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Factorial experimental design to enhance methane production of dairy wastes co-digestion

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ABSTRACT

Factorial design was used to investigate the parameters involved in co-digestion mixture of dairy wastes (from a Moroccan dairy industry) in order to improve methane production of this mixture. Indeed, evaluation of methane yield as a function of three parameters (pH, inoculum and organic load) showed the correlation between the experimental and statistical data in terms of pH 8 and inoculum 1 (constituted by sludge diluted in 1 L of basal medium of methanogenic bacteria, in addition to formic acid (5 mL L^{-1}), propionic acid (5 mL L^{-1}), lactic acid (5 mL L^{-1}) and micro-nutrient (10 mL L^{-1})) as optimum parameters. However, a discrepancy was detected about organic load. The interaction between parameters had a positive effect on methane yield because it led to produce experimentally a maximum methane using the higher load (3.44 g VS). These results allow selecting the parameters for the improvement of methane production. Furthermore, the validity of the fitting model to describe and improve the efficiency of dairy wastes co-digestion was investigated. In addition, an abatement of 89% of volatile solids was observed and the mineral solids was increased from 4 to 7.2 g L^{-1}, which is important of digestat value as fertilizer.

1. Introduction

Nowadays Morocco has becomes a big producer and consumer of milk and its derivatives according to the Ministry of Agriculture and Rural Development. Dairy production increased from 475 million liters in 1970 to 1300 million liters in 2005 with an increment annual rate of 3–7% [1,2]. The water consumption was equivalent of 2–7 times of the milk volume treated and the diversity of the product manufactured. Therefore, dairy industries are a major consumer of water and the largest producers of pollution. Indeed, dairy effluents were characterized by their higher COD and microorganism content [3,4]. The management of these effluents worries several producers and environmental actors.

To reduce the environment and public health impact of these wastes, several treatment and/or valorization process are used. The choice of one of these treatments depends mainly on the physicochemical and biological characteristics of the dairy wastes in terms of organic matter biodegradability, presence or absence of pathogenic germs, acidity, composition, etc.

Composting the solid organic matter waste is typically used for sewage and dairy sludge [5,6]. However, this technology demands a large space and control of temperature. Physical-chemical and biological treatment are also used to treat dairy wastewater [7–10] but the cost of reagents used in physical chemical treatment is expensive and the aerobic biological treatment requires high energy. Anaerobic digestion is a very promising biological technology to treat dairy wastewater. This technology is based on series of biological processes in which microorganisms break down biodegradable organic matter in the absence of oxygen to final products consisting mainly of a biogas - composed of methane (55–70%), carbon dioxide (25–40%) and trace gases of hydrogen sulfide (H_2S) - and a digestat which can be used as fertilizer for agricultural soils [11]. However, the dairy waste composition in terms of nitrogen, acidity, alkalinity, and germ composition makes anaerobic monodigestion of dairy waste very difficult [10]. For these reasons,

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anaerobic co-digestion (ACD) is used to remediate the problem encountered during mono digestion of these wastes.

ACD is an effective technique used for treating dairy wastes [12,13]. Nevertheless, the factors optimized during ACD (pH, buffer capacity, strength and duration of agitation, temperature, retention time, pretreatment, load) [14–16], need to be investigated to control these independents parameters. The statistical modelization of the response (methane yield) as a function of input parameters using experimental design is currently investigated in different areas. However, the use of this method for anaerobic digestion of some wastes has been reported in the literature. The results obtained on these works are very promising in terms optimum parameters (environmental factors, feeding composition, co-digestion, among others) and the interactions between them [17–21]. One particular study was performed to evaluate the effect of four factors using 2^4 full factorial designs for four substrates (anaerobic sludge, garden waste, cellulose and lipid rich waste). The results indicate that the ambient temperature was found to be the most significant contributor to errors in the methane potential [21]. Zou et al. show that orthogonal experimental design is more suitable to optimize time for ultrasonic pretreatment in anaerobic digestion of dairy manure pretreatment to improve methane production [18]. For Oliveira et al. [20] reported that co-digestion with glycerol (Gly) or waste frying oil is a promising process to enhance the biochemical methane potential (BMP) from the macroalgae *Sargassum* sp. Indeed, the higher BMP (283 ± 18 L CH_4 kg^{-1} COD) and *k* (65.9 ± 2.1 L CH_4 kg^{-1} COD d^{-1}) was obtained with 0.5% total solids (TS) and 3.0 g Gly L^{-1}.

