Biogas production assessment for a semi-continuous anaerobic co-digestion pilot plant

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Abstract

Biogas production as a second-generation biofuel technology is a promising alternative for the transformations of residual biomass. Accordingly, this study evaluates the performance of different agricultural byproducts to identify the potential effect of different control variables (C/N ratio, organic loading rate and nitrogen rich substrate provider) for the optimization of the biogas production. The Box-Behnken design experiment was carried out in a pilot plant of four stirred stainless-steel digesters, under mesophilic semi-continuous digestion. A stable behaviour was achieved in 12 of the 13 experiments proposed and the highest value of daily biogas production was 2200.15mL d-1 obtained in combination 7, with a stabilization time of 14 days, an OLR of 4gVS feed daily, low C/N ratio and 1:1 relation of nitrogen providers. The concentrations of CH_4 remained stable after the production stabilization, an average composition of 60.6% CH_4, 40.1% CO_2 and 0.3% O_2. Consequently, the co-digestion process evaluated in this paper is a feasible option for the diversification of Colombian energy matrix and the development of the agro-industrial sector.

Introduction

Energy consumption is strongly related to economic growth, this relationship has given electricity markets an important place in the economy of developing countries [1]. In Colombia, the national energy demand has sustained increase in the last decades, also, is estimated that between 2016 and 2030 the electric energy demand will raise over 52% [2]. Although, one of the major challenges for the Colombian electric energy system are the Not Interconnected Areas (ZNI), which generates unsatisfied basic needs; due to the centralized generation of electrical power and the geographical distribution of generation resources, principally hydro power plants which cover the 67% of the energy generation. Consequently, one of the main priorities is the diversification of Colombian energy matrix and the inclusion of renewable energy alternatives, such as the transformation of biomass for energy production [3,4].

Biomass for energy production is a renewable alternative for the valorization of organic waste through biochemical conversion, such as anaerobic digestion of byproducts for biogas production [5]. Anaerobic digestion (AD) is a waste valorization technology that has been studied and used worldwide, due to its advantages over other organic waste treatments and bioenergy processes. The Anaerobic co-digestion (ACoD) allows to include different organic waste in order to avoid inhibitions and boost the production, a mixture of substrates reduce the organic load by the dilution of nitrogen, improves the biodegradation, dilutes the potential inhibitory or toxic compounds [6]. The search of optimum co-digestion mixtures, along with the evaluation of the process efficiency through operational conditions and reactors design will determine the potential of AD [7].

In ACoD process, the characterization of the different chemical composition of substrates is basic for the process definition and the prediction of the biogas production, the correct mixture of carbon and nitrogen rich byproducts improves process stability of nutrient contents required for the microorganisms [8]. Semi-continuous processes main variable is the organic loading rate (OLR) which is linked with an increase in methane production, this variable allows the control of total solids (TS) converted to biogas, however, overloading by rich carbon co-substrates could inhibit the process by the acidification of the system [7,9]. Lab-scale and pilot-scale experiments are useful as input the simulation of AD under different conditions [10].

In this framework, the implementation of agricultural by-products in anaerobic co-digestion processes for energy production is a potential that grows vigorously and rapidly [11], increase employment, support ecosystems and biodiversity, and reduce waste and pollution [12]. In addition, it brings many environmental benefits: the reduction of CH_4 emissions derivate of organic matter degradation and avoids concentration of atmospheric CO_2 from fossil fuels consumption by the diversifying the energy supply. In addition, related to substrate management, a hermetic storage of manure prevents the emission of N_2O; enhance the soil nitrogen absorption using the digestate as fertilizer and reduces ammonia emissions.

Consequently, the aim of this paper is the evaluation of the anaerobic co-digestion of four agro-industrial byproducts: pig manure (PM), sewage sludge (SS), residues from the bottled fruit drinks industry (RBFDI) and cocoa industry residue (CIR); to identify the potential effect of different control variables for the maximization of the biogas production in pilot-scale reactors. Therefore, the starting point was the definition of the constant operational conditions (pH, temperature and agitation) and generation of a Box Behnken design for the evaluation of the organic loading rate (OLR fed d^{-1}), carbon/nitrogen ratio adjustment and the nitrogen provider substrate as independent variables. The results presented in this study, are useful for the development of Colombian agro-industries and the diversification of the energy matrix. Specifically, these results enable the scale-up of this kind of processes and the implementation of distributed generation facilities where the substrates are available.

