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Production of Biogas and Biofertilizers from Only Sugarcane Waste in a Pilot-Scale Two-Phase Anaerobic Digester System

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ABSTRACT
Sugarcane is cultivated in more than 100 countries around the world. Industrial agriculture and processing of sugarcane results in the generation of large amounts of wastes such as straw, bagasse, ashes, vinasse, pressed muds and other liquid and gaseous residues. Thus, the sustainable management of sugarcane waste is a critical issue for the agroindustrial field.The purpose of this work was to produce biogas using sugarcane waste as the only substrate. The experiments were performed in a pilot-scale two-phase anaerobic digester properly designed and built to provide the right technology for the substrate to be processed.The performance of the digester was measured by the volumes of biogas and biofertilizers produced and their production yield, as well as the degradable efficiency of the system. Biogas and biofertilizers obtained were characterized by physicochemical analysis.The production of biogas and biofertilizers from only sugarcane waste was successfully achieved. The system proved to be highly effective using only a mix of sugarcane straw and sugarcane pressed mud as substrate. The process showed a higher degradation efficiency compared to other studies where conventional substrates were used. Moreover, the design of the scale-pilot digester enabled a better control of operational parameters in both hydrolytic and methanogenic phases.
Keywords - biofertilizers, biogas, pilot-scale, sugarcane waste, two-phase anaerobic digester.

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I. INTRODUCTION

Sugarcane is cultivated in more than 100 countries around the world. Industrial agriculture and processing of sugarcane results in the generation of large amounts of wastes such as straw, bagasse, ashes, vinasse, pressed muds and other liquid and gaseous residues [2]. Thus, the sustainable management of sugarcane waste, with regard to economic and environmental factors, is a critical issue [3, 5].

In the last 25 years, a large number of companies in the industry have been developing strategies to mitigate the generation of organic waste through the application of biotechnological processes [3]. One of the most promising is the anaerobic digestion process, in which the excess of waste is reused as raw material for the production of biogas, which can be used as an energy source [5, 6, 9].

Biogas is normally produced by anaerobic digestion or fermentation of biodegradable materials from biomass, manure, sewage, municipal wastes, green wastes, plant materials to non-conventional crops [1, 5, 6, 7, 8, 9]. Anaerobic digestion is a complex treatment approach which occurs in four different phases: hydrolysis, acidogenesis, acetogenesis, and methanogenesis [7, 9]. The second and third phases are called as acid formation stage and the last phase is called as methane formation stage [5, 7]. As a result of anaerobic digestion, the organic material, used as substrate, is stabilized and gaseous by-products, primarily methane (CH4) and carbon oxide (CO2) are released [3, 7, 9]. The process is generally highly dependent on the nature of the organic material used as substrate and on process parameters like temperature, pH, C/N ratio, and hydraulic retention time (HRT), among others [4, 5, 8].

Sugarcane waste is a source of biomass suitable for biogas production [1, 2, 5]. Most of its components are rich in nitrogen content which makes them ideal for the biodigestion process [2, 3, 6]. Nevertheless, since sugarcane is plant biomass, it is mainly composed of lignocellulosic fibers such as cellulose, hemicellulose and lignin which are very difficult to digest in anaerobic conditions [4, 10]. The application of pretreatment methods enhances and improves the digestibility of this kind of feedstock as well as the methane production yield during the methanogenic stage [4, 10, 11].

Although a number of studies have addressed the use of sugarcane waste for the production of biogas through anaerobic digestion process, lignocellulosic waste faces trace element

deficiencies that influence the digestion performance decreasing the yield and quality of biogas produced [3, 5, 6]. Therefore, co-digestive processes are normally carried out using two or more substrates; lignocellulosic waste and additional animal manure, wastewater or bacterial inoculums [1, 2, 4, 5, 7].

The purpose of this work was to produce biogas using sugarcane waste as the only substrate. A pilot-scale two-phase anaerobic digester system was designed and built according to the nature of the substrate used. The anaerobic digestion took place coupled with a pre-treatment process in order to enhance the degradation of the complex substrate [10, 11, 12]. Moreover, the particular design of the pilot enables the recovery of the digestate which is a slurry rich in nutrients that can be used as liquid and solid biofertilizers [1, 2, 7].

The performance of the pilot-scale two-phase anaerobic digester system was measured by the volumes of biogas and biofertilizers produced and their production yield, as well as the degradable efficiency. Additionally, the biogas and biofertilizers obtained were characterized by physicochemical analysis.

