JSEE Journal of Sustainable Energy & Environment

Journal of Sustainable Energy & Environment

11 (2020) 93-100

Determination and removal of siloxane in energy recovery of biogas from municipal solid waste

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Abstract: Biogas produced from municipal solid waste requires proper purification due to the presence of siloxanes and other impurities. Siloxanes are converted to silicon dioxide at the combustion temperature and this forms deposits on the combustion surfaces of the engine components. Thus, to prolong the equipment life, purification is requires. This study therefore focused on the determination and removal of siloxane in energy recovery of biogas from municipal solid waste. Air Toxics Method was adopted for sampling and the composition of siloxane and other impurities were determined using gas chromatograph. Two purification filters were developed using transparent plastic container, iron sponge, iron fillings, silica gel, activated carbon made from palm kernel shell and calcium oxide. The biogas produced was divided into sample A, sample B, sample C, and sample D and evaluated before and after purification for the presence of siloxanes and other possible impurities. It was observed that the percentage composition of produced methane increases with continuous anaerobic digestion of substrates while the percentage composition of siloxanes, and other impurities decrease with duration and extent of biomethanation. The results obtained also confirm the present of siloxanes in the four samples analyzed (0.628 mg/m^3 for sample A, 0.638 mg/m^3 for sample B, 0.613 mg/m^3 for sample C, and 0.625 mg/m^3 for sample D). However, after purification, the concentration of siloxane was reduced tremendously (0.628 mg/m^3 for sample A, 0.638 mg/m^3 for sample B, 0.613 mg/m^3 for sample C, and 0.625 mg/m^3 for sample D).

Keywords: Siloxane, Biogas, Purification, Municipal Solid Waste, Impurities.

1. Introduction

Municipal Solid Waste Management (MSWM) is a major problem across the globe, although more noticeable in developing countries [1-3]. In Nigeria for instance, one can see a heap of solid waste dumped in open places as shown in Figure 1. Several researchers over the year have shown that the biodegradable portion of Municipal Solid Waste (MSW) can be converted to an energy source that is renewable and sustainable via the anaerobic digestion process [4-12]. The biogas technology involves the production of biogas from anaerobic digestion (AD) process of biodegradable solid waste in landfills and waste treatment plants [13]. The gas produced from the technology comprises mainly of 50-70% methane (CH_4), 30-40% carbon dioxide (CO_2), 0.5-1.0% hydrogen sulphide (H_2S), and water vapours (H_2O) [14-15]. However, a trace amount of undesirable siloxane and other impurities such as nitrogen, carbon monoxide, hydrogen, etc., are equally present [16-17].

Siloxane remains one of the most difficult impurities present in biogas to purify from the gas composition. The present of siloxane in biogas could hinder their use in energy recovery equipment such as internal combustion engine (ICE) [17-19]. During combustion of biogas, siloxanes oxidize to silicon (IV) oxide (SiO_2). These silicon (IV) oxides are deposited on the engine components such as cylinder heads, valves, and pistons as shown in Figure 2.

Figure 1. Open Dumpsite in Nigeria.

