Experimental Characterization of Influential Parameters of Cow Dung Substrates in Biogas Production

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Authors' contributions

This work was carried out in collaboration among all authors. Author NB wrote the equations and the experimental protocol. IO wrote the first draft of the manuscript. Authors NB, YMB and IO implemented the simple computer calculations. Authors DWNK, GWS and IO managed the analysis of the study and made corrections. Authors YMB and IO managed the bibliographic searches and checked the various calculations. Authors SD and SK checked the différent calculations and confirmed the discussion. All authors read and approved the final manuscript.

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ABSTRACT

The present work on the production of biogas from cow dung substrates was carried out at the Laboratory of Renewable Thermal Energy (L.E.T.RE) of the Joseph KI-ZERBO University in collaboration with the National Program of Biodigester of Burkina Faso (PNB-BF). We focus in this work on the determination of the physicochemical characteristics of the substrates allowing the follow-up of the biogas production. We experimented with anaerobic digestion for $100\,days$ each at mesophilic temperature, i.e. a hermetic enclosure included between 27 and $38°C$. The average flow of biogas is $Q = 2.5\,m^3d^{-1}$, and that of methane is $1.9\,m^3d^{-1}$. This methane-rich gas CH_4 with a flow rate of $1.9\,m^3d^{-1}$, i.e. a rate of 60% in the biogas, has a high energy capacity of $23kJm^{-3}$ and can be used for cooking and lighting especially in rural areas. The following physicochemical characteristics were observed for cow dung: dry matter 11.85%, volatile matter 58.83%, hydrogen potential 7.01, density $731\,kg.m^{-3}$, carbon content 33.32%, nitrogen content 1.46%, nitrogen-carbon ratio 24.70, acids are around $0.18\,kgCODm^{-3}$, and the average chemical oxygen demand $30\,kgCODm^{-3}$. This characterization was performed on a $4m^3$ pilot bioreactor with daily substrates loading ranging from 20 to $30\,kg$. The temperature of the reaction medium remained around $35°C$, optimal for the mesophilic operation of the bioreactor. The results obtained making it possible to demonstrate the possibility of producing a large quantity of flammable biogas in a continuous manner are in agreement with the literature on methanization.

ABBREVIATIONS

Symbols	senses	Units
VFA :	Volatile fatty acid	$kgCODm^{-3}$
BMP :	Biochemical Methane Potential	m^3d^{-1}
C/N:	Carbon to nitrogen ratio	- -
CT:	Total carbon	$\%$
D :	Dilution rate	d^{-1}
COD :	Chemical Oxygen Demand	kgm^{-3}
DM :	Dry matter	kg
MM :	Mineral matter	kg
m_s :	Mass of the sample	kg
VM :	Volatile matter	kg
ρ :	Density of the waste	kgm^{-3}
pH :	Hydrogen potential	- -
Q :	Biogas flow rate	m^3d^{-1}
Q_{in} :	Flow rate of the influent in the reactor	m^3d^{-1}
T :	Digester temperature	$°C$

1 INTRODUCTION

Burkina Faso, a landlocked Sahelian country with a total area of $274,400km^2$, is implicitly characterized by an energy situation that depends mainly on imported fossil fuels [1]. According to the fifth general population and housing census of Burkina Faso published in 2022, the total population is 20,505,155 with 3 488,258 households. This census shows that 76.4% of households use firewood and coal [2]. To meet energy needs and preserve the environment, it is necessary to explore and develop new energy sources such as biogas. Anaerobic digestion of organic substrates is receiving increasing attention as a way to produce renewable energy, biogas, and fertilizer (digestate) while eliminating waste and by-products [3]. The biodigester is a system of production of biogas used to meet domestic energy and fertilizer needs, but also to counter the effects of climate change. He is a receptacle hermetic semi-buried in which matter is added and mixed with water. In the absence of oxygen, the mixture decomposes producing biogas (mainly methane). This fuel is then sucked up and redirected to cooking or lighting appliances. Residual sludge, rich in nutrients, is recovered at the end of the fermentation for use as fertilizers. In 2022, 16,000 biodigesters have been