II. MATERIALS AND METHODS

Substrate characteristics and pretreatment

In this study, a mix of sugarcane pressed mud y straw wastes (supplied by IPSA) generated from sugar-manufacturing processes was used as substrate for the production of biogas in a pilot-scale two-phase anaerobic digester. Both residues were characterized in terms of pH, conductivity (CD), carbon content (C), nitrogen content (N), C/N ratio, density and moisture content.

The mix of sugarcane waste used as substrate is hardly degradable due to its high content of lignocellulosic biomass [10]. The substrate was then pretreated, by composting, to improve its digestibility during the anaerobic digestion process [4]. The compost was prepared with 120 Kg of sugarcane pressed mud and 15 Kg of sugarcane straw piled up in a trapezoidal shape stack (60 cm x 80 cm x 100 cm) with intercalated layered of both residues. A total of 15 L of water were added to the stack. Composting was carried out under the optimum conditions described in Table 1. After a 10-day maturation period, turning was done manually to homogenize the content. A dry grinding machine was used to mill the compost 1-2 mm particle size powder to increase surface contact between substrate and microorganisms during biodigestion.

Table 1. Optimal conditions for composting pretreatment of sugarcane waste substrate.

Operational parameters	Start	End
pH	6.5-8.0	7.0-8.0
CD (μS/cm)	2.5-5.0	2.5-7.5
Moisture (%)	60-70	10.0-45
C/N ratio	15:1-25:1	10:1-20:1
Temperature (°C)	45-60	TA
Total Solids (%)	32.0-45.0	7.0-15.0

The temperature and the height of the compost pile were daily measured. In addition, physicochemical analysis of the final compost mix were carried out for the characterization of the substrate.

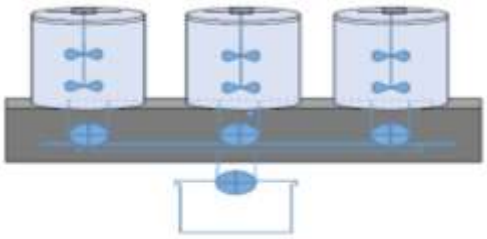
Experimental set-up

The experiments were performed in a pilot-scale two-phase anaerobic digester system designed and built properly to provide the right technology for the substrate to be processed.

A two-phase anaerobic digester system refers to the development of unique biomasses in separate reactors. The first phase is referred to as ‘‘acid fermentation’’ and involves the production of volatile fatty acids (VFAs), while the second phase is referred to as ‘‘methane fermentation’’ because in it the VFAs are converted to methane and carbon dioxide [13]. This kind of system is applied aiming a briefer global hydraulic retention time [14].

The two-phase digester employs two sequential reactors to separate the acid-forming phase (hydrolytic phase) from the methane-forming phase (methanogenic phase) [13]. The hydrolytic phase takes place in a set of 3 200 L capacity batch reactors hermetically sealed and a 1m3 container (Fig. 1a). The fermented effluent of each batch is delivered through a PVC output pipeline to a single point of discharge that feeds a 8 m3 horizontal plug-flow reactor where the methanogenic phase takes place (Fig. 1c). The horizontal plug-flow reactor has a trapezoidal base with a storage capacity of 6.5 m3 for liquid, and a geomembrane cover with a storage capacity of 12.4 m3 for biogas (Fig. 1b). Besides the reactors, the system also includes: a recirculation pump system, a heating system, a differential pressure manometer to measure the volume of biogas produced, boxes for the recovery of solid and liquid effluents, a biogas flare and a pressure relief valve.

Unlike single-stage processes, in a two-phase process the phase division in two separate reactors enables the reduction of hydraulic retention time and the increase of organic loading rate, enhancing the production yield of biogas and biofertilizers [8,14].



A

B

C

Figure 1. (a) 200 L batch reactors, (b) 8 m^3 horizontal plug-flow reactor, (c) pilot-scale two-phase anaerobic digester system.

System operation

The hydrolysis/acidification batch reactors were fed with 1400 L of diluted substrate. The substrate was homogenized with a mechanical stirrer incorporated to the lid of each reactor. The hydrolytic phase took place under optimal conditions described in Table 2. Due to the low density of organic matter (0.25 g/L), particles gathered on the surface forming a scum film, so additional stirring was needed twice a day.

The aim of this phase was to hydrolyze the organic matter through the enzymatic activity of microorganisms carried within the substrate, releasing simple structure carbon compounds (organic acids), which are better assimilated in the methanogenic phase [2,8,14].

Table 2. Optimal conditions for the hydrolytic phase.

