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Enhancement of Biogas Production from Sawdust through Sustainable Alkaline and Biological Pretreatment Methods

[1]Olanipekun, A. A. and [2*]Oladejo, O. S.

[1]Department of Civil and Environmental Engineering, College of Engineering, Bells University of Technology, Ota, Ogun State, Nigeria.
[2]Department of Civil Engineering, Faculty of Engineering and Technology, Ladoke Akintola University of Technology, Ogbomoso, Oyo State, Nigeria.

Corresponding Author: osoladejo@lautech.edu.ng, +234-803-577-0398

Abstract
This study aims to enhance biogas production from sawdust fibers Quercus genus (oak) using sustainable pretreatment methods. Sawdust and poultry dung (substrates) were obtained in Ogbomoso and delignification of oak samples was carried out through gravimetric method on 5, 10, and 15 days before and after digestion. Physico-chemical parameters were analyzed according to American Public Health Association (APHA) (2018) standard. Anaerobic digestion on substrates was performed through alkaline and biological pretreatment processes, biogas samples were collected and analyzed using gas chromatograph mass spectrometer. Pretreatment results for delignification of raw sawdust, alkaline and biological pretreated at 5, 10 and 15 days were; 13.5, 2.0, 1.5, 1.0 and 1.0 in mg/l. Physico-chemical results of substrates before and after digestion respectively were; pH (7.38, 8.12); alkalinity (350, 475); Nitrogen (25.5, 38.2); Phosphorus (2.67, 2.43); Carbon (192.8, 342.1); Potassium (4.1, 3.5); Phosphate (56.0, 128); Sulphate (74.0; 69.0), Calcium (12, 44); Magnesium (65, 36); Manganese (0.010, 0.031), Iron (5.2, 6.1); Zinc (25.0, 17.4); Aluminium (0.35, 0.34); Copper (2.5, 2.5); Biochemical Oxygen Demand (110, 175); Chemical Oxygen Demand (964, 1440); Carbon/Nitrogen ratio (8:1, 9:1); Volatile Solids (1216, 4560); Moisture Content (25.8%, 63.1); Total Solids (66.2%, 17.8%) and Volume of Solids (58.2%, 77.7%) in mg/l. Biogas samples composition from biological and alkaline pretreatments were: Methane (65.05%, 62.27%); and Carbon Dioxide (17.51%, 13.28%). Both pretreatment methods produced enough methane percentages, but biological pretreatment performed better. This work is applicable in sustainable biogas production for environmental protection.

Keywords: Biogas, Cattle rumen, Enhancement, Substrates, Sustainable pretreatment

Introduction
Globally, nations rely on sustainable energy because it is the major driver of the development and growth required particularly in developing and underdeveloped nations (Subramani, 2012). This is more expedient especially when the provision of clean and environmentally friendly energy has become a global issue (Dahunsi *et al.,* 2018). However, the major advances recorded in the areas of energy production have also been accompanied by associated increases in toxic emissions, while their consequences for change in climate have equally become an issue of serious concern (Salihu and Alam, 2015). Therefore, ensuring that clean and sustainable energy are provided both in developing and underdeveloped nations is one of the ways by which this problem challenging humanity can be solved (Dahunsi *et al.*, 2019).

Furthermore, Andriani *et al.,* (2014) and the World Health Organization (2014) concluded that researchers must continually explore and develop new ways of producing clean and sustainable energy for man's use. This is more imperative because the continued exploitation of fossil fuels have been mentioned as the primary causative agent of climate change and the consequences (high CO_2 levels, high temperatures, wildfires, global warming, irregular rainfall and water scarcity) of that are too deleterious for the environment (European Commission, 2022). Also, Dodds and Demoulin (2013) reported that if the planet must survive, then researchers must continually develop better ways of powering automobiles, factories and even cooking stoves. This explains why scientists and researchers turned to the production and use of biogas as an alternative and environmentally friendly source of energy (Gaur and Suthar, 2017).

According to Oladejo *et al.,* (2020), biogas which is methane rich gas, can be produced from the breakdown of organic materials and anaerobic digestion. Similarly, Latha *et al.,* (2019) reported that improvised anaerobic digesters can often be used to transform energy that is stored in biological wastes like human excreta, agricultural, food and animal wastes into biogas. Thus, the eventual biogas is used as electricity, lighting and cooking fuel, but cooking is often the most economical and common use. According to Zumalla *et al.,* (2018), pretreatment methods are carried out to achieve the delignification of wood samples, especially wood samples which will be used for digestion as part of the processes required for enhanced biogas production.