built and are functional [4]. These biodigesters have volumes ranging from $4\,m^3$ to $10\,m^3$. Solid substrates are poultry droppings, pig dung, and cow dung. cow whose biogas volumes are approximately $2\,m^3 d^{-1}$ for a bioreactor of, and the rate of methane produced around 65% of biogas [5]. The monitoring of physical-chemical parameters can improve the technical performance of the digester process and make it more attractive especially in rural areas. The applications of these characterization results will concern the modeling of small on-farm anaerobic digestion units as well as larger installations treating residues of agro-industrial origin. The application of a mathematical model to anaerobic digestion is used to optimize the process, control the operation of the digesters, to formulate and validate hypotheses to better understand the methanization reaction. This model will allow us to have a knowledge of the influential factors and to bring a correction if necessary [6]. This model will be built from the specific parameters of the substrate by determining the BMP and the degradation kinetics. The main objective of the work whose first results are presented in this publication is to determine the different components of cow substrates for future use in a model for the simulation of the operation of a reactor in continuous mode. The article is organized by presenting the materials and methods used for waste characterization as well as biogas production in the first section. The second section is devoted to the presentation of the results and their discussion by comparing them with those of the literature. The article ends with remarks and concluding words on future work.

2   MATERIALS AND METHODS

2.1   Biodigester and Biogas Measurement

To carry out the continuous tests a pilot reactor of half-cylindrical shape is built in cement brick, a material resistant to temperature and pressure. Its height is $h = 1.5\,m$ and its inner radius $R = 1.5\,m$. The resulting total volume is $V = 4m^3$ with a maximum useful volume of 2,800 liters and a minimum of 1,600 liters. The cement shell allows to control the temperature in the reactor at around $T_{reactor} = 35°C$ and makes it hermetic to control the anaerobic conditions. On the upper part of the reactor is the biogas storage dome. Also, the biodigester has an inlet basin and compost pits (Fig. 1a). The connection system allows the recovery of the biogas accumulated in the digester when the internal pressure becomes important compared to the atmospheric pressure.

The volume flow rate (Q) of the gas is obtained using the *smart-biogas* device which is a sensor using Arduino for online data acquisition (Fig. 1b). The Arduino is a microcontroller board based on the $ATmega328$ allowing the detection of the biogas flow (Flash-BMP method), temperature and pressure, and storing them directly in the memory of a computer. The biodigester model and the biogas bit measuring device are shown in Fig. 1.

(a) Biodigester

(b) Smartbiogas system

Fig. 1. Biodigester model (left) and the smart biogas system (right)

2.2 Cow Dung

The reactor was seeded with an initial load $S_{in} = 2.4\,m^3$ of cow dung from a farm in Kadiago province, Burkina Faso. To determine the kinetic parameters of the substrate that will be used to model the reactor continuously, daily feedings were performed with the addition of a dilution rate of $0.095\,d^{-1}$ to $0.228\,d^{-1}$. The substrate loading is between 20 and 30 kg and supplemented with water at a ratio of $substrates/eau = 1$. The monitoring of the reactor operation was performed by measuring the concentration of volatile fatty acids (VFA) using the Perkin Elmer Clarus 580 type GC equipped with RFID detectors at $250°C$ and FID heated to $280°C$, and the soluble COD with NOVA 60 spectroquant photometer. Also, pH, total alkalinity, the volume of biogas produced, and its methane composition are determined.