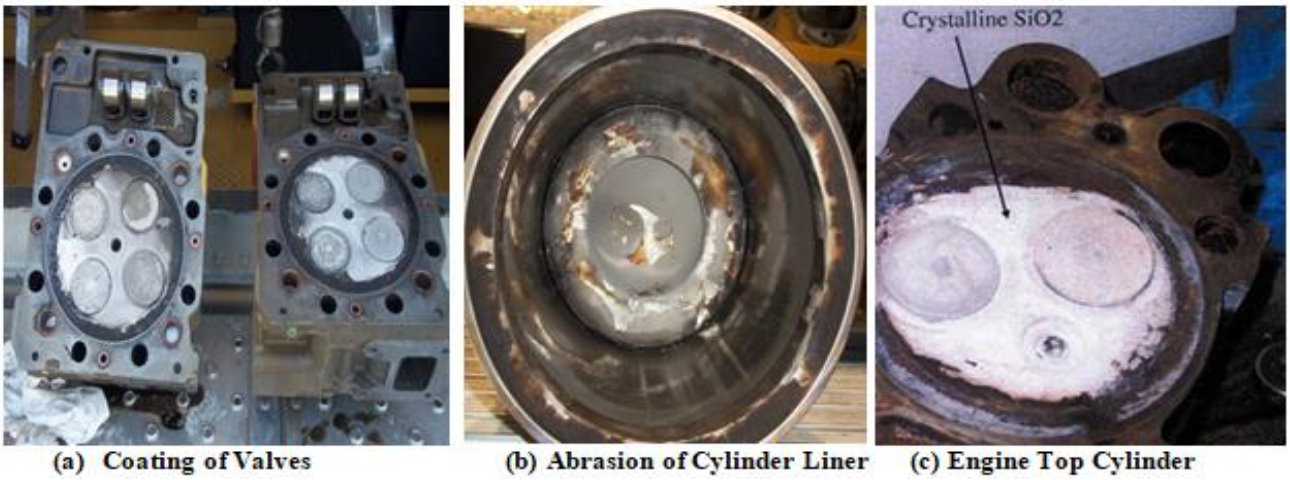

Figure 2. Effect of Siloxane on Engine Components [16-17].

Table 1. Some Typical Properties of Siloxanes Compound [16].

S/N	Compound	Abbreviation	Formula	MW	% Si	VP	BP	SW
1	Hexamethylcyclotrisiloxane	D_3	Si_3-O_3-$(CH_3)_6$	222	0.380	10	273	1.56
2	Octamethylcyclotetrasiloxane	D_4	Si_4-O_4-$(CH_3)_8$	297	0.378	1.3	347	0.06
3	Decamethylcyclopentasiloxane	D_5	Si_5-O_5-$(CH_3)_{10}$	371	0.379	0.4	410	0.02
5	Octamethyltrisiloxane	L_3	Si_3-O_2-$(CH_3)_8$	236	0.357	3.9	306	0.04
6	Trimethylsilanol	MOH	Si-$(CH_3)_3$-OH	90	0.312	-	210	-

*%Si-% Silicon, *MW-Molecular Weight, * VP-Vapor Pressure (mmHg), BP-Boiling Point (ºF), WS-Water Solubility (mg/l)

Upon the usage of the engine, the hard silicon residues abrade and rapidly wear off the engine surfaces [19-20]. Besides, silicon oxide as a good thermal insulator also contributes to the overheating of the engine components thereby reduces the compression and engine efficiency [21]. Also, as a result of the abraded nature of the slip liners of the ICE, the piston crowns become worn. These problems usually cause an estimated 75% reduction in engine life, valve plugging, and spark plug fouling [22].

Siloxanes a volatile organic silicon compounds (VOSCs) are usually found in commercial and consumer products, such as cosmetics and detergents. Irrespective of the fact that some siloxanes quickly volatilize into the atmosphere but other less volatile compounds of siloxanes end up in biogas as impurities. Some typical siloxane compounds, formula and their properties are shown in Table 1. The quantity of total volatile siloxanes content and their relative percentage composition in biogas vary depending on the sources of waste composition used for the production of biogas. In wastewater for instance, hydroscopic siloxanes accumulate in the sewage sludge and under anaerobic digestion process; it is transfer as an impurities in produce biogas. Similarly, siloxanes in the anaerobic environment of an open dumpsite and landfill also transfer to the biogas [17, 23-24]. The presence of siloxanes in biogas is found in the form of a white powder in gas turbine hot section components, or as a light coating on various types of heat exchangers. Also, it can be reveal as a deposit on combustion surfaces in reciprocating engines and as a light coating on post-combustion catalysts [16, 25]. The white powder formed on engine and power plant components are primarily silicon dioxide (SiO_2), a product of siloxane combustion [16, 23].