Furthermore, Monlau *et al.,* (2012) mentioned that there are principally two major types of pretreatment processes for woods and sawdust, these are the physical methods and the chemical methods often used by researchers to achieve the process of delignification in biomasses. This assertion was further researched and buttressed by (Saha *et al.,* 2016) and (Naran *et al.,* 2016) in similar studies. Therefore, this study carried out the enhancement of biogas production from sawdust fibers using sustainable pretreatment methods. This was carried out in order to solve the problem of indiscriminate disposal of sawdust in sawmills and poultry dung by poultry farmers, through open burning and dumping in the environment (Ajayeoba *et al.,* 2021).

Consequently, this indiscriminate culture of disposing sawdust and poultry dung both contribute to the generation of greenhouse gases and environmental pollution (Dahunsi *et al.,* 2017) and Food and Agriculture Organization (2019). To this end, the objectives of this study included the pretreatment processes (alkaline and biological) of the sawdust sample, the physico-chemical analyses carried out on the sawdust and the laboratory analyses of the biogas produced, all aimed at achieving the enhancement of biogas production.

Materials and Methods

The equipment used for this study were milling machine, laboratory sterilizer, weighing scale, Erlenmeyer flasks, desiccator, ultraviolet spectrophotometer, conical flasks, atomic absorption spectrophotometer (AAS) and gas chromatograph mass spectrometer.
The materials used for this study were; sawdust, poultry dung, cattle rumen (inoculum), sodium hydroxide (NaOH), laboratory filter and improvised gas bags. The poultry dung sample and the cattle rumen sample used for this work were obtained at a poultry farm in YOACO area, Ogbomoso.

The sawdust used for this work was also obtained at Anuoluwapo sawmill very close to Ogbomoso-Ilorin express road, with Global Positioning System Coordinates (GPS) 8.299, 4.34496, Ogbomoso, Oyo State, Nigeria. Furthermore, the poultry dung was stored in a polythene bag and properly tied at the top to ensure it does not lose any of its vital ingredients. In the same way, the sawdust obtained were stored in a sack and tied with a rope at the top before both substrates were transported immediately to the Water and Environmental Engineering Laboratory at Landmark University.

Alkaline and biological pretreatment processes

For the alkaline pretreatment, the substrates samples were thoroughly grinded and then sieved accordingly with the use of a 20-fraction sieving mesh of 841 µm that was also retained on a 40 mesh screen. Thereafter, the alkali pretreatment process was carried out on the samples according to Zumalla *et al.*, (2018) by using 10 g samples of the sieved substrates samples thoroughly mixed with a 100 mL of NaOH of the American Chemical Society (ACS) grade and placed in Erlenmeyer flasks of 250 mL. The mixtures were subjected to heat at a temperature of 100^0C in a laboratory oven for eighty minutes. Furthermore, the mixtures were allowed to cool down under room temperature and were later filtered out through the laboratory filter. Eventually, the solid components of the mixtures were squeezed out and subjected to drying.

For the biological pretreatment process, 500 g of oven dried substrates samples that have been thoroughly sterilized were measured and transferred into Erlenmeyer flasks of 250 mL (Zumalla *et al.*, 2018). These were thereafter inoculated with 250 mL of cattle rumen which corresponded to 12 mg of dry weight of the substrate's samples. The flasks were then closed with the use of laboratory cotton stoppers and were also incubated 32^0C for a minimum of 10 days. Consequently, the acid detergent lignin (ADL), the neutral detergent fiber (NDF) and the acid detergent fiber (ADF) values were all determined through the gravimetric method as suggested by the APHA (2018). Furthermore, the NDF, ADF and ADL values were then obtained for the fifth day, the tenth day and the fifteenth day for both the alkaline and biological pretreatment processes respectively.

Physico-chemical analyses of the substrates before and after digestion

To analyze for the physico-chemical parameters of the substrates before and after digestion, the following characteristics of the substrates were analyzed; pH, total alkalinity, total nitrogen, total phosphorus, total carbon, potassium, phosphate, calcium, magnesium, manganese, iron, zinc, aluminium, copper, BOD, COD, carbon/nitrogen ratio, total solids and volume of solids, using the AAS as shown in Figure 1 according to the APHA (2018) standard. The physico-chemical parameters were all carried out in triplicates.

Laboratory digestion of the substrates, collection and analysis of biogas produced

The substrates used for this research were chosen because they are materials that are high in organic content, solids content, pH and carbon/nitrogen ratio which are all highly essential for biogas production (Oladejo *et al.,* 2021). The two separate substrates categories selected were regularly agitated in order to ensure that there was an even mixing of the materials.

Figure 1: Atomic absorption spectrophotometer used for analysis

This also ensured that the production of bacteria needed for the biological processes that took place in the conical flasks were enhanced. Subsequently, the mixing adopted for this work was in two categories, the poultry dung and the sawdust. Suffice to mention here that the retention time used in this analysis was 32days.