2.3 Measurement Protocol of Physicochemical Parameters

2.3.1 Organic matter

The contents of dry and volatile matter were determined according to the standardized methods implemented in the analysis of soils and organic waste $(AFNOR, 1985)$ [7]. The dry matter (DM) content is deduced by the difference in weight after passing the sample in an oven for $24\,hours$ maintained at a temperature of $150°C$. The dry matter is calculated by equation 2.1.

$$DM = \frac{m_{150°C} - m_0}{m_S} \qquad (2.1)$$

In this equation, m_0 = the mass of the empty crucible (kg), $m_{150°C}$ = the mass of the crucible with the sample after drying (g), and m_S = the mass of the sample with the crucible (g).

The mineral content (MM) is obtained by calcining the sample from $150°C$ in a muffle furnace at $550°C$ for four hours. The volatile matter content is deduced by the difference between the dry matter content (DM) and the mineral matter content (MM). The mineral matter is calculated by equation 2.2.

$$MM = \frac{m_{150°C} - m_{550°C}}{m_{150°C}} \qquad (2.2)$$

Finally, the mass of volatile matter (VM) is deduced by the difference between the dry matter (DM) and

mineral matter (MM) rates. It is calculated by equation 2.3.

$$VM = DM - MM \qquad (2.3)$$

With: MM (g), DM (g), and VM (g).

2.4 Substrates Density

A quantity of 1 to 10 g of the dry substrate is placed in a volumetric flask of suitable volume (50 to $250\,ml$) previously tared. Water is added to the mixture to the mark. The total volume of water added is determined by weighing with a Pioner precision $PX4201/E - Ohaus$, allowing the determination of the volume of the sample. The density used to calculate the initial concentration of the substrate (Q_{in}) is calculated according to equation 2.4.

$$\rho = \frac{m}{V} \qquad (2.4)$$

With: ρ = the density (kgm^{-3}), m = the mass obtained (kg) and V = the volume of the container (m^3).

2.5 Determination of Organic Carbon and Total Nitrogen

The binder thermostat type furnace $DIN\,12880$ and the $NAbertherm$ type incinerator series $L1/12 - LT40/12$ allow the calculation of the carbon content. This method of determining the total carbon content is the standard $ISO\,10694\,(1995)$ [8]. The total carbon is calculated by equation 2.5.

$$TC = \frac{VM}{2} \qquad (2.5)$$

In this equation, TC = total carbon (%) and VM = volatile matter (%).

The technique used for the determination of nitrogen is the $Kjeldahl$ method developed in 1965 [7]. The sample collected is mineralized, consisting of the conversion of protein nitrogen from organic waste to ammonia nitrogen by oxidation of the organic matter in concentrated sulfuric acid at high temperature in the presence of 5 grams of $Kjeldahl$ catalyst $CuSO_4$ and a salt. The nitrogen content of the sample is calculated by equation 2.6.

$$N_{NTK} = \frac{0,0014\text{x}0,1\text{x}\,(V_2 - V_1)}{m_s}\text{x}10 \qquad (2.6)$$

With: m_S = the mass of the test sample; V_2 = the volume of sulfuric acid added from the indicator turns from green to pink and V_1 = the volume of sulfuric acid used for the blank determination. The titer of the solution is 0.1 and the molar mass of the nitrogen is $M_N = 0.0014\,kgm^{-3}$.

2.6 Dilution Rate Charges Made

The digester is fed with the substrate at an inlet flow rate obtained in the experiment noted D. The dilution D is defined as the ratio between the flow rate of the influent (Q_{in}) and the volume of liquid (V_{Liq}) in the digester [9]. Two phenomena can occur, a low dilution can lead to a crusting of the substrate but a high dilution can also lead to a leaching of the reactor. The dilution rate is obtained by equation 2.7.

$$D = \frac{Q_{in}}{V_{Liq}} \qquad (2.7)$$

In this equation, Q_{in} = the concentration of the substrate to be vented (in $m^3 d^{-1}$); V_{Liq} = the volume of liquid (in m^3) and D = dilution rate (in d^{-1}) over periods of $100\,days$.