Furthermore, if the technology for the purification of biogas is efficiently developed, overdependence on fossil fuel for powering of engine and power plant will be reduced. Methane an active greenhouse gas contributes to global warming. However, biogas usage via effective burning will help to controls the amount of methane gas released into the atmosphere. Adoption of biogas technology as part of solutions to energy problem is not complete without considering the purification process. As a result of this, there is need to determine and remove unwanted impurities such as siloxane in energy recovery of biogas from municipal solid waste**.** Although, efforts have been made by several researchers in the past in finding solution to the above mentioned problems, but more need to be done especially in Sub-Sahara Africa where the technology is still at the embryo stage.

2. Materials and Methods

2.1 Materials

Table 2 shows the list of equipment and materials used in this research work.

Table 2. List of Materials and their Usage.

S/N	List of Equipment and Materials	Usage
1.	Anaerobic digestion plant	Anaerobic digestion plant was used for co-digestion of substrates.
2.	Nose mask	For protection against poisonous gases, contaminants from collected household wastes.
3.	Substrate	The substrates used in this research work include biodegradable portion of municipal solid waste.
4.	Gesa thermometer	A Termometros SL analogue thermometer manufactured by GESA with precision of $\pm 0.01^{o}C$ was used to monitor the temperature reading of the slurry.
5.	Pressure gauge	A CPG pressure gauge manufactured by WIKA company with precision of $\pm 0.01\, bar$ was used for monitoring pressure buildup of generated biogas
6.	Gas Chromatograph	Trace 1310 gas chromatograph, manufactured by Thermo Scientific with precision of ± 0.01 was used to determine the percentage composition of biogas components present.

The composition of waste used in this research work is shown in Figure 3.

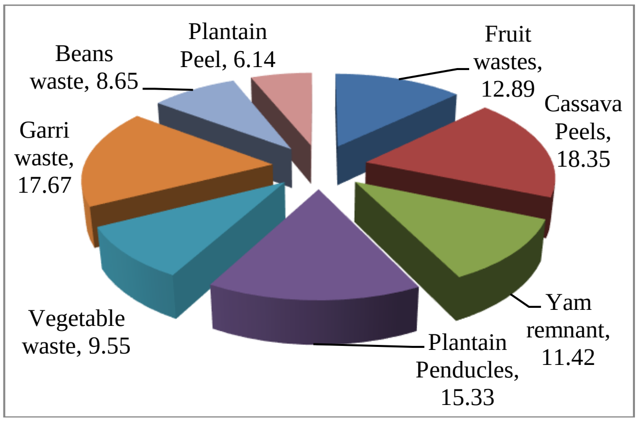

Figure 3. Compositions of Solid Wastes.

2.2 Methods

2.2.1 Biogas Production

Biodegradable portion of MSW that comprises of garri waste, plantain peels, fruit waste, cassava peels, yam remnants, vegetable wastes, beans waste, and plantain peduncles were collected from open dumpsite in Okada, Nigeria. The sorted portion was properly pulverized to reduce its size and same time increase it surface area of contact for microbe to act on [26]. The fine portion was mixed with water in a ratio of 1:2 and then charged into AD plant for anaerobic digestion to take place. Pressure gauge was monitored in an interval of 12 hours for the production of biogas and flame test was immediately carried out once the pressure gauge indicates an increase. Formation of blue flame is an indication of biogas production. At this stage the biogas produced is evacuated for analysis. The evacuated biogas sample is divided into four portions (sample A, sample B, sample C, and sample D) and four successive tests were carried out on each sample before and after purification.