The objective of this work is to study the efficiency of multivariate statistical techniques (experimental design) in ACD of four wastes generated by a Moroccan dairy industry. For that, three parameters were chosen (pH, inoculum and organic load) and evaluated in order to determine optimum parameters and their interaction. Therefore, ACD could be improved by using the fitting mathematical model.

2. Materials and methods

2.1. Origin of the substrate

Four dairy wastes from a Moroccan dairy industry situated at 7 km south of Taroudant were selected (physical chemical sludge (PCS), liquid biological sludge (LBS), pure whey (PW) and loss in dairy product (LDP)). Two of these wastes were collected in wastewater treatment plants located in this industry. The treatment plant treats 41 300 m^3 (in average) of effluents monthly using physicochemical treatment yielding the production of PCS and biological treatment generating liquid biological sludge. The amount of organic matter (OM) produced in these wastes was 1188 T yr^{-1}; this makes them a serious environmental problem. The sample of LBS was performed at 40 m^3 tank wherein this sludge is stored, while the PCS was taken at flocculation/flotation tank. Also, 20 000 L d^{-1} (in average) of milk are destined to the cheese manufacturing of this industry. Approximately, two thirds of this account was releasing as PW generating in large quantities (940 T OM yr^{-1}) and it was collected in cheese separation unit. The LDP was collected at the washing unit. This substrate includes all unsold and expired dairy products in addition to the loss in machines and laboratories of analysis (milk, yogurt, fresh cheese, flan, chocolate dessert, etc.).

For each waste a volume of 5 L was retrieved to constitute the mixture used in this study. The characteristics of wastes and the mixture are presented in Table 1. The mixture was prepared according to the percentage of production of each dairy waste by this industry.

Table 1
Characteristics of substrates.

Parameter	PCS	LDP	PW	LBS	Mixture
PH	6.4	5.3	4.8	7.9	5.0
Alkalinity as $CaCO_3$ mg L^{-1}	5870	1570	1230	3380	1200
Total Solids (TS) (g L^{-1})	34	214.8	62.8	15.2	44
Volatile Solids (VS) (g L^{-1})	24	205.5	55.6	11.2	38.4
Mineral Solids (g L^{-1})	10	9.3	7.2	4	5.6
VS (%)	71	96	89	74	87

The mixture was prepared with the following proportions: 23.9% PCS + 42.4% of LBS + 27.6% of PW + 6.1% LDP.

2.2. Physicochemical analyses

The physicochemical analyses have been focused on pH measured by a pH meter (AD 1030 pH/mV) and the alkalinity, which was determined with standard methods by pH titration until 4.5. While TS was determined at 105 °C and the VS at 550 °C [22].

2.3. Experimental procedure

The batch reactors were used to determine BMP of dairy wastes mixture in laboratory scale. These reactors used opaque serum bottle of 200 mL.

In each reactor, 40 mL of the principal inoculum and 5 mL of inoculum 1 (IN_1) or inoculum 2 (IN_2) were added to 45 or 90 mL of dairy wastes mixture which corresponded to 1.72 and 3.44 g of VS, respectively. The inoculum (IN_1 and IN_2) were added to the principal inoculum to increase the methanogenic and/or acetogenic germs and assess their effect on methane production. The initial pH was adjusted into reactors to 7 or 8 by the NaOH (2 N), whereas alkalinity was adjusted by adding 20 mL of $NaHCO_3$ solution (18 g L^{-1}). The mesophilic condition (38 ± 1 °C) was maintained using a bath thermostat. The NaOH (9 N) solution was used to remove the CO_2 from biogas and methane production was measured using displaced water technique at standard conditions (0 °C, 101 kPa) [23]. Indeed, the control tests were performed to determine the quantity of endogenous gas in inoculums (principal, IN_1 and IN_2). All tests were triplicated and the averages were reported in the results.