3 RESULTS AND DISCUSSIONS

3.1 Physicochemical Parameters of the Reactor

A characterization (MV, DM, COD, VFA, and biogas flow rate) on the capacity of the bacteria in the removal of substrate overloads and their persistence over time within the reactor is conducted at the beginning and during the digestion period. Table 1 shows the composition of the substrate. The pH of the reaction medium remained around neutrality i.e. 7.015 which is to the advantage of using cow dung. Studies in the literature have shown that the pH of the substrate of farmers is neutral (7.015 ± 0.23) ranging between 5 and 9 for solid waste [10]. The dry matter (DM)

content is part of the criteria for the classification of substrates according to their ability to be more or less degradable in biochemical ways. This substrate with a dry matter content of 11.38% ± 0.6 is suitable for anaerobic digestion by the *wet method*, which is adapted to the treatment of waste with a dry matter content of between 5 and 20% (DM). The COD values at the outlet are approximately equal to those at the inlet. It is the same for the acids.

These remarks are because the daily loading increases the levels of these acids and the COD in the reactor, which the bacteria are unable to eliminate. It is possible to deduce that, to obtain a high COD abatement, a significant digestion time must be considered. In this study, the ratio of carbon to nitrogen is 24.70 (in Table 1) and therefore in the order of the optimal values which are around 20 and $30\,kg$ [11]. The composition of substrates in anaerobic digestion plays a role in the growth rate of anaerobic bacteria and the production of biogas. Carbon is the main source of energy for microorganisms while nitrogen is an essential element for the synthesis of amino acids and proteins. Nitrogen is converted to ammonia which, as a strong base, neutralizes volatile acids produced by fermentative bacteria and thus helps to maintain the neutral pH conditions essential for cell growth. On the other hand, an overabundance of nitrogen in the substrate can lead to excessive formation of ammonia, leading to toxic effects. Density, volatile matter, and COD are necessary for the determination of input parameters such as (Q_{in}) of the reactor. The nitrogen content of 1.35% is still below the toxicity threshold of this compound for digestion ranging from 1.5 to $3\,kgNm^{-3}$ [12].

Table 1. Physicochemical characterization of cow substrates

$Features$	DM	VM	ρ	VM	C	N	COD	VFA	pH	C/N
$Units$	$\%$	kgm^{-3}	kgm^{-3}	$\%$	$\%$	$\%$	kgm^{-3}	kgm^{-3}	$--$	$--$
$Values$	11.38	895.23	731	58.83	33.32	1.35	30.882	0.18	7.015	24.705