The substrates samples were measured and placed in conical flasks which served as the small-scale digesters or fermentation vessels for this experiment. The samples were filled adequately into the flasks and were eventually placed into the laboratory water bath in order to ensure that the desired digestion temperature was maintained for the anaerobic digestion process. Furthermore, the flasks were connected accordingly to the collecting system of the biogas through improvised taps with glass bores as shown in Figure 2. The substrates combination was monitored regularly in order to ensure that the operating conditions in the conical flasks necessary for the optimal production of the microbes and eventually the production of biogas were adequately maintained.

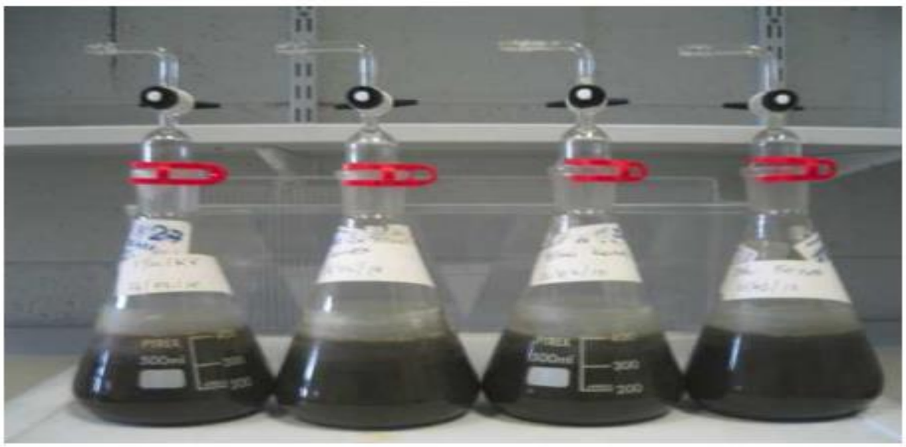

Figure 2: Conical flasks for digestion of substrates

Also, there were red coloured clips attached to the setup in order to make sure that there was no leakage of biogas via the gaps present in the flasks. Again, the volumetric flask as shown in Figure 3 was used for the measurement of the biogas produced in the setup. Water was filled into the volumetric flask and was used to mark the right water level for the purpose of comparison. Thereafter, a 250 mL inverted cylinder was placed inside the cylinder and whenever biogas was pumped into the cylinder the water level naturally rose. As such, the biogas level in the setup was determined through the marking of the water level and the subtraction of the prior chosen water level.

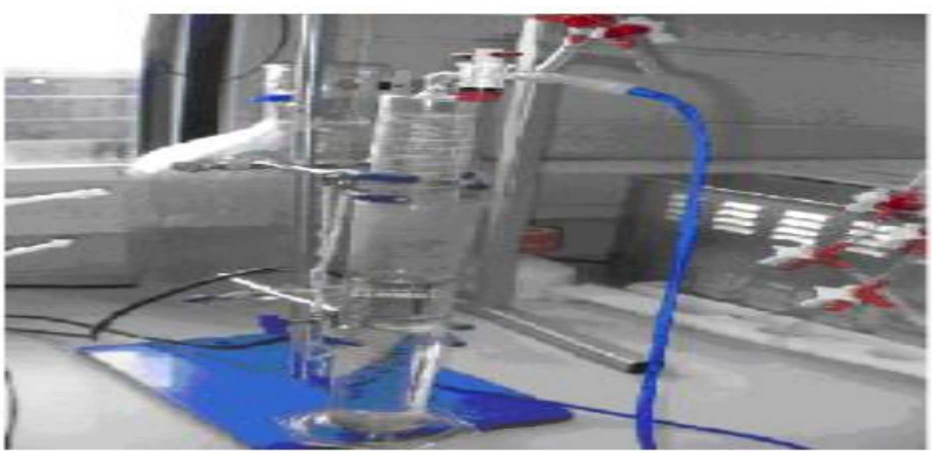

Figure 3: Volumetric flask for measurement of biogas produced

Furthermore, a valve containing three outlets as shown in Figure 4 was then connected to an improvised aluminium bag to help in the channeling of the movement of the biogas produced. Also, by just turning the tap, the movement of the biogas could be controlled and directed, and it was also useful for stopping the movement of the biogas produced. Since the retention time adopted for this study was 32 days, the process of biogas collection also started after the 32 days of retention time were completed.