The Inoculums were prepared by the sludge taken at an anaerobic tank from wastewater treatment plants located in M'Zar, Agadir. The sludge was washed and sieved before being divided into three inoculums:

- **Principal inoculum:** 600 g of sludge diluted in 10 L of distilled water and stored in ambient temperature.
- **Inoculum 1 (IN_1):** 250 g of sludge diluted in 1 L of basal medium (g L^{-1}) (KH_2PO_4 0.41; $Na_2HP0_4 \cdot 7H_2O$ 0.53; NH_4Cl 0.030; NaCl 20; $CaCl_2 \cdot 2H_2O$ 0.11; $MgCl_2 \cdot 6H_2O$, 0.11; $NaHCO_3$ 5; $Na_2S \cdot 9H_2O$ 0.3; cysteine 0.3; resazurin 0.001; yeast extracts 1; biotrypcase 1) of methanogenic bacteria, in addition to formic acid (5 mL L^{-1}), propionic acid (5 mL L^{-1}), lactic acid (5 mL L^{-1}) and micro-nutrient (10 mL L^{-1}) for developing acetogens bacteria and methanogenic archaeas [24].
- **Inoculum 2 (IN_2):** 250 g of sludge diluted in 1 L of basal medium of methanogenic bacteria, in addition to acetic acid 5 mL L^{-1}, methanol 5 mL L^{-1} and micro-nutrient 10 mL L^{-1} for developing the methanogenic archaeas [24].

Table 2
Characteristics of inoculums.

Parameter	IN_1	IN_2	Principal inoculum
pH	7.82	7.59	5.86
Alkalinity as $CaCO_3$ mg L^{-1}	5000	4000	1900
VS (g L^{-1})	9.21	6.24	6.33
VS (%)	35.2	29.0	74.8
TS (g L^{-1})	26.2	21.5	12.8
TS (%)	2.66	2.19	1.29
Moisture (%)	97.3	97.8	98.7

The pH of the inoculums IN_1 and IN_2 was adjusted to 7.73 then incubated at 38 °C during four weeks. The physical-chemical characteristics of the three inoculums are presented in Table 2.

2.4. Statistical study

In this study, 2^3 full-factorial experimental design was employed to assess the influence of three parameters (pH, organic load and inoculum) (Table 3). For each factor, two levels were selected: low level (−1) and high level (+1) (Table 4). The data were analyzed using the Nemrodw_OPEX_2007 software. The polynomial equation based on the first-order model with three parameters (X_1, X_2, and X_3) is represented in Eq. (1) [25].

$$Y = a_0 + a_1X_1 + a_2X_2 + a_3X_3 + a_{12}X_1X_2 + a_{13}X_1X_3 + a_{23}X_2X_3 + a_{123}X_1X_2X_3 \quad (1)$$

where Y is the response calculated by the model (methane yield) and a_0 represents the sum of the methane yields calculated in these tests on number of experiment $\left(a_0 = \frac{\sum_{i=1}^{N} Y_i}{N}\right)$. X_1, X_2, and X_3 are coded variables corresponding to pH, inoculum and organic load, respectively, and the X_1X_2, X_1X_3, X_2X_3 and $X_1X_2X_3$ represent interactions between the individual factors. The a_1, a_2 and a_3 are the linear coefficients while a_{12}, a_{13}, a_{23} and a_{123} represent the interactions coefficients.

Table 3
Factorial experimental design matrix.

Essay	Combination facteurs		
	X_1	X_2	X_3
	pH	Inoculum	Organic load (g VS)
1	7	IN_2	1.72
2	8	IN_2	1.72
3	7	IN_1	1.72
4	8	IN_1	1.72
5	7	IN_2	3.44
6	8	IN_2	3.44
7	7	IN_1	3.44
8	8	IN_1	3.44

Table 4
Experimental domains and level of factors.

Factors	Low level (−1)	High level (+1)
Quantitative factors		
PH	7	8
Load	1.72 g	3.44 g
Qualitative factors		
Inoculum	IN_2	IN_1

The main effect (coefficient) may be calculated as the difference between the average of measurements made at the high level (+1) and low level (−1) of the factor. A large positive or negative coefficient indicates that a factor has a large impact on response (positive or negative respectively); while a coefficient which close to zero means that this factor has a less or no effect [26].