2.2.2 Sampling and Analyzing for Siloxanes

The Air Toxics Method (ATM) via Methanol Impinger Method (MIM) as reported by [16] was adopted in sampling and analysis of the presence of siloxanes in evacuated biogas sample. The following steps were carried out;

i. The evacuated biogas is bubbled separately through two midget impingers in series

ii. Siloxanes are absorbed into the methanol and run for 3 hours

iii. The present and percentage composition of siloxane is determined using gas chromatograph (GC)

iv. The concentration of siloxanes in biogas is calculated based on gas volume analyzed

Figure 4 shows the experimental setup.

2.2.3 Development of a Purification Filter

Biogas purification filter for removal of siloxane from biogas was developed from a transparent plastic container. It carries both inlet and outlet valves. The inlet valve receives impure biogas while the outlet valves evacuate purified biogas from the filter. The biogas sample confirm with the presence of siloxane is purified after which the processes/stages in section (2.2.2) is repeated to determine the efficacy of the developed purification filter.

2.2.4 Biogas Purification

Two separate biogas purification filter were used in this research work. The first biogas purification filter labeled "Purification Filter A" was used mainly to remove carbon (IV) oxide, water vapour, and hydrogen sulphide while the second purification filter labeled "Purification Filter B" was used for the removal of siloxane from the biogas composition. The purification filter A consists of three compartments. Each compartment consists of three separation units arranged in the following order: Water vapor separation unit followed by carbon (IV) oxide (CO_2) separation unit and hydrogen sulphide (H_2S) separation unit. The masses of the samples are as follows: 500 g of iron fillings, 500 g of silica gel and 500 g of quicklime. The masses were impregnated on separate iron sponges and placed in the cylindrical polyethene container in this order: the iron sponge with silica gel was inserted first followed with that of calcium oxide (CaO) and finally iron filings. The purification filter carries inlet valve and outlet valve. The inlet valve receives biogas from the digester while the outlet valve takes the biogas to the evacuation unit. Figure 5 shows the pictorial view of the biogas purification filters.

Figure 4. Testing for the Presence of Siloxane in Biogas.

2.2.5 Preparation of Activated Charcoal

Palm kernel shells were collected from Okada, Nigeria. The collected palm kernel shells were washed thoroughly with clean water and allowed to completely dry in laboratory oven with temperature set at 105°C mainly to enhance combustion. The completely dried palm kernel shells were burned to ashes. To activate the charcoal from the palm kernel shell, 500 g of quicklime (CaO) was dissolved in 1000 ml of water as chemical activator. The aqueous solution was mixed with the charcoal in a 1000 ml beaker and allowed for duration of twenty four (24) hours. The charcoal was transferred to a mesh and allowed to properly drain. The charcoal was washed in water repeatedly and properly dried in a laboratory oven. The dried charcoal was pulverized and impregnate in a steel iron sponge filter B.

(a) Purification Filter A (b) Purification Filter B

Figure 5. Pictorial View of Biogas Purification Filter.

Part	Name
A	Injection port
B	Detection under flip-up covers
C	Control pad
D	Oven door
E	Gas flow control
F	Monitor

Figure 6. Gas Chromatograph.

2.2.6 Characterization of Produced Biogas

An analytical technique via gas chromatograph (GC) was adopted. A gas chromatograph (GC) was utilized to separate the biogas sample into its components followed by detection using a mass spectrometry (MS) detector. Trace 1310 gas chromatograph, manufactured by Thermo Scientific with a precision of ± 0.01 was used to determine the percentage composition of biogas yields before and after purification. Figure 6 shows the experimental set up. The following procedures were followed:

i. The evacuated biogas sample was injected into the gas chromatograph.

ii. A gas stream (Helium gas carrier) was used to transport the biogas sample into a separation tube (column).

iii. The various biogas compositions were separated inside the column.

iv. Mass spectrometry (MS) detector was used to measures the quantity of the components that exit the column.

3. Results and Discussion

The results of tests run to check the concentration of siloxanes in biogas is shown in Table 3. However, to know the exact percentage composition of constituent present in biogas produced from biodegradable portion of municipal solid waste collected from open dumpsite in Okada, Nigeria, a gas chromatograph was used to analyze the impure and purified biogas. The results obtained are shown in Figures 7-11 and Figure 12.