3.2 Dilution of the Substrates Applied in the Reactor

The profile of the results of the temporal evolution of the dilution rate D in the 4000 liters reactor is given in Fig. 2. The horizontal areas correspond to the time interval dates when the same loads were performed. The inlet flow rate was chosen with a constant evolution in pieces between $D = 0.095\,d^{-1}$ and to $D = 0.228\,d^{-1}$ observe its influence on the dynamic behavior of the chemical species. The first loading gives a dilution of 0.11 per day and after we observed a fall between the 10th day and the 20th and a gradual rise in load whose average is around $D = 0.15\,d^{-1}$. To improve the productivity capacity of the biogas, we carried out heavy loading of the substrate after the twentieth day.
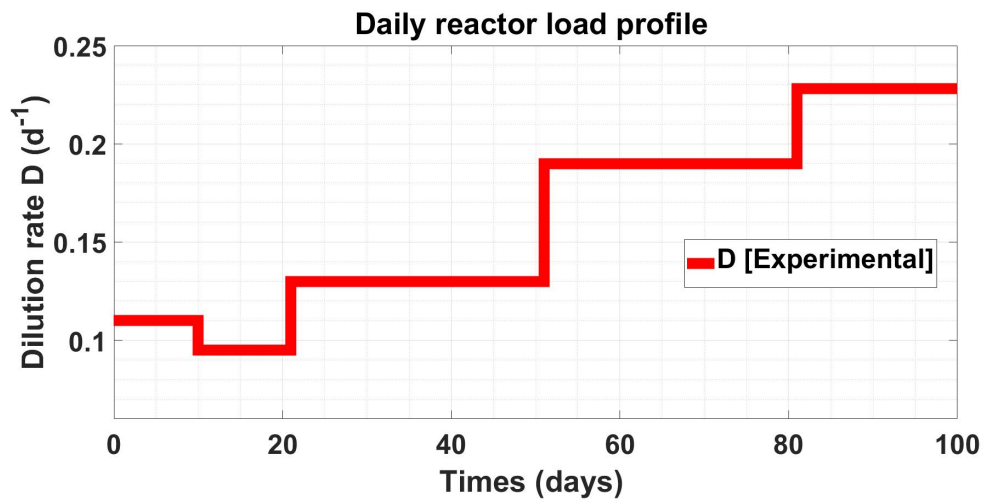
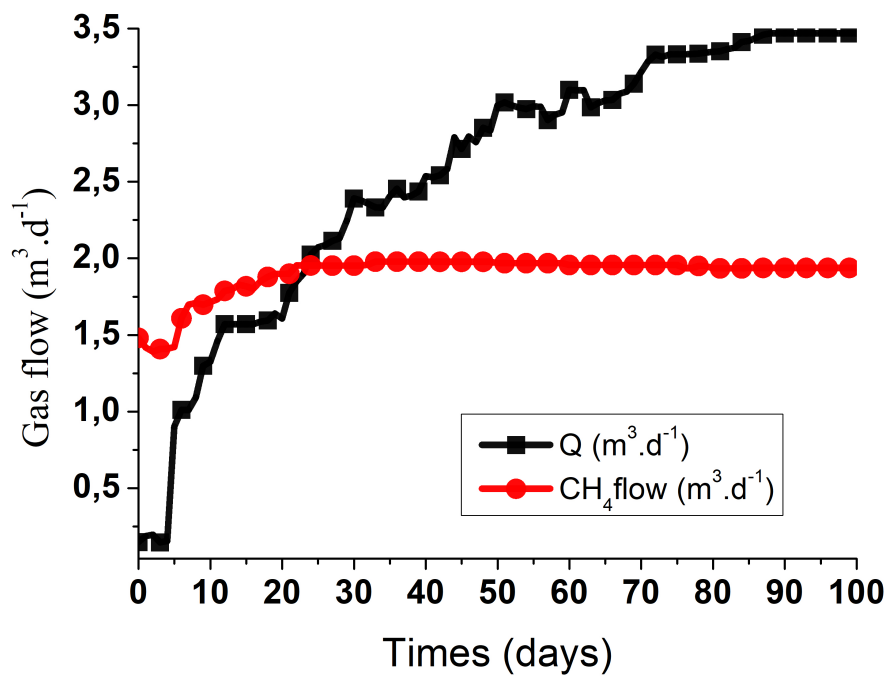**Fig. 2. Loads of substrates dilution rates applied to the reactor**

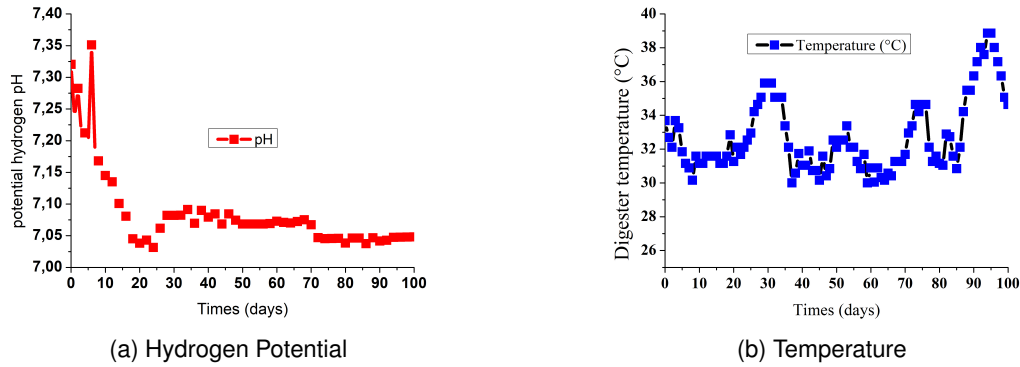**Fig. 3. Experimental evolution of biogas (Q) and methane flows as a function of time**