3. Results and discussion

3.1. Optimal parameters in experimental test

Fig. 1a presents the methane yield as a function of time in all performed tests. The test 8 gave the maximum accumulation which corresponded to methane yield of 93.3 NL kg VS^{-1} (Fig. 1b). The conditions applied in this test were: pH 8, IN_1 and organic load of 3.44 g VS. Decreasing organic load to 1.72 g VS under the same conditions (test 4) yielded second highest methane yield (89.8 NL kg VS^{-1}) (Fig. 1a and b). Therefore, both pH 8 and IN_1 parameters improved methane production in co-digestion of dairy wastes. However, after the ACD process the final pHs all decreased as compared to the initial pHs for all test runs (Fig. 1c).

3.2. Kinetic study

In the present work, the experimental results were analyzed according to the most frequently used models in batch system; pseudo-first-order [27,28] and Monod-type alternative approach [29]. To confirm the best kinetic model, the coefficient of determination was used to see the correlation between experimental data and the model-predicted values. The pseudo-first order and Monod equations are expressed as following Eqs. (2) and (3), respectively.

$$BMP_{(t)} = BMP_{\infty}\left(1 - \exp^{-k_h t}\right) \quad (2)$$

$$BMP_{(t)} = BMP_{\infty}\left[\frac{k' t}{1 + k' t}\right] \quad (3)$$

where $BMP_{(t)}$ is the cumulative methane production at time t [L CH_4 kg VS^{-1}], BMP_∞ is the ultimate methane production [L CH_4 kg VS^{-1}], k_h and k' are the rate constants for the first order and Monod equations, respectively.

The values of the BMP_∞, k' and k_h can be derived from the slope of plotted experimental data using the linearized version of Eqs. (2) and (3). The linear plots of BMP kinetics and the calculated kinetic parameters are given in Fig. 2 and Table 5. As can be seen, the correlation coefficients obtained from Angelidaki et al. [27] approach were high than those obtained from Monod model in all tests.

The values of BMP_{∞}, k' and k_h presented in Table 5 indicate the positive effect of the pH 8 on methane production of dairy wastes mixture in tests 2, 4, 6 and 8 where the BMPs increase compared to tests 3, 5 and 7. In addition, the substitution of IN_2 by IN_1 for the same pH 8 increases BMP (tests 2 compared to 4 and 6 compared to 8). This showed the important effect of the IN_1 on promotion of the methane production from dairy wastes mixture. Theoretically, IN_1 contains acetogens bacteria and methanogens archaeas, while IN_2 contained methanogenic archaeas [24]. So, the digestion of the dairy wastes mixture needs more acetogens bacteria, which transfer volatile fatty acids into acetate, H_2, CO_2 and produces more methane [24]. The kinetic study confirms the results found in experimental test in terms of pH 8 and IN_1.

