [4]. Besides, the changing of VFA concentration is the major influencing factor of pH during AD. pH desired range for methanogens is generally in the range (6.5–8.2) which means that an appropriate VFA concentration is very crucial to AD system [17]. At the beginning, the biological activity for all microorganism is high in the reactor. So, the generation of VFA is immediate and massive. The concentration of VFA increases during the first month of the treatment and reaches a maximal value of 47 meq/L. A decrease of VFA to an average value of 12 mg/L is also observed from 90th day. This VFA decline indicates a good methanogenic activity in the reactor; this implies that the acidogenic bacteria produce VFA that can be utilized by the acetogenic and methanogenic bacteria. After eight months, the produced VFA accumulated in the reactor increases progressively to reach 50 mg/L. This result is a sign of the start of an incomplete degradation of the organic substrate which can be caused by the deterioration of methanogenesis.

The methane yield during the start-up period is presented in Fig. 1b. Methane yield follows approximately the same trend as the OLR. Biogas production starts on the 20th day with r_{CH_4} of 0.1 L CH_4/L d and methane content of 55%. At the end of stage 1 the r_{CH_4} and methane content are improved to 0.9 L CH_4/L d and 80% with a methane yield of 0.4 m³ CH_4/kg COD. Toward the stage 2, the r_{CH_4} and methane content are decreased to 0.5 L CH_4/L d and 71% with a methane yield of 0.15 m³ CH_4/kg COD. During stage 3, the r_{CH_4} and methane yield increases proportionally with the OLR, as the increasing readily available organic matter is converted to biomass. The r_{CH_4} reaches 3 L CH_4/L d which is equivalent to the methane yield of 0.4 m³ CH_4/kg COD. This result again indicates the satisfactory start-up of the anaerobic compartment. In the stage 4 methane content and methane yield decline progressively. This decline can be attributed to the presence of VFA hat have a strong influence on the biogas and methane content.

3.2. Statistical analysis of the variables

A statistical summary of the operating parameters measured in the anaerobic digester is shown in Table 3.

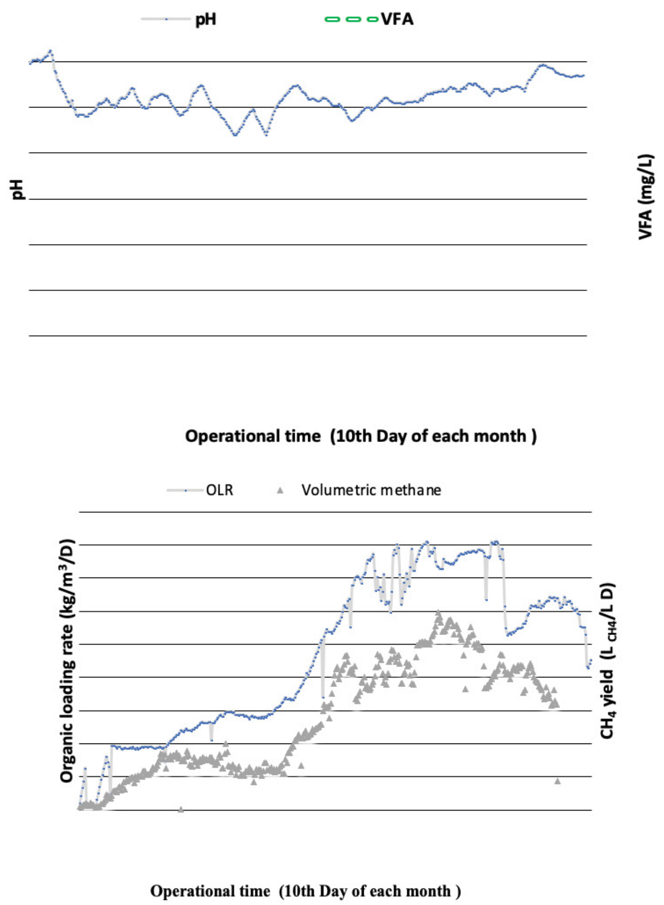

Fig. 1. Progress over time of the three process stability indicators (pH, CH_4 yield and VFA) as function of OLR.

Overall, there is high variability of each parameter over time as indicated by the minimum, maximum, average and standard deviation values for each measurement.

3.3. Coefficient de corrélations linéaires: Pearson

Pearson's correlation analysis is used to assess pair-wise correlations among the pH, VFA, OLR, alkalinity, HRT, CH_4, and biogas of Sotrameg vinasse. Fig. 2 shows the value of Pearson coefficient between the methane production from Sotrameg vinasse and certain variables composition.

Fig. 2 reveals that methane production is positively and strongly correlated with OLR (r = 0.946; p < 0.01), pH (r = 0.948; p < 0.01) and alkalinity (r = 0.152; p < 0.01). It implies that the degradable organic matter present in vinasse contributes most to their methane yields. Moreover, methane production is significantly and negatively correlated with TRH (r = –0.843; p < 0.01). However, the methane and VFA offer a correlation but not statistically significant (r = 0.061).