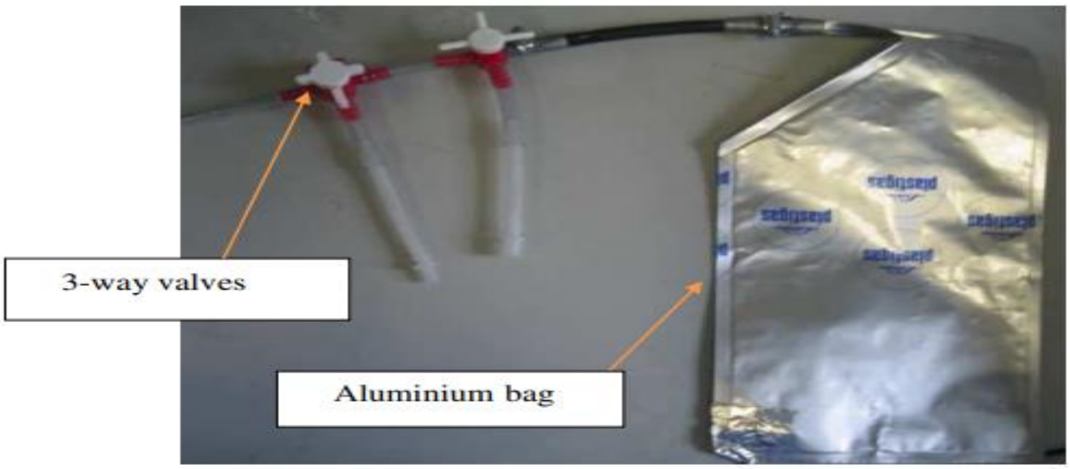

Figure 4: Three-outlet valves connected to improvised gas bag

Also, the outlets of the valve also provided opportunity for the biogas produced to be stored safely in the improvised aluminium bag. By storing and sealing the gas safely in the improvised gas bag, this also helped to prevent the entry or diffusion of atmospheric air through the bag, thereby ensuring that the biogas sample that was subjected to laboratory analyses remain untainted or tampered with.

The laboratory analyses of the biogas produced and collected from the digestion setup was also carried out in the Water and Environmental Engineering Laboratory, Landmark University, Omu Aran, Kwara State, Nigeria through the use of gas chromatography. The gas chromatography device is fitted with an injection port and the representative samples were injected into that port. Thereafter, evaporation and separation of the component parts was done and finally, the equipment was used to identify the component parts present in the corresponding sample. A particular spectral pick was produced for each component and this was recorded electronically on a paper chart. This analysis was carried out using a Varian 3800/4000 gas chromatograph mass spectrometer equipped with an Agilent and a capillary column DB5ms (30.0m x 0.25mm, 0.25µm film thickness).

Results and Discussion

Pretreatment and delignification of sawdust fiber samples

The results of the analysis of the parameters of the sawdust fiber samples are presented in Table 1 which covers the values of the percentage reduction of the ADL, the NDF, the ADF, hemicellulose and the cellulose of the sawdust fiber samples. The results showed that after the pretreatment processes, the ADL values fell significantly from the raw 13.5mg value to 1mg value obtained after the 10^{th} and 15^{th} day of analysis. These values indicate that both the pretreatment processes reduced the lignin concentrations of the sawdust samples; however, the biological pretreatment process returned a better result. The results obtained in Table 1 are very similar to those of Tables 2a and 2b because the values of the digestate samples all fell to 1.00mg for ADL.

Table 1: Fiber fraction lignin determination

S/N	Samples	NDF (mg)	ADF (mg)	Hemicellulose (mg)	Cellulose (mg)	ADL Lignin (mg)
1	Raw Sawdust	74.00	67	7.00	19.5	13.5
2	Alkaline Pre-treated	13.00	67	7.00	3.5	2
3	Biological Pre-treated Day 5	13	10	4.00	5	1.5
4	Biological Pre-treated Day 10	9	4	2.50	2	1
5	Biological Pre-treated Day 15	6	6	0.00	1.5	1

Table 2a: Fiber fraction determination for substrates

	S/N	Samples	ADF (mg)	Hemicellulose (mg)	Cellulose (mg)	ADL Lignin (mg)
1	Alkaline + Poultry dung	14.5	4.5	10.00	3.50	1.00
2	Biological + Poultry dung	11.5	4.5	7.00	3.5	1

Table 2b: Fiber fraction determination for digestate samples

3	Alkaline + Poultry dung	10.5	3.0	7.00	3.0	1
4	Biological + Poultry dung	8.0	3.0	5.00	3.0	1

For analyzed values of the raw NDF sawdust fiber sample, the result in Table 1 was 74.00mg which was significantly high, but when the pretreatment processes were carried out for day 5, day 10 and day 15, the results fell to 13.00mg, to 9.00mg and 6.00mg respectively, showing that the pretreatment processes brought down the initial high values of the NDF concentration in the fiber sample. Furthermore, when the pretreatment processes with poultry dung samples and the analyses of the digestate samples were done, the results in Table 2b showed 10.5 and 8.0mg respectively for the alkaline and the biological pretreatment processes.