Materials and methods

Raw materials and Inoculum

The raw materials evaluated in this work are Pig Manure (PM), Residues from the Bottled Fruit Drinks Industry (RBFDI), Sewage Sludge (SS) and Cocoa Industry Residue (CIR); those residues correspond to rural/agricultural processes previously studied for [13]. Pig Manure was obtained from the Agricultural Research Center Marengo (C.A.M) of the Universidad Nacional de Colombia located in Mosquera – Cundinamarca, the animals are fed with commercial feeding formulas. Residues from the Bottled Fruit Drinks Industry (RBFDI) were simulated in laboratory based on the references related to the residual streams of this sector: mango, banana, blackberry, lulo, and passion fruit. Sewage Sludge (SS) was obtained from a water treatment plant in Madrid – Cundinamarca. Cocoa Industry Residue (CIR) was simulated from different references found of this industry, there were used the cocoa husks and pods, obtained from a private farm in Santander – Colombia [14]. A mechanical pre-treatment was applied to RBFI and CIR, which were reduced to a particle size of approximately 0.5 mm. The residues were preserved in a freezer at -4°C to avoid microbiological degradation before the assay.

Mesophilic inoculum (35±1°C) was used in all the test to guarantee proper start up conditions in each reactor. The inoculum was obtained from an anaerobic digester (water treatment plant of the dairy industry) of Alpina Company in Sopó - Cundinamarca, this inoculum is considered to have the adapted bacteria for the operating conditions arranged for the experiments. Table 1. presents the physical-chemical characteristics of the inoculum and each substrate; including moisture content, Kjeldahl total nitrogen (KTN), total solids (TS), volatile solids (VS), carbon/nitrogen ratio (C/N) and chemical oxygen demand (COD).

Table 1. Chemical characteristics of the inoculum and substrates.

		Substrates				
		CIR	**SS**	**PM**	**RBFDI**	**Inoculum**
pH (1:5 extract) [b]		5,42± 0,87	7,55± 0,85	7,16± 0,78	4,2± 0,80	--
Moisture[b]	%	89.62± 0.07	39.56± 0.03	74.65± 0.02	70.67± 0.03	91.45± 0.02
KTN[c]	%	0.70 ± 0.02	0.91 ±0.07	1.38 ± 0.08	1.16 ± 0.01	--
TS	%	10.37±0.02	60.43 ± 0.18	29.32 ±0.04	26.12±0.01	20.50±0.01
VS	%	7.94±0.01	8.49±0.04	22.92±0.07	22.73±0.07	16.72±0.08
C/N		59.57	14.88	33.18	48.01	--
COD[b]	g/L	8.17	13.14	24.6	8.62	--

[a] Average ± standard deviation, over three samples.
[b] Sample on wet basis.
[c] Sample on dry basis

A Box Behnken design was built based on the individual physicochemical characterization of the residues. The independent variables evaluated were C/N ratio, organic loading rate feed daily (OLR gSV fed d^{-1}) and the percentage of the source of nitrogen. Each variable manage three different levels: for C/N ratio (25, 35 and 45); for OLR gSV fed d^{-1} (2.5, 3.25 and 4); and for percentage of the source of nitrogen, samples containing only pig manure as nitrogen source (100%) or samples containing only sewage sludge as nitrogen source (0%) and those containing both substrates (1:1 or 50%), to generate a total of 13 combinations evaluated (Table 2). In this case, the C/N ratio is adjusted according to the characteristics of each waste and the gVS of the substrates and inoculum

(S/X) ratio was three (3). In fact, the different conditions in each reactor attend for the proper quantification of the response variable (biogas production).

Table 2. Experimental design description and composition.

Combination	C/N	%*	OLR (gVS fed d-1)
1	25	0	3.25
2	45	0	3.25
3	25	100	3.25
4	45	100	3.25
5	25	50	2.5
6	45	50	2.5
7	25	50	4
8	45	50	4
9	35	0	2.5
10	35	100	2.5
11	35	0	4
12	35	100	4
13	35	50	3.25

* Where 0% responds to the addition of only sewage sludge as nitrogen source, 100% only pig manure and 50% both substrates in equal quantities.