3.4. Principal component analysis

The PCA is performed to investigate the possible interactions between the characteristics (chemical, physical and biological) of Sotrameg vinasse in relation to the feed stocks, operating parameters (OLR, HRT), biogas production and methane production. Thus, the corresponding analysis defines the main components, which are presented in Fig. 3. Obtaining a minimum number of components that could explain most of the total variation in variables is the main objective of the test. In order to extend the representativeness of the statistical analysis, two main components PC1 and PC2 are selected by the Eigen value (>1) as proposed by Kaiser [18] and Nair et al. [19], which explain almost 81.95% of the overall variance with respectively 59.74% for axis 1 and 22.21% for axis 2. The other components from F_3 to F_9, respectively explain 8.82%, 5.05%, 1.82%, 1.24% and 1.11% of the total variance of the dataset (Fig. 3).

The main F can be represented by a characteristic value, which allows for the partitioning of the total variation accounted for each principal component via a linear combination of the original variables as follows as emphasized by Anderson [20]. The two F_1 and F_2 are represented in terms of chemical descriptors by Eqs. (2a) and (2b) and their coefficients demonstrate that all of them have similar load contributions on Factor 1 and Factor 2.

$$\begin{aligned} F_1 = & -0.007\text{OLR} + 0.859T + 0.982\text{pH} + 0.988\text{COD\%} \\ & - 0.914\text{HRT} + 0.051\text{VFA} + 0.135\text{Alkalinity} \\ & + 0.960\text{CH}_4 + 0.960\text{Biogas} \end{aligned} \tag{2a}$$

$$F_2 = 0.0817\text{OLR} - 0.385T + 0.038\text{pH} + 0.003\text{COD\%} - 0.080\text{HRT} + 0.673\text{VFA} + 0.824\text{Alkalinity} + 0.206\text{CH}_4 + 0.027\text{Biogas} \quad (2b)$$

Classes are separated along Factor 1 and 2 direction. According to Eq. (2a), loading indicates that pH and T are the variables that most contribute to this component composition, while according to Eq. (2b), loading indicates that VFA, alkalinity and OLR are the variables that most contribute to this component composition. This chemometric information is coherent with AD aspects, since VFA, alkalinity and pH are related to methanogenic activity (responsible for the major COD removal), when its feed tank values are greater than 5 g/L, it caused a decrease on the treatment efficiency.

Fig. 4 illustrates the correlation circle of the various operating parameters in the two first components F_1 and F_2 during the AD of the vinasse. Each line represents an individual variable. The cosine angle between any two line will determine the correlation between the variables [21]. If cosine angle between two variables tends to zero (cos 0° = 1), the relationship between the variables is positively correlated and if it tends to 180 (cos 180° = −1) then the variables are inversely proportional.

It was found from Fig. 4 shows that the correlations tend to present three data groups with well-defined patterns, indicating that stabilization and adaptability to different loads is important in reactor performance. Although, the first group corresponding to four nearly horizontal and very tightly knit variable markers such as biogas, pH, methane and COD%. The second corresponding to three nearly vertical variable markers OLR, VFA and alkalinity. The last group corresponds to single variable which that is HRT. According to the first component F_1, analysis of the results shows that in the positive axis, biogas, methane production, pH and T are strongly correlated. However, the close correlation between pH and specific biogas and methane yield confirms that pH is the strong governing factor of the process performance. In addition, the temperature and % COD removal are shown by PCA as important parameters

Table 3
Statistical summary of the operating parameters

Variables	Minimum	Maximum	Average	Standard deviations
pH	7.0	8.2	7.60	0.17
T (°C)	34.30	39.90	37.78	1.41
$\text{COD}_{\text{input}}$ (mg/L)	499.99	12,264.00	6,928.13	3,536.65
OLR (kg/m^3 d)	0.10	4.8	2.45	1.20
HRT (d)	10.52	50.00	17.55	7.16
VFA (mg/L)	11.20	52.40	22.03	11.75
Alkalinity	188.00	294.00	230.40	28.12
Biogas (m^3/d)	100.00	11,660.00	5,343.96	3,356.01
CH_4 (m^3/d)	8.00	7,637.80	3,304.77	2,026.68

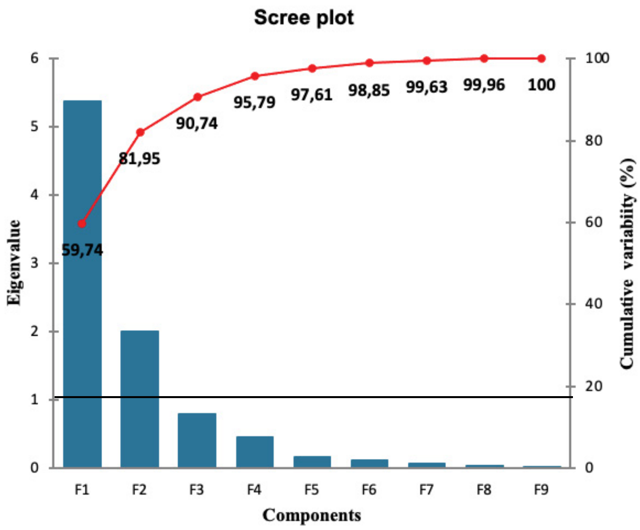

Fig. 3. Results of the PCA in main components.