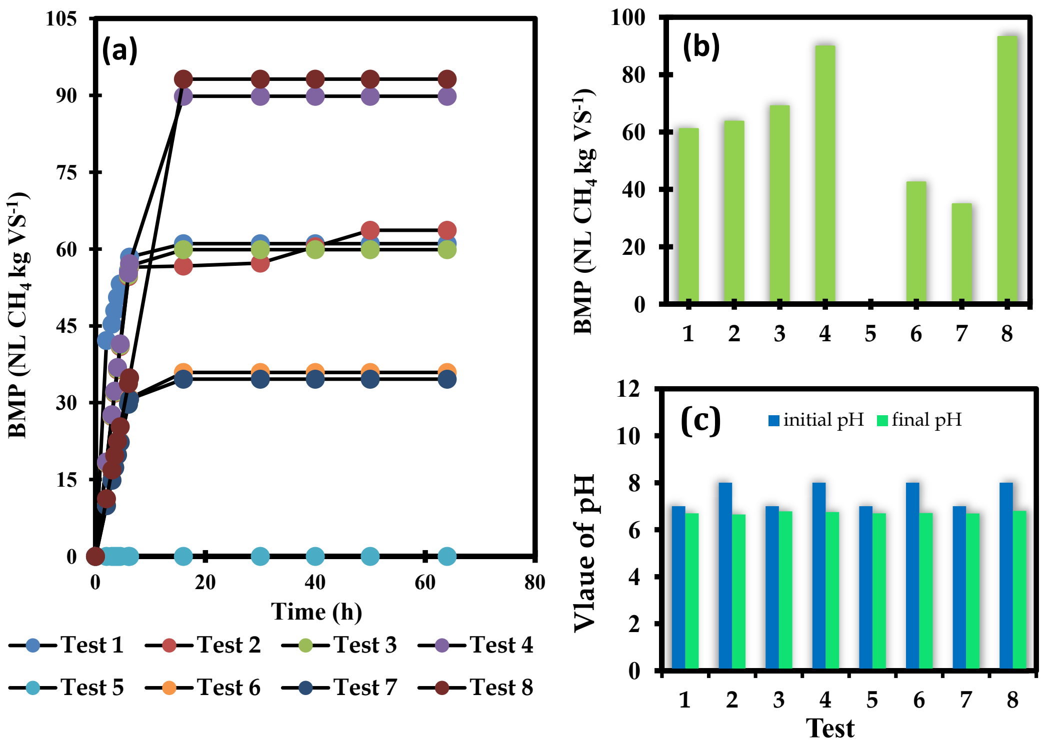

Fig. 1. Methane yield for all tests as a function of time (a), maximum methane yield for each tests (after 64 h) (b) and pH variation (initial and after 64 h) (c).

3.3. Fitting model and improvement of methane production

The parameters influenced the methane production during co-digestion were determined by statistical analysis as shown in Fig. 3. The pH, inoculum and organic load were the most parameters influencing the methanogenic potential by 31, 27 and 19% respectively (Fig. 3b). The interaction between these parameters had also an effect on methane production with a percentage ranging from 5.6 to 9.4%. This interaction showed the importance of pH when it was increased from 7 to 8 independently of the inoculum and load used. These interactions showed also the importance of IN_1 regardless the load and pH used (Fig. 3c and e). The results confirm those revealed by the kinetic study in terms of pH 8 and IN1 as the optimal parameters. However, a discrepancy was detected between experimental and statistical analysis for organic load. Indeed, statistical analysis showed that less organic load (level −1) produced more methane than a higher load (level +1) in experimental tests. However, the statistical and the experimental results yielded the same optimal parameters in terms of pH 8 and IN_1.

The difference on experimental methane production between these two charges was less important. It was only 3.6% although the high load was twice higher than the low load. We note that the increase of the charge introduced into the digester slightly increased the methane production, but it led to the saturation of the system. Indeed, the negative effect of the increase in load on reactor performance has been demonstrated in some studies [30]. Yu et al. [31] suggest that the rate of charge of OM does not have a strong impact on the methanogenic community at the temperature in which the effect is more important. In our study, the interactions between the parameters explained the origin of this difference. The substitution of IN_2 by IN_1 and pH 7 to 8 led to a multiplication of performance of ACO of dairy wastes mixture when the high load was introduced. However the same substitution had not improved methane production in the case of the low load (Fig. 3c–e). Consequently, there was another interaction between microorganisms contained in inoculum and substrate which was not taken into consideration in this work and which influenced the methanization of the mixture. On the other hand, the interaction between the three parameters was less important (0.012%). Contrary to organic load, the level +1 gave more methane for pH and inoculum (Fig. 3a). According to these results a mathematical model was proposed (Eq. (4)).

$$Y = 56.8 + 15.5X_1 + 15.0X_2 - 14.1X_3 + 4.2X_1X_2 + 9.7X_1X_3 + 6.4X_2X_3 \quad (4)$$

The validity of the model was evaluated by different tests. Indeed, the analysis of variance was a way to validate the mathematical model by using Ficher criterion test. This test is used to compare two dispersions, one due to the residual and the other due to the mathematical model [32]. The value $F_{critical}$ in table of Fisher–Snedecor with 6 and 1 degrees of freedom for a confidence level of 95% is close to 234. The value F_{obs} obtained in our work was 1374. Since $F_{obs} > F_{critical,}$ the regression explained the phenomenon studied with a confidence level of 95% (P-value = 0.0206 < 0.05) (Table 6) [33].