For the ADF, the analyzed raw values obtained as shown in Table 1 was 67.00mg, but after the biological pretreatment process was conducted on day 5, day 10 and day 15, the results were 10.00mg, 4.00mg and 6.00mg respectively. After, the digestate samples were analyzed, the results in Tables 2b were 3.0mg and 3.0mg respectively. These results prove that the biological pretreatment process is a good approach for improving the digestibility of a wood material.

Results of physico-chemical analyses of substrates

The results of the physico-chemical parameters of the substrates before digestion and the digestate samples, with their mean and standard deviation values respectively are presented in Tables 3a and b respectively.

Generally, the results obtained for the physico-chemical parameters showed that with the exception of parameters such as total nitrogen, total phosphorus, total carbon and chemical oxygen demand, all the parameters were brought down by the pretreatment processes. The parameters that experienced increase instead of decrease may have experienced that because of the salts content of the chemicals used during the pretreatment processes, especially the chemical pretreatment process. However, this does not have any significant impact on their suitability for biogas production when the results are evaluated holistically. Furthermore, the results also revealed that the pretreatment processes were very effective in bringing down the raw values obtained for some of the parameters such as pH, total alkalinity, potassium, phosphate, manganese, iron and zinc as seen in Table 3a.

Table 3a: Physico-chemical analysis of substrates before digestion with mean and standard deviation values

S/N	Parameters	Sawdust	Alkaline Pretreatment	Biological Pretreatment	Alkaline + Poultry	Biological + Poultry
1	pH	7.38±0.6	9.02±0.64	8.15±0.63	8.21±0.64	7.59±0.6
2	T. ALK	350±125.8	165±125.82	230±125.8	490±125.82	260±125.81
3	T. N	25.5±5.4	24.0±5.42	28.0±5.40	34.0±5.41	36.5±5.42
4	T. P	2.67±0.2	1.85±0.1	2.07±0.15	4.67±0.3	2.52±0.2
5	T. CARBON	192.8±0.14	210.1±0.2	256.4±0.18	362.7±0.3	364.2±0.3
6	K	4.1±0.2	4.35±0.1	3.6±0.2	4.9±0.24	3.7±0.2
7	PO_4	56.0±0.1	86.5±0.2	95.0±0.2	140±0.3	125±0.2
8	SO_4	74.0±0.2	61.5±0.2	61.0±0.2	96.0±0.3	65.0±0.2
9	Ca	12±0.1	10±0.1	56±0.3	46±0.2	68±0.4
10	Mg	65±0.3	33±0.1	43±0.2	48±0.3	39±0.2
11	Mn	0.010±0.1	0.060±0.4	0.029±0.2	0.038±0.2	0.032±0.2
12	Fe	5.2±0.2	3.53±0.2	3.8±0.2	6.4±0.3	3.5±0.2
13	Zn	25.0±0.2	16.9±0.2	13.0±0.1	32.5±0.3	18.0±0.2
14	Al	0.35±0.2	0.24±0.1	0.36±0.2	0.53±0.3	0.38±0.2
15	Cu	2.5±0.3	1.95±0.1	2.36±0.2	3.7±0.2	2.8±0.3
16	BOD	110±0.2	94±0.1	132±0.1	150±0.2	196±0.3
17	COD	964±0.2	1210±0.2	1226±0.2	1468±0.3	1316±0.2
18	C/N	8:1±0.2	9:1±0.2	9:1±0.2	11:1±0.2	10:1±0.2
19	VOL. S	1216±0.1	2426±0.2	2430±0.2	4650±0.3	4650±0.3
20	% M. C	0.258±0.1	0.523±0.2	0.532±0.2	0.653±0.2	0.651±0.2
21	% T. S	0.662±0.5	0.128±0.1	0.116±0.1	0.156±0.1	0.147±0.1
22	% F. S	0.418±0.3	0.1875±0.1	0.207±0.2	0.197±0.2	0.246±0.3
23	% V. S	0.582±0.2	0.8125±0.3	0.793±0.2	0.803±0.3	0.754±0.2

N = 3

Note: T. ALK = Total Alkalinity; T.N = Total Nitrogen; T.P = Total Phosphorus; T. Carbon = Total Carbon; K = Potassium; PO_4 = Phosphate; SO_4 = Sulphate; Ca = Calcium; Mg = Magnesium; Mn = Manganese; Fe = Iron; Zn = Zinc; Al = Aluminium; Cu = Copper; BOD = Biochemical Oxygen Demand; COD = Chemical Oxygen Demand; C/N = Carbon/Nitrogen Ratio; % M.C = % Moisture Content; % T.S = % Total Solids; % F.S = % Fecal Sludge; % V.S = % Volatile Solids

When the digestion process was also completed with alkaline pretreatment processes and poultry dung samples as shown in Table 3b, the values showed that the alkaline pretreatment provided better results with the heavy metals, total solids and volatile solids and this may be caused because of the salts present in the chemicals used for the pretreatment.