Table 3. Test Results for Concentration of Siloxane in Biogas before Purification.

Test	Portion (mg/m^3)			
	A	B	C	D
1	0.67	0.65	0.68	0.66
2	0.61	0.59	0.60	0.59
3	0.68	0.64	0.58	0.67
4	0.55	0.67	0.59	0.58
$\sum$	2.51	2.55	2.45	2.50
Ave.	0.628	0.638	0.613	0.625

As presented in Figure 7, the average concentration of siloxanes in biogas before and after purification showed that for impure biogas, the average concentration of siloxanes present in the four samples used in this study are as follow; 0.628 mg/m^3 for sample A, 0.638 mg/m^3 for sample B, 0.613 mg/m^3 for sample C, and 0.625 mg/m^3 for sample D. The concentration of siloxane obtained agreed with the research work of Jeffrey et al. [16] titled "Siloxane sampling, analysis and data reporting recommendations on standardization for the biogas utilization industry". Also, it was gathered that after purification, the concentration of siloxane in biogas reduces tremendously, the values obtained be; 0.0005 mg/m^3 for sample A, 0.0008 mg/m^3 for sample B, 0.0005 mg/m^3 for sample C, and 0.0007 mg/m^3 for sample D. The drop in the concentration of siloxanes in biogas can be attributed to the efficacy of the chemical activator produced from the activated charcoal of a palm kernel shell using 500 g of quicklime calcium oxide.

Furthermore, the results of the percentage composition of methane (CH_4), carbon(IV) oxide (CO_2), water vapour (H_2O), hydrogen sulphide (H_2S), and other gases that comprises of siloxane carbon (II) oxide (CO), nitrogen gas (N_2), and hydrogen as shown in Figures 7-10 keep on varying with the duration and extent of biomethanation. As the anaerobic digestion process continues in the AD plant, there was continuous increase in methane yield and subsequent drop in production of carbon dioxide, water vapour, hydrogen sulphide, and other gaseous constituents that comprised of nitrogen gas, hydrogen, and carbon (II) oxide. The range of percentage composition of impure biogas yield from biodegradable MSW was as follow; methane (58.06-67.45%), carbon (IV) oxide (30-38.36%), water vapour (1.62-2.50%), hydrogen sulphide (0.85-1.05%), and other gaseous impurities that comprises of siloxanes, carbon (II) oxide, hydrogen gas, and nitrogen gas (0.02-0.16%). These results go in line with the research work of [15, 23] that reported similar range of percentage composition of biogas yield. The findings also agreed with the work of [28-31]. In the research work carried out by Al Mamum and Shuichi [28] entitled enhancement of methane concentration by removing contaminants from biogas mixtures using combined method of absorption and adsorption reported percentage composition of biogas of 60-70% combustible methane and 30-40% non-combustible carbon (IV) oxide along with smaller amounts of other gases such as nitrogen (<1%), carbon (II) oxide (<0.6%), hydrogen sulphide (0.005-2%), and water vapours (5-10%). However, according to Herout et al. [29], the percentage biogas composition depends mostly on the type of substrates used. In their research work, they reported the percentage composition of biogas as follow: 50-85% methane; 20-35% carbon (IV) oxide. While, Franco et al. [30] reported methane percentage composition between 50% and 80% in their research work entitled Lignocellulosic biomass feeding in biogas pathway: state of the art and plant layouts.