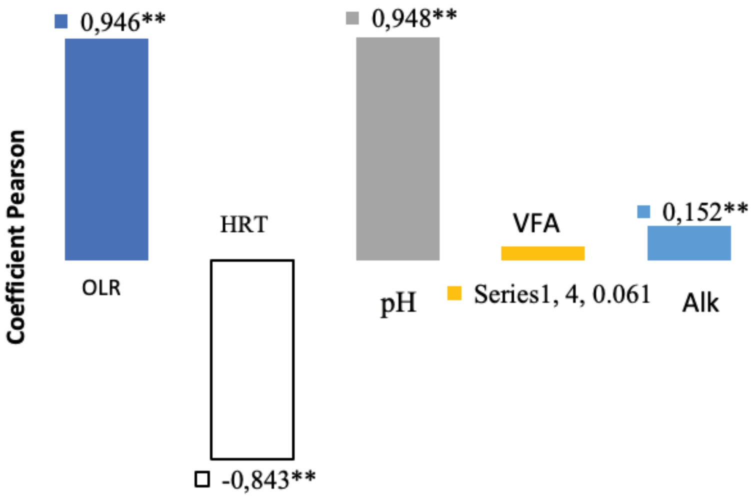

Fig. 2. Pearson's correlation coefficients of methane production with process parameters statistically significant values are indicated by symbols: **$P < 0.01$; *$P < 0.05$.

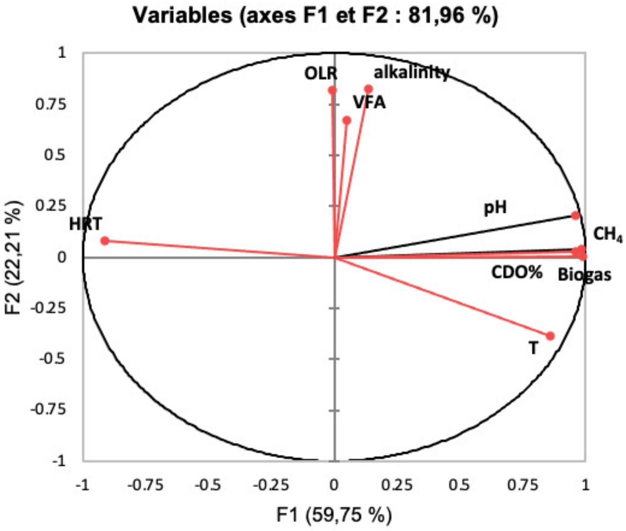

Fig. 4. Correlation circle of variables plot components F_1 and F_2.

that affect the rate of methane production and reactor's pH during the degradation of vinasse. The analysis also shows that the rate of methane production is positively affected by reactor's temperature. This result is consistent with the findings of Xu et al. [22] where the authors reported that the decrease in the gas production rate at lower temperature was due to the decrease in biological activity.

In contrast, opposite correlation is found between methane production and HRT. While in the positive side of component 2: OLR, alkalinity and VFA are also correlated and a very close relationship between alkalinity and VFA. After exploring the methane production potential with an advanced statistical treatment, PCA reveals that the stabilization of pH in the digester has a positive effect on specific biogas yield and methane content. This information is coherent with AD aspects and it represents a basic reference for their development for the vinasse treatment.

4. Conclusion

In this study, anaerobic treatment of vinasse in a full-scale digester is investigated during a period of one year. The reactor indicates a satisfactory performance during the mesophilic AD of Sotrameg vinasse, thanks to the stability of physicochemical parameters with biogas production containing 71% of proper methane. However, the experimental runs show also that AD system is distinctly influenced by OLR in terms of VFA, methane rate and system stability. Moreover, in the whole operation period, the content of VFA indicates a good methanogenic activity in the reactor; this implies that the acidogenic bacteria produce VFA that can be utilized by the acetogenic and methanogenic bacteria. Consequently, a progressive decline of methane yield is attributed to the presence of higher values of VFA which could presumably lead to the inhibition of the AD process.

The statistical analysis using PCA identified a reasonable and significant correlation between operational and process parameters and could be summarized that (i) pH directly influenced the maximum biogas production, methane yield and % COD removal, this can be explained by the high positive correlation among this variables, (ii) PCA shows that the temperature is an important parameters that affect the rate of methane production and reactor's pH during the degradation of vinasse, (iii) A strong correlation between VFA and alkalinity during the AD process. Finally, this chemometric information is coherent with AD aspects, since VFA, HRT, *T*, alkalinity and pH are related to methanogenic activity responsible for the major COD removal and methane production. In addition, the models developed in this study can provide guidance for future feedstock evaluation and process optimization in AD.

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