Analytical Methods

Total Solids and Volatile Solids of the initial substrates and the digestate were determined by drying the samples at 105±5°C in a drying oven and ignition at 550±10°C in a muffle furnace, this according to 2540B APHA - SM and D3174 of the America Society for Testing and Materials (ASTM) respectively. Measurements of pH were determined using a pH meter Edge model HI2002, following the standard test method D 4972-01 of the ASTM. Volatile fatty acids (VFA) and Alkalinity where measured according to (APHA, 2005). The Chemical Demand of Oxygen (COD) was measured using commercial vials with a range of 0 to 150 mg/L (HI 93752). Total Kjeldahl Nitrogen (NTK) according to the D1426 of the ASTM. The quantification of the volume of biogas produced was performed by RITTER flowmeters (MiligasCounter - RIGAMO software), which allows the total gas measurement in real time. Finally, the gas composition measurements (CO2, CH4 and O2%) was determined by the gas analyzer Biogas 5000 (Geotech).

Experimental set up

The anaerobic co-digestion processes were carried out in a pilot plant of four (4) identical semi-continuous stirred stainless-steel digesters that handle a workload of 4L (see Figure 1). Reactors are provided with three automatized systems to monitor each process: temperature system, agitation system and pH measurement system. For the temperature system, reactors are provided with a jacket through which the heating fluid will circulate and a PT-100 is used to record internal temperature sent to the embedded Arduino system. The agitation system is constituted by a servomotor, palettes with four blades each, and a driver provided for its control. The pH measurement system is controlled by HI 6100405 pH meters. Moreover, the reactors have a side orifice for the outlet of the digested effluent, and at the top for the biogas output to be conducted to the gas flow meters [15].

In order to guarantee the stability of the process, the following operative conditions were established: mesophilic temperature (35 ± 1 °C), an agitation speed of 30 RPM and a pH between 6.5-7.5 approximately, for a hydraulic retention time (HRT) between 17-21 days. During the process, samples were taken to monitor physicochemical parameters every 5 days (COD, AGVs, Alkalinity, gSV). At the same time, within the monitoring, gas sampling was carried out in order to evaluate the composition of the biogas (Biogas 5000 - Landtec) and record the flow of production of the biogas generated (milligascounter counters (RITTER) - RIGAMO software).

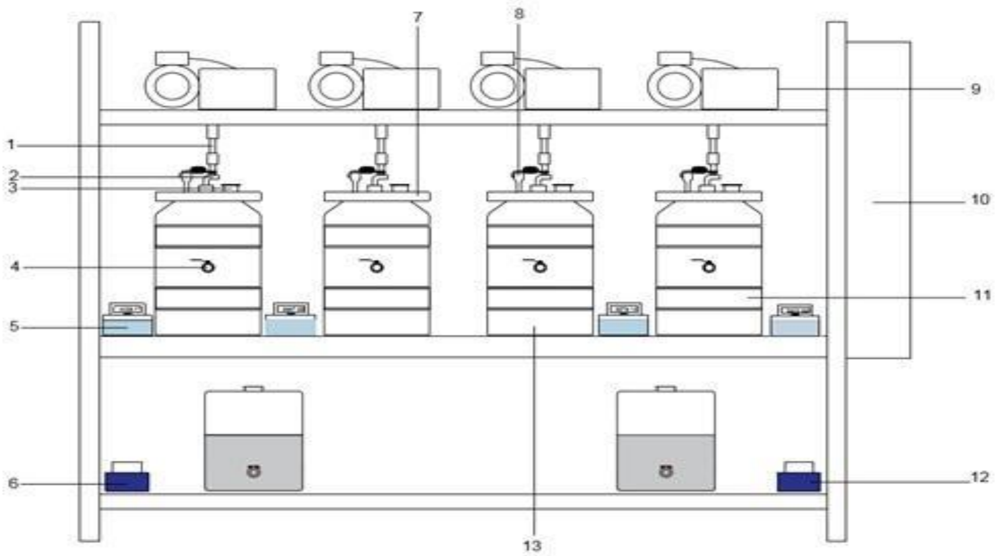

Figure 1. Semi-continuous anaerobic co-digestion pilot plant. (1. palettes, 2. upper valve, 3. pH meter, 4. lower valve, 5. milligas-counter, 6. acid supply pump, 7. top of the reactor, 8. sensor of temperature, 9. servomotor, 10. control panel, 11. temperature system, 12. base supply pump, 13. Reactor.)