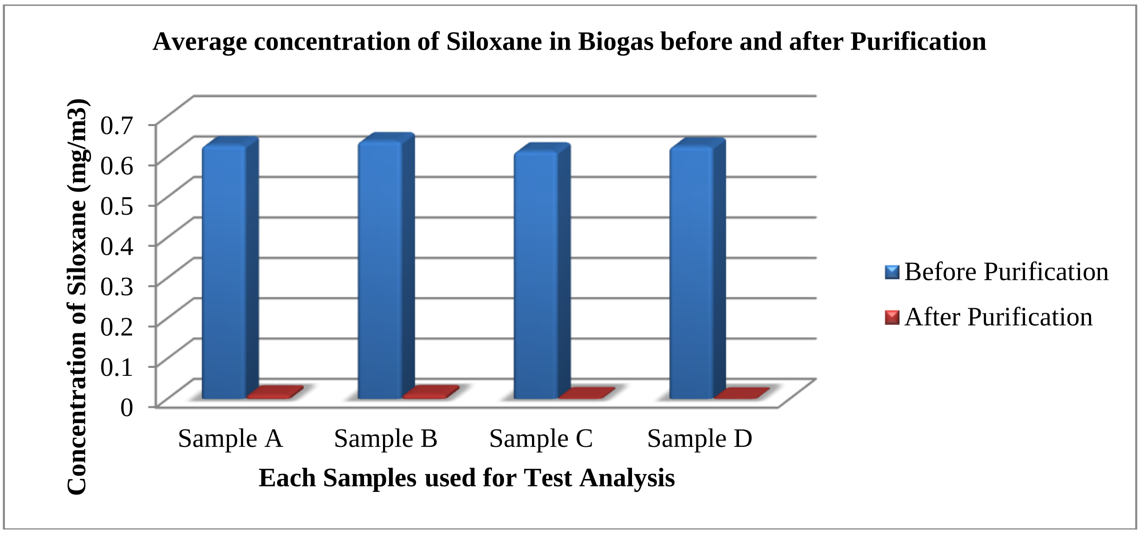

Figure 7. Average Concentration of Siloxane in Biogas before and after Purification.

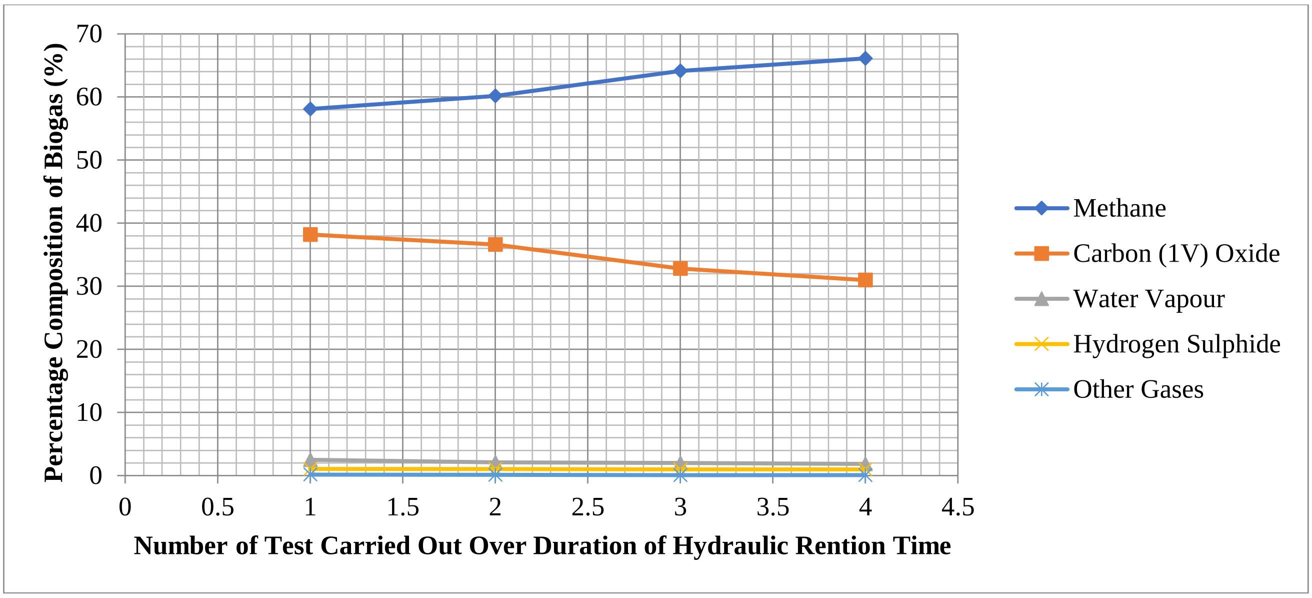

Figure 8. Biogas Percentage Composition of Sample A.