Also, the biological pretreatment digestion with poultry dung process provided better results with parameters such as total alkalinity, total nitrogen and total carbon because of the decomposition process that is responsible for the entire process. Suffice to mention that the results obtained from the entire process renders the digestate samples obtained in this study fit for conversion into bio-fertilizer according to the standards prescribed by the United States Environmental Protection Agency (2009).

Table 3b: Physico-chemical analyses of digestate samples with mean and standard deviation values

S/N	Parameters	Alkaline + Poultry dung	Biological + Poultry dung
1	pH	8.12±0.5	7.38±0.4
2	T. ALK	475±0.6	268±0.4
3	T. N	36.8±0.4	38.2±0.5
4	T. P	4.48±0.6	2.43±0.4
5	T. CARBON	331.4±0.5	342.1±0.5
6	K	4.7±0.6	3.5±0.4
7	PO_4	143±0.5	128±0.5
8	SO_4	98±0.6	69±0.4
9	Ca	44±0.4	65±0.6
10	Mg	42±0.5	36±0.5
11	Mn	0.031±0.5	0.029±0.4
12	Fe	6.1±0.6	3.30±0.4
13	Zn	30.8±0.6	17.4±0.3
14	Al	0.43±0.6	0.34±0.4
15	Cu	3.3±0.6	2.5±0.4
16	BOD	164±0.3	175±0.6
17	COD	1440±0.5	1280±0.5
18	C/N	9:1±0.4	9:1±0.5
19	VOL. S	4480±0.6	4560±0.4
20	% M. C	63.1±0.5	62.7±0.45
21	% T. S	18.37±0.5	17.8±0.4
22	% F. S	22.2±0.4	26.1±0.5
23	% V. S	77.7±0.5	73.9±0.4

N = 3

Note: T. ALK = Total Alkalinity; T.N = Total Nitrogen; T.P = Total Phosphorus; T. Carbon = Total Carbon; K = Potassium; PO_4 = Phosphate; SO_4 = Sulphate; Ca = Calcium; Mg = Magnesium; Mn = Manganese; Fe = Iron; Zn = Zinc; Al = Aluminium; Cu = Copper; BOD = Biochemical Oxygen Demand; COD = Chemical Oxygen Demand; C/N = Carbon/Nitrogen Ratio; % M.C = % Moisture Content; % T.S = % Total Solids; % F.S = % Fecal Sludge; % V.S = % Volatile Solids

Biogas production from alkaline and biological pretreatment processes

The results of the biogas produced through the alkaline and biological pretreatment processes are presented Figures 5 and 6 respectively. The analyzed data showed that an average pH of 7.54 and an average temperature of 35.0^{0}C in the digester, the biological pretreatment process produced an average of 2mm quantity of biogas per day and a cumulative biogas production that rose steadily per day until the 32nd day of production, where the production reached 0.87912 L/KgTS. The data confirms that the biological pretreatment process enhanced biogas production, especially throughout the retention period.

Similarly, at an average pH of 8.15 and an average temperature of 35.1^{0}C in the digester for the chemical pretreatment process, the digester produced an average of 2mm quantity of biogas per day, an average of

0.03256 liter of production per day and a cumulative biogas production that rose steadily for the 32 days of retention up to 0.9768 L/Kg on the last retention day. Suffice to mention that the chemical pretreatment process yielded the higher value of biogas cumulatively when compared with the biological pretreatment process.

Laboratory analyses of biogas produced

The laboratory analyzed values, gas chromatography, mass spectrometry values and the percentage compositions of the biogas produced and collected in the digesters for the biological and chemical