As seeing in Fig. 4, the experimental results were in excellent correlation with the values calculated by the polynomial equation (R^2 = 0.9999), which proved the validity of our model [34]. Furthermore, all the coefficients were different from zero (p-value < 0.05) (Table 7) [35,36]. As consequence, the proposed mathematical model is validated with a risk of 5%. Thus, for all parameters, we took the level (+1) to improve performance of ACD except for organic load for which we took the level (−1) [37].

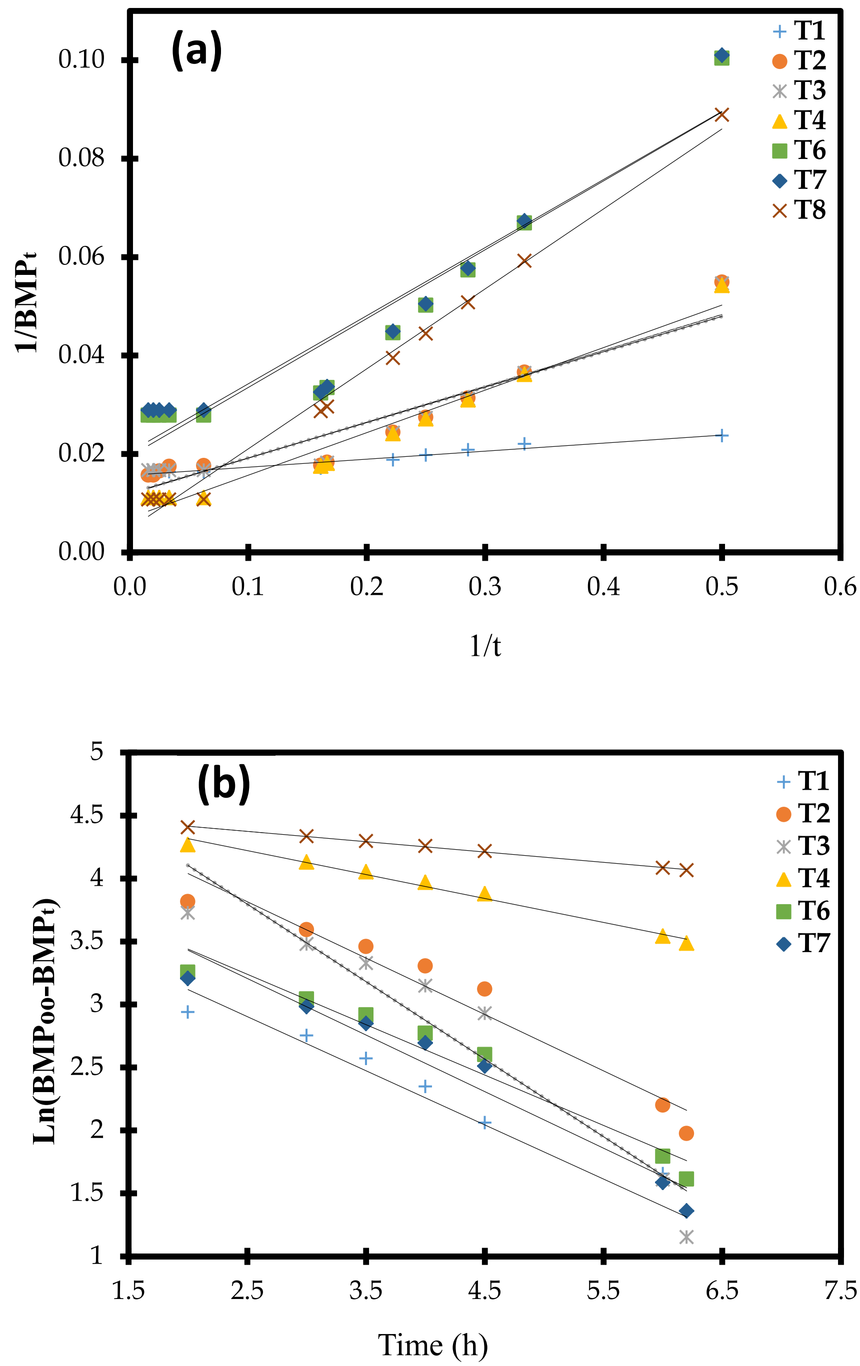

Fig. 2. Liner plot for first and monod-type kinetics.