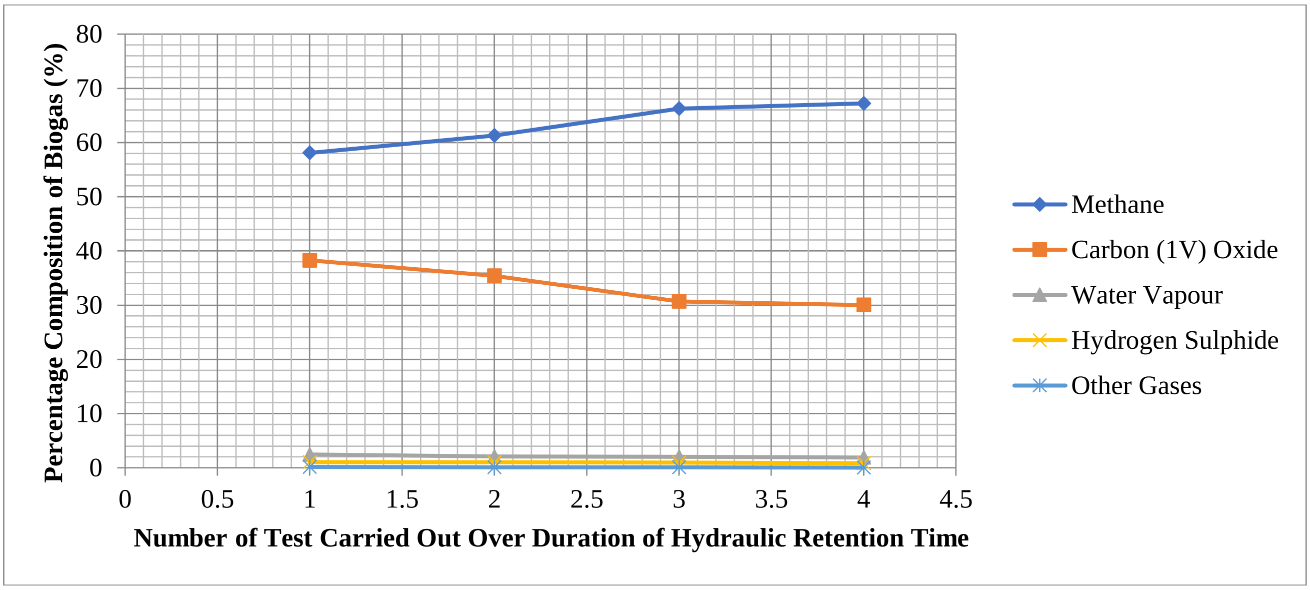

Figure 9. Biogas Percentage Composition of Sample B.