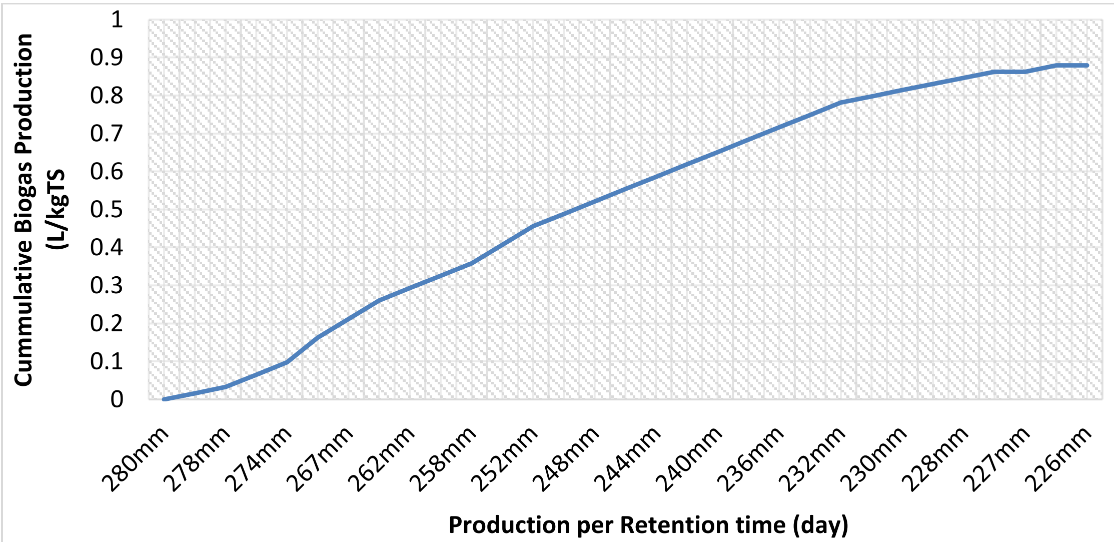

Figure 5: The cumulative of biogas production per day in the biological pretreatment against the number of retention time in days

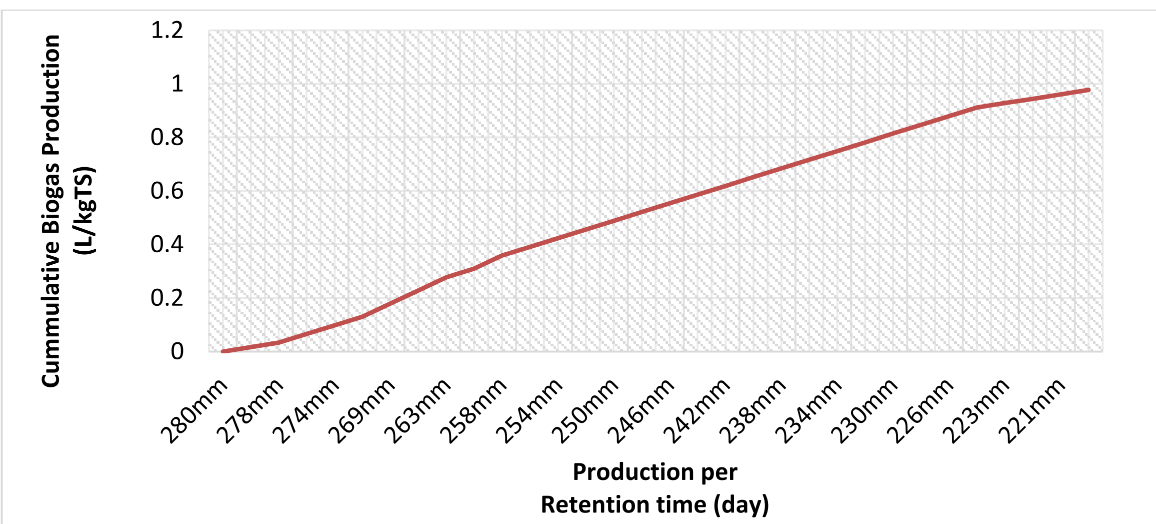

Figure 6: The cumulative of biogas production per day from chemical pretreatment against the number of retention time in days

pretreatment processes with poultry dung substrates samples are presented in Figures 7 and 8 respectively.

The results as presented in Figure 7 showed that out of all the gases analyzed, methane had the highest value of percentage composition which was 65.05%. According to Shayad and Mahmoud (2015), a standard grab sample of biogas that will perform satisfactorily should be one that is composed of over 60% methane and other gases in very negligible amounts, including carbon dioxide. To this end, the biogas sample obtained from this biological pretreatment and poultry dung substrates samples analysis meets the minimum requirement and can be described as wholesome. Similarly, as seen in Figure 8, methane returned the highest value of percentage composition (62.27%) of all the gases analyzed which also met the minimum requirements of a standard grab sample of biogas for the alkaline pretreatment process.