Operational parameters	Start	End
PH	6-7	5.5-6.5
CD (µS/cm)	7	7
DO (%)	40-55	40-55
TS (%)	6-7	5-6
VS (%)	5-6	3-5
FOS/TAC ratio	1-2	1.5-2.5

After 5 days of fermentation process, the resulting slurry was used to feed the anaerobic reactor with from 200 L to 500 L of slurry per day. A recirculation system was turned on during the unloading of the batch reactors so as to homogenize the substrate as well as to prevent clogging of the outlet pipe and density variations at the digester´s inlet and outlet pipes. Recirculation of the slurry also enhanced the release of gases trapped in the surface of the liquid phase. A sample of slurry was analyzed in terms of: pH, conductivity (CD), dissolved oxygen (DO), FOS /TAC, total solids (TS), and volatile solids (VS).

The methanogenic phase took place under optimal conditions described in Table 3. Maximum and minimum hydraulic retention times were calculated, being the minimum 12 days and the maximum 30 days. The pressure and temperature were constantly monitored during the anaerobic digestion using a temperature display and a differential manometer connected to the cover of the anaerobic digester. A sample of the recovered effluent was analyzed in terms of: pH, conductivity (CD), dissolved oxygen (DO), FOS /TAC, total solids (TS), and volatile solids (VS).

Table 3. Optimal conditions for the methanogenic phase.

Operational parameters	Start	End
PH	5.5-5.5	6-7
CD (µS/cm)	7	3-5
DO (%)	40-55	50-60
TS (%)	5-6	1.5-4.5
VS (%)	3-5	1-0
FOS/TAC ratio	1.5-2.5	0.06 - 1.5

The biogas produced by the pilot-scale two-phase digester system was collected in 1 m3 PET bags. Furthermore, the solid and liquid effluents were recovered at the output pipe of the digester and collected in boxes (74 cm x 72 cm); left to stabilize for later storage in containers.

Analysis of produced biogas, liquid and solid biofertilizers

The volume of produced biogas was measured using a floating dome gasometer at constant pressure. Since the gasometer is connected to the digester, the measurements taken were dependent on the physical conditions inside the digester.

The biogas composition was analyzed using a gas detector PGD3-IR. The detected compounds were methane (CH4), carbon dioxide (CO2), oxygen (O2) and hydrogen sulfide (H2S).

Additionally, the volumes of the recovered solid and liquid effluents were measured and their physicochemical compositions were analyzed.

III. RESULTS AND DISCUSSION

Characterization of sugarcane waste and elaboration of the substrate

The result of the physicochemical analysis of sugarcane pressed mud and straw is shown in Table 4.

Table 4. Physicochemical analysis of sugarcane pressed mud and sugarcane straw.

Parameters	Sugarcane pressed mud	Sugarcane straw
PH	5.39	7.51
CD (µS/cm)	1.34	3.39
C (%)	29.37	46.89
N (%)	1.34	0.54
C/N ratio	21.92	86.83
Density (g/cc)	0.26	0.08
Moisture (%)	12	-

The results show that sugarcane pressed mud and straw have a high carbon content. Additionally the C/N ratio of both components is ideal for high biogas yield [15]. These latter were used to calculate and elaborate the formulation of the substrate for the compost treatment Table 5.

Assessment of hydrolytic phase

The hydrolytic phase was assessed according to the physicochemical characterization of the slurry obtained after fermentation (Table 6). It was important to establish optimal operation ranges for this phase because the performance of the methanogenic phase depends heavily on the characteristics of fermentation products [13]. The following physicochemical parameters were considered: pH, Total Solids (TS), Volatile Solids (VS) and FOS/TAC ratio.

Table 5. Substrate composition for compost pretreatment.

Parameters	Sugarcane straw	Sugarcane pressed mud	Water	Sum	Substrate		
					Volume (ml)	TS (%)	C/N ratio
C (%)	46.8	29.37	0		800	6.9	25.67
N (%)	0.54	1.34	0				
TS (%)	89.35	32.12	0				
C/N ratio	86.83	21.91					
Weight (g/100g) =P	15	120	585				
C*P	703.35	3524.4		4227.75			
N*P	8.1	156		164.1			
TS*P	1314.5	3854.4		5168.9			

Table 6. Physicochemical parameters of the fermented slurry.

Parameters	PH		TS (%)		VS (%)		FOS/TAC	
	Min	Max	min	max	min	Max	Min	Max
Feed load	5.5	6.5	5.0	7.0	3.0	5.0	1.5	2.5

The hydraulic retention time was shorter for the hydrolytic phase: 5 days, compared to the hydraulic retention time of the methanogenic phase: 12 -30 days. Differences in HRT between phases is due to bacterial growth rates. Acidogenic bacteria have higher growth rates than methanogenic bacteria [13,14].