Results and discussion

Box Behnken methane yields

The results of the methane production obtained for the anaerobic co-digestion tests in semi-continuous are presented in Figure 2. It is appreciable that the combination that achieved the highest cumulative biogas production is combination 2, which contains sewage sludge as the primary source of nitrogen, a C/N ratio of 45 and an OLR of 3.25 gVS fed d^{-1}. The reactor was fed for 21 days, during this time the pH remained between 6.6 - 6.85, average alkalinity of 615 mg $CaCO_3$/L and barely any accumulation of VFA's (an average value of 244 mg $CaCO_3$/L) at the end of co-digestion process. Under these conditions, the reactor manages to stabilize its production between day 15-16, with a total production of 744.31 mL CH_4/gVS and a biogas composition of 51.3 ± 0.31% CH_4, 44.8 ± 0.27% CO_2 and 0.2 ± 0.06% O_2. Although, combination 1 performed one of the lowest productions, 138.33 mL CH_4/gVS during 17 days of stabilization, where the values for OLR and the nitrogen provider were the same of combination 2, the C/N value was 25. There was a stable performance of the ACoD for the almost all the tests performed.

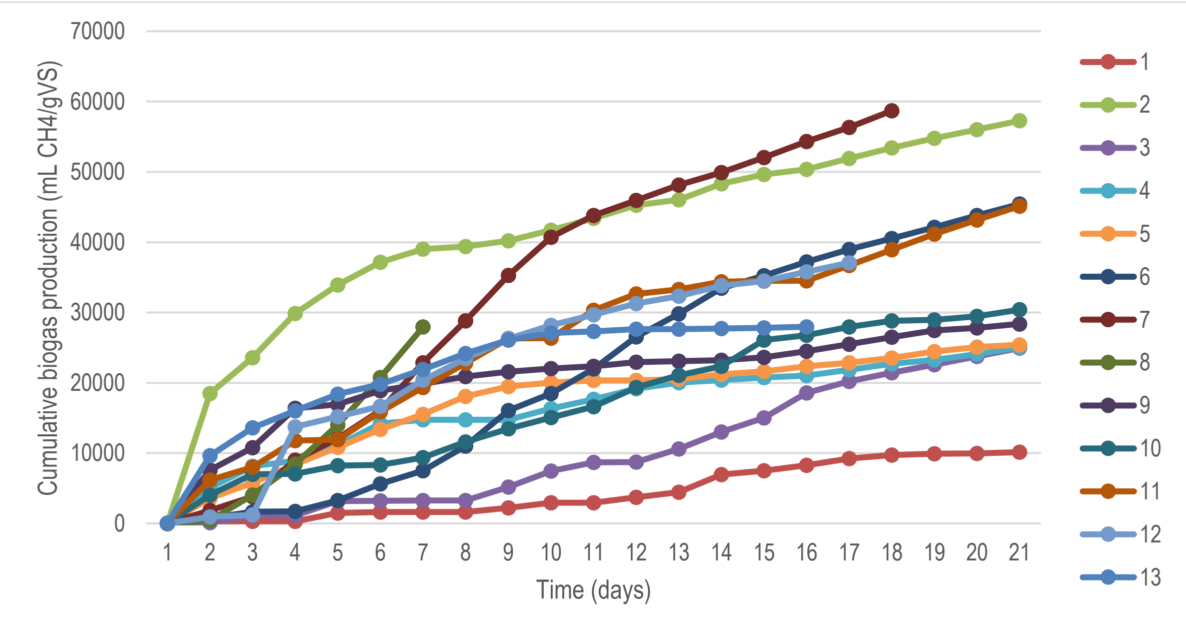

Figure 2. Cumulative biogas production of each combination evaluated.

Monitoring results

The results associated with the monitoring of the anaerobic co-digestion process of each combination, are presented in Table 3. A stable behaviour was achieved in 12 of the 13 experiments proposed, with productions between 400 – 2000 mL d^{-1} and low values of production of VFA's. The highest value of daily biogas production was 2200.15mL d^{-1} obtained in combination 7, with a stabilization time of 14 days. Then, it had the highest daily productions with one of the shortest stabilization time; followed by combination 11 (2110.35 mL d^{-1}) and 6 (17007.29 mL d^{-1}). Combination 11 presented one of the highest daily productions by day 17, under similar treatment conditions, high OLR feed daily, low C/N ratios (25 and 35 respectively); but different percentages for the nitrogen providers. Besides, combination 6, has contrary conditions, low OLR feed daily, high C/N values and a relation 1:1 between nitrogen providers. One of the main characteristics of combination 7 is the inherent buffer capacity given by the defined operational conditions, which favours the process stability and the development microbial community.