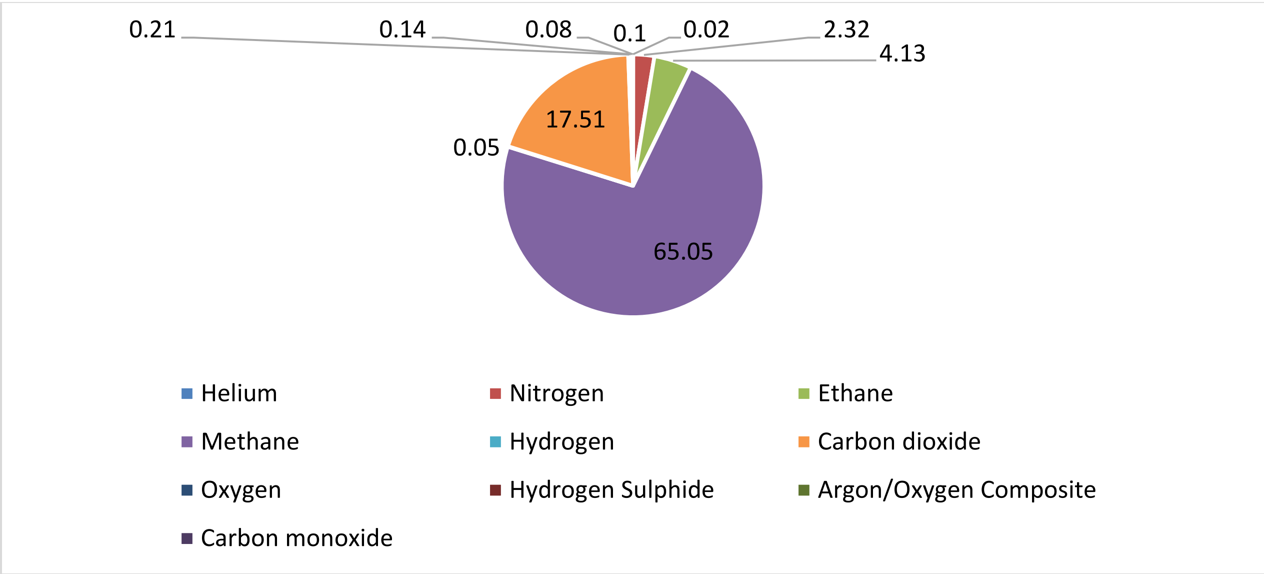

Figure 7: Analyses of biogas production samples from biological pretreatment process with poultry dung substrates

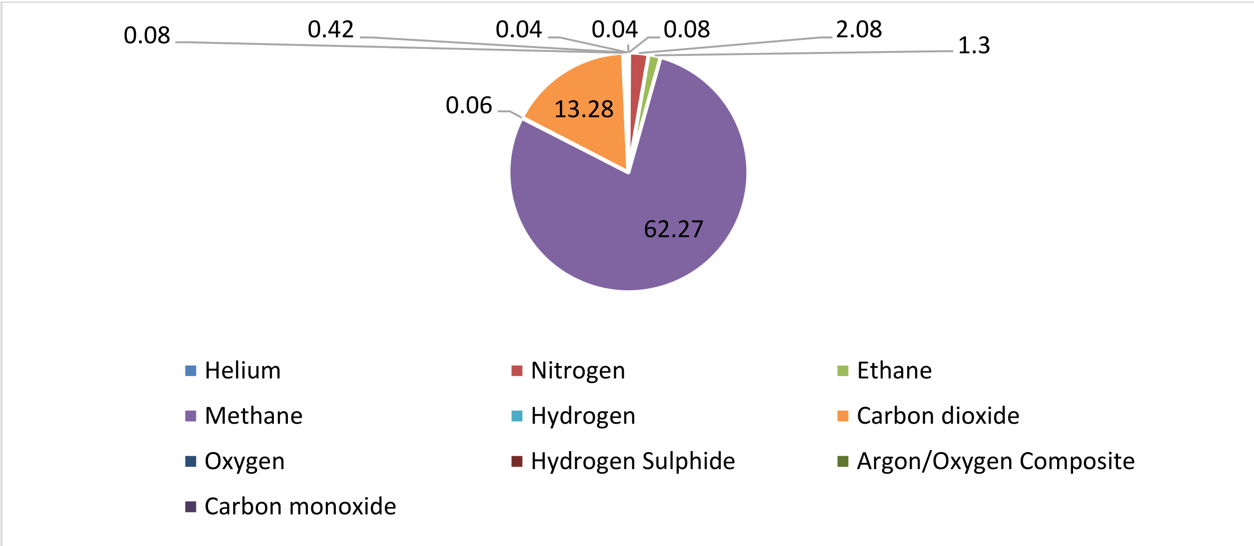

Figure 8: Analyses of biogas production samples from alkaline pretreatment process with poultry dung substrates

Conclusions

This study enhanced the production of biogas from sawdust through sustainable pretreatment methods. This was done through a laboratory scale pretreatment and digester and based on the results, it is concluded that;

i) Some of the parameters analyzed were not suitable for the anaerobic digestion process in the digesters after the pretreatment processes. However, after digestion, the biological pretreatment process with poultry dung substrates produced better conditions for biogas production relative to the chemical pretreatment process.

ii) The biological pretreatment process with poultry dung substrates produced the higher value of methane and ultimately, better biogas quality.

Recommendation

This study recommends that this work can be applied in sustainable biogas production for environmental protection, while further work can also be carried out on the conversion of the digestate samples obtained from the sustainable pretreatment methods into bio-fertilizer.

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