(a) Hydrogen Potential

(b) Temperature

Fig. 4. Evolution of hydrogen potential and temperature as a function of time
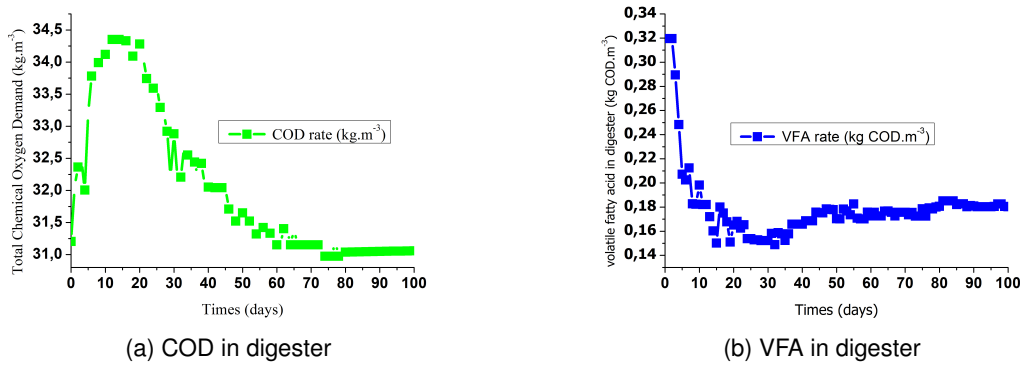

(a) COD in digester

(b) VFA in digester

Fig. 5. Evolution of chemical oxygen demand and volatile fatty acids as a function of time

3.3 Biogas and Methane Production in the Reactor

Fig. 3 shows the biogas and methane flow rates as a function of time. The production increases to reach $3\,m^3 d^{-1}$ on day 70 before stabilizing around $3.4\,m^3 d^{-1}$ between 70 and $100\,days$. From 0 to $5\,days$, the production remains low around $0.1\,m^3 d^{-1}$. This result is because the reactor was not yet stabilized in the absorption of the daily substrate overloads. As can be seen from the experimental production figure, the behavior is not the same when the load has been incremented by $D = 0.13\,d^{-1}$ the flow $Q = 0.19\,m^{-3}d^{-1}$, the $D = 0.19\,d^{-1}$ the flow $Q = 2.1\,m^{-3}d^{-1}$. The highest value of biogas production was obtained followed by a stable period phase. This stability could be due to a slowing down of the metabolic pathway of the bacteria (presented by Fig. 5b). At the very beginning of the test, the production of biogas remained low given the dilution rate still around $D = 0.095\,d^{-1}$ as in the literature [13].

For methane production (still in Fig. 3), the time between 0 and $10\,days$ methane production is around $1.4\,m^3 d^{-1}$. This drop can be explained by the plateauing of production kinetics during the first seven days after the daily substrate overloads. Indeed, during this overload, there was a notable accumulation of VFA due to a delay in the development of methanogenic bacteria. Also, during this period, the hydrogen potential

(pH) was period, the hydrogen potential (pH) rose very quickly from 7.5 to a value of 7, effectively showing the acidification of the culture medium. This accumulation of acid led to a decrease in methane production around $1.3\,m^3d^{-1}$ during the first few days. From 10 to 20 days, the production increases exponentially to reach $rate_{CH_4} = 1.9\,m^3d^{-1}$. After the 10th day, the production remains stable at around $1.95\,m^3d^{-1}$ per day for the rest of the time. This stability is due to the satisfactory rhythm taken by the microorganisms in the digester. The results obtained show that the increase in the load does not necessarily lead to the total net production of biogas and methane. Knowing the right recipe for biomass allows good optimization of anaerobic digestion.