Additionally, the results of the digestate analysis showed removals of VS between 27 to 84%, the removals are higher than the ones reported in literature [16,17]. Although, the highest removal was achieved for the combinations with low OLR (2 gVS d^{-1}), with a 65.1% average, and the lowest removals were for high OLR (3.25-4 gVS d^{-1}), 57.3% average, as expected.

Table 3. Follow-up parameters of the co-digested assays in a semi-continuous regime.

	1	**2**	**3**	**4**	**5**	**6**	**7**	**9**	**10**	**11**	**12**	**13**
Stabilization day	17	16	17	17	14	16	14	15	16	17	11	9
Daily production (mL CH_4 d-1)	375.62	1400.58	1333.42	795.71	618.63	1707.29	2200.15	794.21	725.94	2110.35	1230,99	884,10
CH_4 (%)	46.9-52.7	50.7-51.3	57.5-59.9	52.4-54.5	54.2-55.8	57.9-59.5	54.7-57.5	58.9-59.1	56.4-59	54.3-55.4	49.8-50.3	49.8-50.1
Methane yield (mL CH_4/gSV)	138.33	744.31	313.73	322.50	411.17	736.70	617.98	463.02	531.00	446.49	363.52	396.59
Volatile solids (g/L)	32.16	22.50	55.66	10.71	14.43	23.63	17.40	23.82	11.64	14.92	14.92	62.84
COD (mg/L)	1750	15362.5	9825	13562,5	13725	3900	17800	1650	15362.5	1350	16550	14150

In turn, combinations 6, 8, 9 and 11 presented low alkalinity conditions (365 - 1013mg $CaCO_3$/L) and high production of VFA's (1526.4-2064 mg DQO/L) during the first day of the process, resulting in recurrent pH variations (4.94 ± 0.13), generating partial inhibition of methanogenesis during the start-up period or complete failure of the anaerobic co-digestion process [10]. These combinations were submitted to the addition of chemical agents, for the stabilization of the process, the pH values registered where 5.1 to 6.7 during the first 6 days of the process, and 7 to 7.4 from day 10 to the end of the experiments. The stabilization in ideal pH values (6.8 – 7.2) is essential for the anaerobic co-digestion process; pH values under 6.6 reduce the methanogensis [19]. For example, combination 8, after the addition of sodium hydroxide to counteract the pH drop reached an inhibited steady-state operation at a pH value of 5.4, the process was interrupted at day 7. Martínez et al.[20], report inhibitions in the co-digestion of sludge under mesophilic conditions, finding that an initial accumulation of VFA's produced during the acid phase can inhibit the activity of acetoclastic methanogenesis. Similarly, Chen, Cheng and Creamer [21], described that the interaction between fatty acids, volatile fatty acids, and pH can lead to the process to develop in a inhibited stable state, but with a low yield in the production of methane. This was reflected during the monitoring, where the productions were between 47.54 - 110.56 mL biogas/gSVd^{-1} with a biogas composition of 20-26.3% CH_4, 69.1% CO_2 and 1.2-0.2% O_2.