Table 5
Values of BMP_∞ and rate constants.

Test	Monod-type alternative			First order (Angelidaki Approach)		
	BMP_∞ (L CH_4 kg VS^{-1})	k' (h^{-1})	R^2	BMP_∞ (L CH_4 kg VS^{-1})	k_h (h^{-1})	R^2
1	63.7	0.96	0.9497	123	0.64	0.9836
2	84.0	0.16	0.8913	139	0.45	0.9464
3	83.3	0.17	0.8841	208	0.62	0.9136
4	142.9	0.081	0.9661	110	0.19	0.9874
6	51.3	0.14	0.9148	76	0.45	0.9462
7	49.0	0.15	0.9059	69	0.40	0.955
8	208	0.030	0.9909	97	0.082	0.9974

However, it is interesting to work with higher load in adjusted optimal condition because it appears that these conditions improve methane production.

Based in these results, another higher load was taken and incubated; pH was adjusted at 8 and IN1 was increased to 25 mL while 20 mL of principal inoculum were introduced in addition to 20 mL of $NaHCO_3$ (18 g L^{-1}). Under the new conditions, experimental yield obtained was 176.3 NL CH_4 kg VS^{-1} with an increased by 90% compared to the test 8 (93 NL CH_4 kg VS^{-1}) (Fig. 5). The methane accumulation reached 520.2 mL during 14 d of incubation with 95% (492 mL) of the production during the two first days. In addition, the OM degradation was high with abatement of 89% of VS.

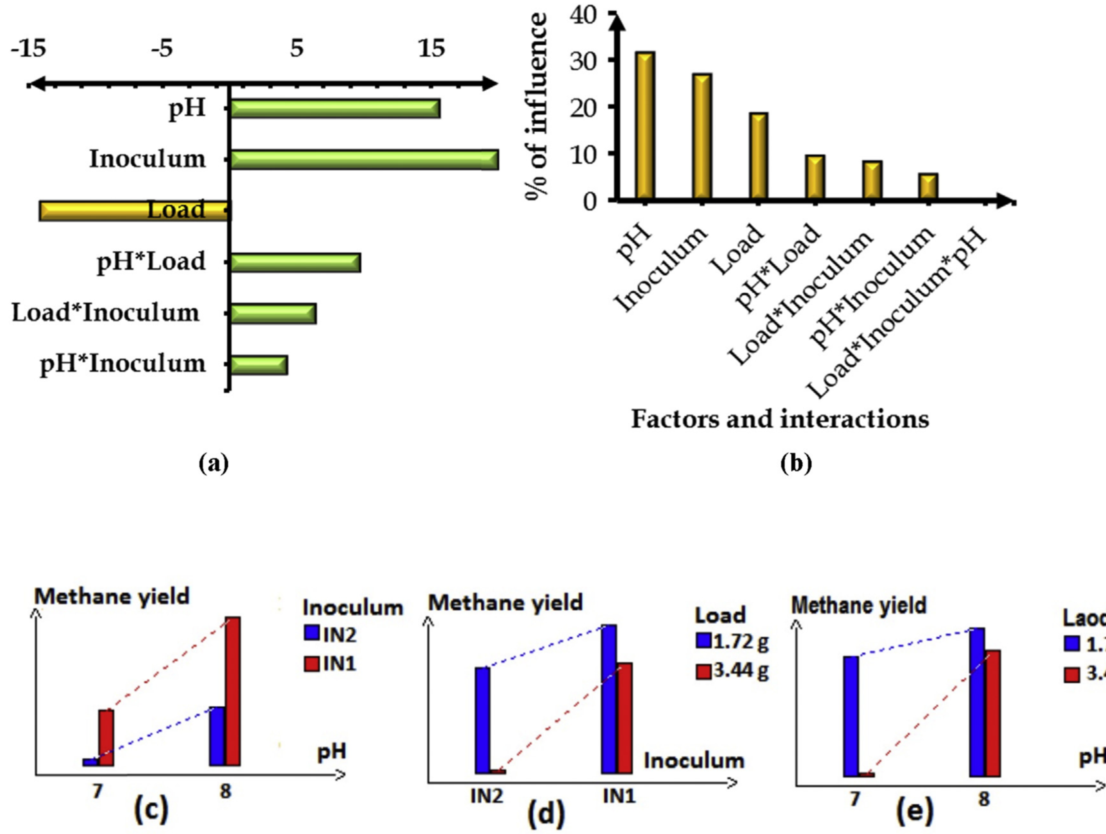

Fig. 3. Statistical study of the parameters: a) Graphic effects study; b) Pareto Analysis; c) Interaction inoculum * pH; d) Interaction Inoculum * load; e) Interaction pH * load.