Biogas production and characterization

Biogas production and production yields were calculated from data registered for a period of 6 months: from January to June of 2017. Table 7 shows the minimum and maximum volumes of biogas obtained per month and the production yields per m3 per Kg per day.

Higher biogas production volumes and yields were achieved during the months of January and February. The production volumes reached up to about 5750 - 5800 L per month and the yields were close to 0.017 m3/Kg/Day. April was the month were the lowest production rates and yields were

recorded. In average, the minimum production volume of biogas per month was around 4550 L and the minimum production yield higher than about 0.012 m3/Kg/Day.

Table 7. Monthly biogas production and production yields.

Month	production (L)		Yield (m^3/Kg/Day)
	Min	Max	
January	4565.87	5817.54	0.016518
February	4553.58	5754.09	0.016795
March	4544.74	5501.84	0.015706
April	4545.67	5060.82	0.012311
May	4554.58	5504.77	0.014867
June	4565.91	5437.84	0.012460

Since January and February are the hottest months of the year there is a correlation between the temperature of the environment where the anaerobic process takes place and the efficiency of biogas production. The latter can be increase by the following factors: forced convection, or direct exposure to solar heat. In the other hand from March to June the ambient temperatures are lower triggering the decrease of the methanogenic capacity of microorganisms. Thus, the results show the production and yield of biogas were dependent on climate conditions.

The results of the chromatographic analysis of samples of the biogas produced are shown in Table 8. Six samples collected one by month, from January to June were analyzed. All samples present highly similar composition. The major component was methane (CH4) with an average content of 55.4%, followed by carbon dioxide (CO2) with an average of 43.6% and in a much smaller percentage oxygen (O2) with an average of 0.53%. The content of hydrosulfuric acid (H2S) was of about 18.2 ppm.

Table 8. Chemical characterization of biogas.

Biogas content			
CH_4 (%)	CO_2 (%)	O_2 (%)	H_2S (ppm)
55.8	43	0.5	18
55.7	44	0.5	21
55.1	45	0.6	21
55.7	43	0.6	14
55	44	0.5	21
55.2	43	0.5	14

The average yield of CH4 in anaerobic biodigestion processes is of 55 - 70% [2]. Since the produced biogas had an average yield of CH4 of 55.5%, the results show that biogas with desirable content of CH4 can be produced using only sugarcane waste as a substrate. The use of a two-phase digester system with a previous compost pretreatment contributed to obtaining biogas with high methane content.

Production and characterization of solid and liquid biofertilizers

Solid and liquid effluents were recovered at the output pipe of the digester and collected in boxes. These effluents contain a large proportion of nutrients and after stabilization and drying (in the case of the solid effluent), they can be used as solid and liquid biofertilizers [16].

The production and yield of the solid and liquid biofertilizers fluctuates according to climate conditions due to the fact that the metabolism of the microbial consortiums is directly influenced by temperature variations. When temperature rises, the production of biogas increases and the production of liquid fertilizer decreases. This behavior was observed after analyzing the data obtained by daily measurements of solid and liquid biofertilizers production and yield (Tables: 9 and 10).

Table 9. Daily production of solid and liquid biofertilizers.

Product	Volume (L)	
Liquid biofertilizer	Min	120
	Max	160
Solid biofertilizer	Min	15
	Max	20

Table 10. Monthly production of solid and liquid biofertilizers and production yields.

Month	Solid biofertilizer			Liquid biofertilizer		
	production (L)		Yield (m^3/Kg/Day)	production (L)		Yield (m^3/Kg/Day)
	min	Max		min	Max	
January	-	-	-	120	160	0.4508385
February	15.00	20.00	0.0512821	120	160	0.4123077
March	17.78	20.00	0.0570265	120	160	0.4233846
April	17.14	20.00	0.0603253	120	160	0.4455385
May	16.89	20.00	0.0597056	120	160	0.4284900
June	20.00	21.00	0.0623077	120	160	0.4320513

Physicochemical characterization of both liquid and solid biofertilizers samples were carried out based on standard methods (Table 11).

Table 11. Physicochemical characterization of solid and liquid biofertilizers.