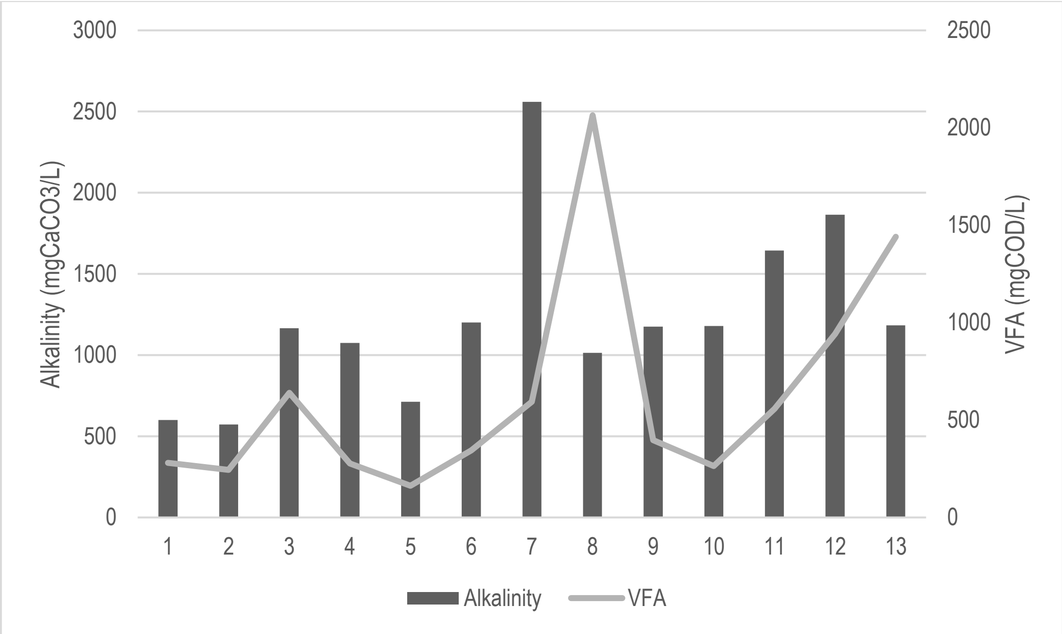

Figure 3. Alkalinity vs VFA comparison for the evaluated tests.

Moreover, Figure 3. shows the behavior of the process in terms of the alkalinity and VFA, which have been recognized as important operational parameters for the recognition of inhibition processes [22]. Indeed, combinations with high organic loads and high C/N values presented inhibitions of the microorganism consortia during the first days of the process, because of accumulation of VFA. In the same way, the combinations whose presented inhibition comportment have sewage sludge as only nitrogen provide or in 1:1 relation with pig manure. Sewage sludge has an initial concentration of VFA (792 mg COD/L), which might not be consumed then along with other conditions reduces the buffer capacity of the system and leads to the acidification by the accumulation of VFA. Although, intermediate and high loading rates are suitable in low C/N ratios, achieving stable methane yields, given the semi-continuous regime and the hydrodynamics that contributes in the evolution, mass transfer, structure and metabolism of microbial community [23].

Biogas composition

The biogas gas composition was evaluated in terms of percentage of CH_4, CO_2 and O_2 produced daily. Figure 4. Shows the biogas composition variation over the co-digestion time. The maximum concentration of methane (62.5%) was reached by day 14, as well as the stabilization in terms of biogas production. Also, can be seen that the methane production started since the first day of the process, CH_4 reached up to 50% at day 10 while CO_2 decreased. The concentrations of CH_4 remained stable after the production stabilization, an average composition of 60.6% CH_4, 40.1% CO_2 and 0.3% O_2. Previous studies for pilot scale systems have register similar compositions of biogas [24,25].

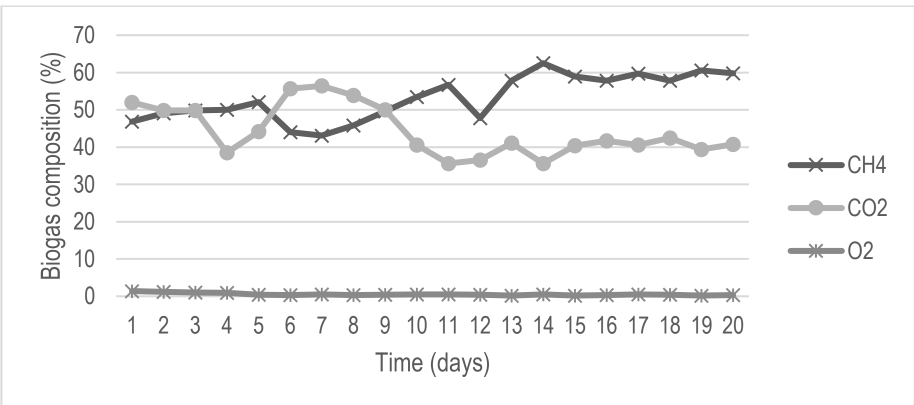

Figure 4. Biogas composition monitoring over co-digestion process of combination 7.

Conclusions

The present work demonstrated that the anaerobic co-digestion of pig manure (PM), sewage sludge (SS), residues from the bottled fruit drinks industry (RBFDI) and cocoa industry residue (CIR); is favored under low C/N ratios (values under 35); and high organic loading rates (4gVS), both nitrogen providers are suitable for the biogas production, although, high concentrations of sewage sludge may reduce the buffer capacity of the system. In general terms, C/N ratios above 35 together with high organic loads and only sewage sludge as a nitrogen source affects the normal development of the process, independently of the maximization of production. As shown, inhibitions can be managed through chemical agents during the initial days of the process to avoid inhibited stable states. Finally, the co-digestion process evaluated in this paper is a feasible option for the diversification of Colombian energy matrix and the development of the agro-industrial sector.

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