3.4 Influence of pH and Temperature on Biogas Production

Fig. 4 shows the evolution of hydrogen potential and temperature within the bioreactor over time. The pH remained neutral during the 100 days of the study. During the first 10 days, we have a unit of $pH = 7.4$ and thereafter its value stabilizes at $pH = 7.015$. This is explained by the high reactivity of the methanogenic bacteria that consume the volatile fatty acids and avoids their strong fall. After 20 days, we noticed a self-adjustment of the pH due to the depletion of the acids formed in the first part by the acetogenic bacteria. This pH adjustment promotes the development of methanogenic bacteria which are responsible for the stable formation of methane (shown in Fig. 4a).

The temperature in the digester remained stable at around $35°C$ throughout the anaerobic digestion (Fig. 4b). This good result is due to the cement shell which allows to control the temperature in the reactor around $35°C$ and makes it hermetic to control the anaerobic condition. This stable temperature made it possible to maintain the proper functioning of the methanization micro-organisms which led to the transformation process. Throughout the process, the temperature remained above the digester target value $T_{digester} = 15°C$ [14].

3.5 Chemical Oxygen Demand and Acid Profile in the Reactor

The curves in Fig. 5 show the changes in chemical demand and total VFA concentration in the reactor over the $100\,days$ of the study. At the very

beginning of the study, the COD production remained high $34.4\,gl^{-1}$. This increase in concentration is explained biochemically because during this period the microorganisms are not sufficient to degrade the effluent (in Fig. 5a). Comparing the concentrations between the inlet and the outlet of the reactor we notice a great efficiency of the process to eliminate the organic matter, until stabilizing this demand. This is justified as subsequently, the value fell around $30\,kgm^{-3}$ despite the daily addition of the substrate. Already from the 30th day the development of bacteria allowed this stabilization.

Studies prove that a fluctuation in the concentration of volatile fatty acids in the digester admits a direct impact on the energy yield of anaerobic digestion [15]. Indeed, an imbalance between the acidogenesis and methanogenesis phases can be caused by an accumulation of acids (in Fig. 5b). This concentration increases weakly as a function of time and does not reach the critical value where VFAs inhibit anaerobic digestion which is around $1000\,mgCODl^{-1}$ according to the work of Kouas et *al.,* 2017 [16]. The concentration obtained through this study is between 0.14 and $0.32\,gCODl^{-1}$. The différent results show that there was no accumulation of VFA in the digester with an average value $rate_{VFA} = 0.18\,gCODl^{-1}$.

4 CONCLUSION

This study showed the importance of characterizing the physicochemical substrates factors within the bioreactor to improve their productivity in biogas. This study has also focused on the monitoring of the productions of the reaction medium in continuous mode of a pilot digester of $4\,m^3$. Thus, we observe that the average biogas flow rate (Q) is $2.5\,m^3d^{-1}$ and a methane flow rate of $1.9\,m^3d^{-1}$. Factors inhibiting biogas production are found to be $0.18\,kgCODm^{-3}$ for fatty acids and a hydrogen potential $pH = 7.015$ which remained below the literature threshold values [17]. One of the objectives we have achieved was to provide decision-makers or operators of anaerobic digestion facilities with reliable information for the quantitative evaluation of the energy potential of organic waste in Burkina Faso. However, the performance of the biodigester in depollution remained low around $30\,kgCODm^{-3}$ practically identical to the input values, but constitutes an important step in the sanitation of the environment because the bioreactor has made it possible to stabilize this chemical demand. Future work will consist of

the implementation of these different parameters of the experiment in a numerical model of anaerobic digestion.

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COMPETING INTERESTS

The Authors have declared that no competing interests exist.

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