Table 6
Analysis of variance and model coefficients.

	Sum of squares	Degrees of freedom	Mean square	F_{obs}	P-value
Regression	6545	6	1090.8	1374	0.0206
Residue	0.794	1	0.794		
Total	6550	7			

Table 7
Model coefficients.

Factor	Coefficient	t.exp	P-value
a_0	56.8	180.2	< 0.00353
a_1	15.5	49.3	0.0129
a_2	15.0	47.5	0.0134
a_3	−14.1	−44.9	0.0142
$a_1 * a_2$	4.3	13.5	0.0470
$a_1 * a_3$	9.7	30.7	0.0208
$a_2 * a_3$	6.4	20.4	0.0312

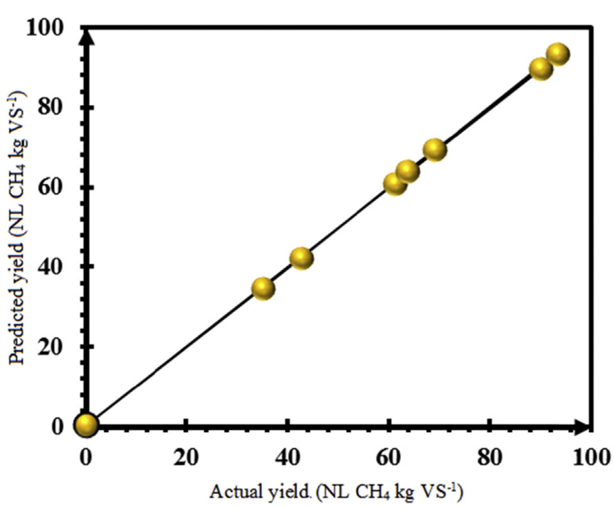

Fig. 4. Predicted yield of the methane in terms of the actual yield.

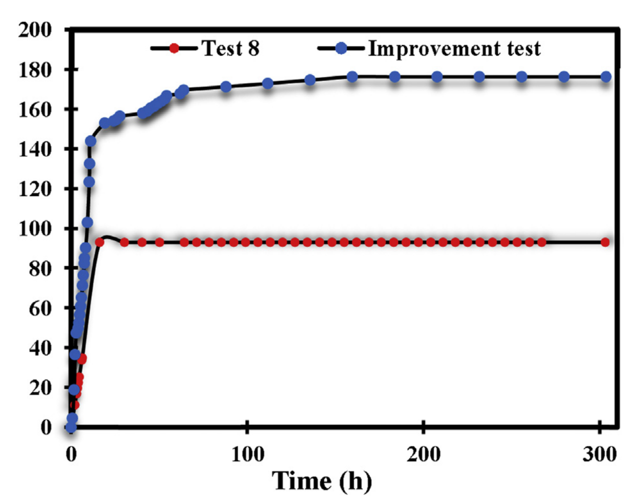

Fig. 5. Methane yield (NL CH_4 kg VS^{-1}) for the improvement test and test 8 as a function of time.

4. Conclusions

The parameters influencing the co-digestion of four dairy wastes from a Moroccan industry have been studied by applying the multivariate approach via the full factorial plan. The modeling of the co-digestion made it possible to confirm the results obtained experimentally at pH 8 with IN_1 inoculum as optimal parameters. However a discrepancy was observed for the organic load. The optimum condition selected (pH 8 and increase in inoculum 1) improved methane production of higher load of this mixture to 62% and increased methane yield to 90%. In addition, the methane yield obtained in these conditions confirms the influence of independent parameters on methane production, which can be made possible using central composite response surface modeling design.

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