Parameters	Solid biofertilizer	Liquid biofertilizer
pH	6.93	6.77
EC (dS/m)	4.29	2.83
TS (g/L)	44.47	108.91
DOM (g/L)	21.54	51.36
Total N (mg/L)	1960	3843.00
Total P (mg/L)	133.26	204.39

Total K (mg/L)	404.50	508.75
Total Ca (mg/L)	2550.00	4475.00
Total Mg (mg/L)	459.50	812.50
Total Na (mg/L)	252.50	217.50
TOC (g/L)	12.82	27.38
Humic acid (mg/L)	0.16	0.46
Fulvic acid (mg/L)	0.18	0.32
Humin	1.87	3.94
Total Pb (mg/L)	1.87	4.11
Total Cd (mg/L)	0.12	0.16
Total Cr (mg/L)	2.00	4.06
Density g/cc	1.01	1.05
Total Zn (mg/L)	7.78	16.08
Total B (mg/L)	1.46	1.95
Nitrogen-ammonia (mg/L)	609.00	658.00

Fertilizers are organic or mineral compounds that are supplied to the soil or irrigation water in order to provide the plant with nutrients [16]. These should contain at least 5% of the three main nutrients: nitrogen, phosphorus and potassium [17]. The results show both products present a high content of inorganic minerals, especially nitrogen (N), phosphorus (P) and potassium (K), which are essential plant nutrients. Furthermore, both products have a high concentration of organic matter that is an important property in organic fertilizers as a source of nutrients for soil microflora.

The bioconversion performance of the pilot-scale two-phase anaerobic digester was deduced from the degradation efficiency percentage. The latter is determined by the difference between the Volatile Solids (%) of Total Solid (%) of the substrate, previous to the process (pretreatment and anaerobic digestion), and the Volatile Solids (%) of Total Solid (%) of the liquid effluent recovered after the process (Table 12).

Table 12. Degradation efficiency for the pilot-scale two-phase anaerobic digester.

Parameters	Substrate mix: Sugarcane straw + Sugarcane pressed mud 1:4 ratio (10 g)	Liquid effluent (100 ml)
TS (%)	40.20	2.00
VS out of TS (%)	21.55	0.95
Degradation Efficiency (%)	-	90

Table 12 shows the total solid content of the substrate and the liquid effluent, the volatile solid content of the total solid content of the substrate and the effluent, and the degradation efficiency of the pilot-scale two-phase anaerobic digester system. The bioconversion performance of the digester can be considered as excellent due to the very high percentage of degradation efficiency obtained: 90%. Other studies using other kinds of substrates have reported lower degradation efficiency (Table 13).

Table 13. Degradation efficiency of previous studies.

Authors	Substrate	Degradation Efficiency (%)
Steffen et al, 1998 [18]	Cow manure	60
	Pig manure	74
	Fruit waste	50
	Chicken manure	60
AINIA & GIRO, 2011 [19]	Urban solid waste	39
	Corn waste	51

Degradation efficiencies percentages from previous studies are in average in between 39% to 74%. The lowest reported degradation efficiency is from a digestive process where urban solid waste was used as substrate. The highest reported degradation efficiency appears in a digestive process where pig manure is used as substrate. Degradation efficiencies are higher in processes that use animal manure as substrate, 60-74%. When plant biomass is used as substrate the degradation efficiencies are lower, around 50%. Nonetheless, the degradation efficiency reached during the two-phase anaerobic digestion process is superior to the degradation efficiency of animal manure substrates and almost twice the degradation efficiency of plant biomass substrate. Thus, this results demonstrate that the two-phase digestive system improves the degradability of substrates and so does the additional composting pretreatment step.

IV. CONCLUSION

The production of biogas and biofertilizers from only sugarcane waste in a pilot-scale two-phase anaerobic digester system was successfully achieved. The system proved to be highly effective for the production of biogas and biofertilizers using only a mix of sugarcane straw and sugarcane pressed mud as substrate for the anaerobic digestion process. The process showed a very high degradation efficiency compared to other studies where conventional substrates were used. Moreover, the design of the scale-pilot digester enabled a better control of operational parameters in both hydrolytic and methanogenic phases.

In all, the two-phase anaerobic digester is a suitable alternative for the treatment of solid waste generated by the sugarcane industry. The separation of the hydrolytic and methanogenic phases in two different reactors results in the decrease the hydraulic retention times and increased biogas production yields without the need of additional co-substrates like animal manure, wastewater or bacterial inoculums.

The implementation of this type of digester system represents an alternative for sustainable management of solid sugarcane waste while producing desirable methane content biogas and enhanced biofertilizers easily assimilated by plants.

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"Validación de la obtención de biogás, biol y biosol a partir de solo residuos de la industria azucarera (cachaza, residuos de la cosecha de caña de azúcar y bagazo), mediante un biodigestor con doble fase, hidrolítica y metanogénica"

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