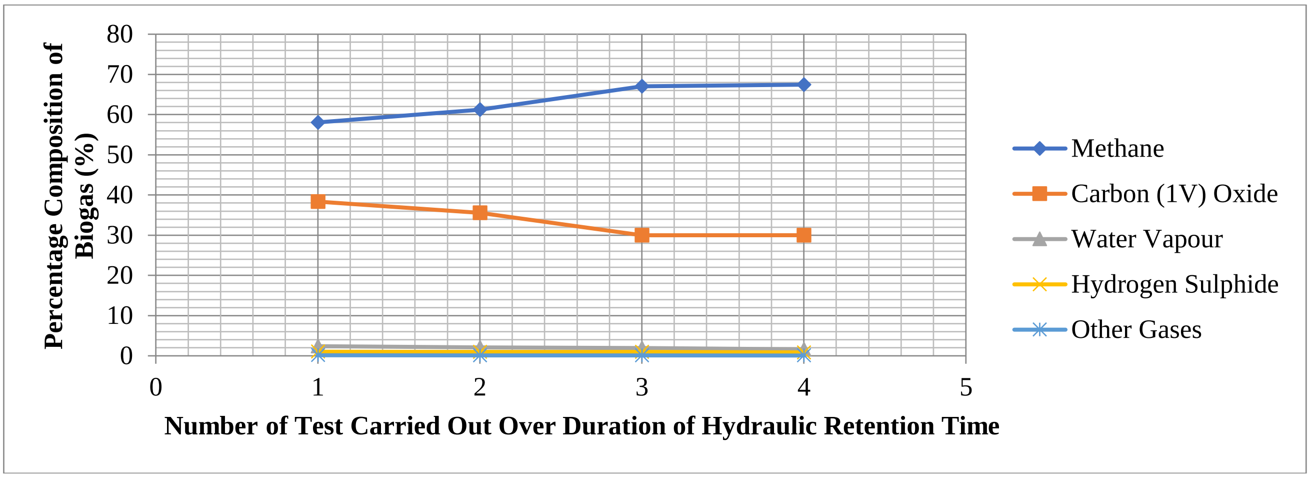

Figure 10. Biogas Percentage Composition of Sample C.

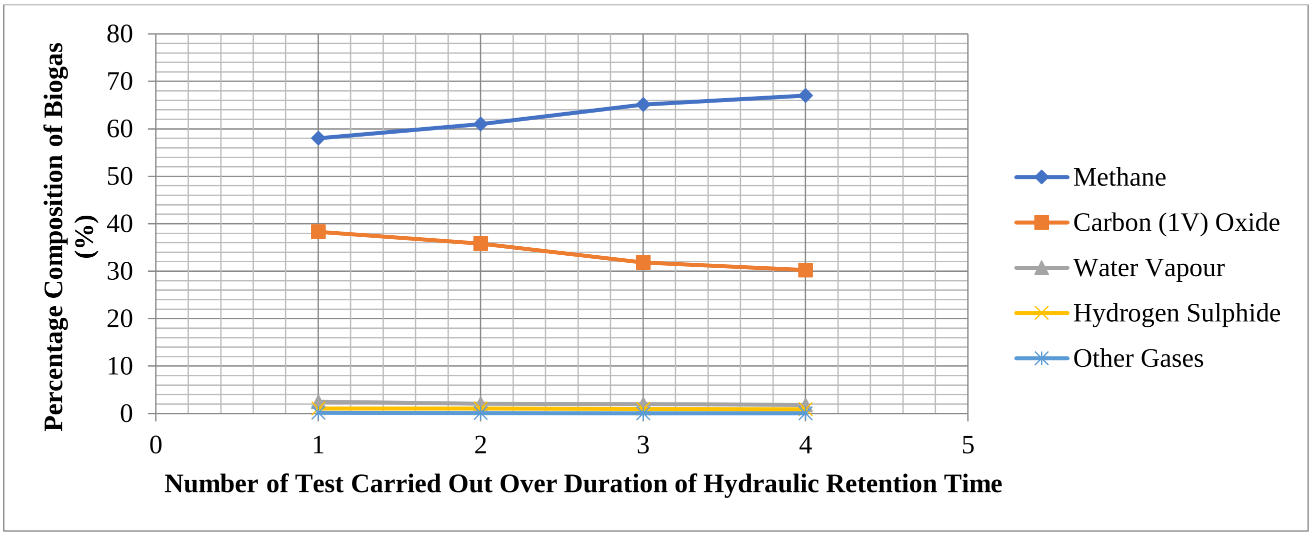

Figure 11. Biogas Percentage Composition of Sample D.

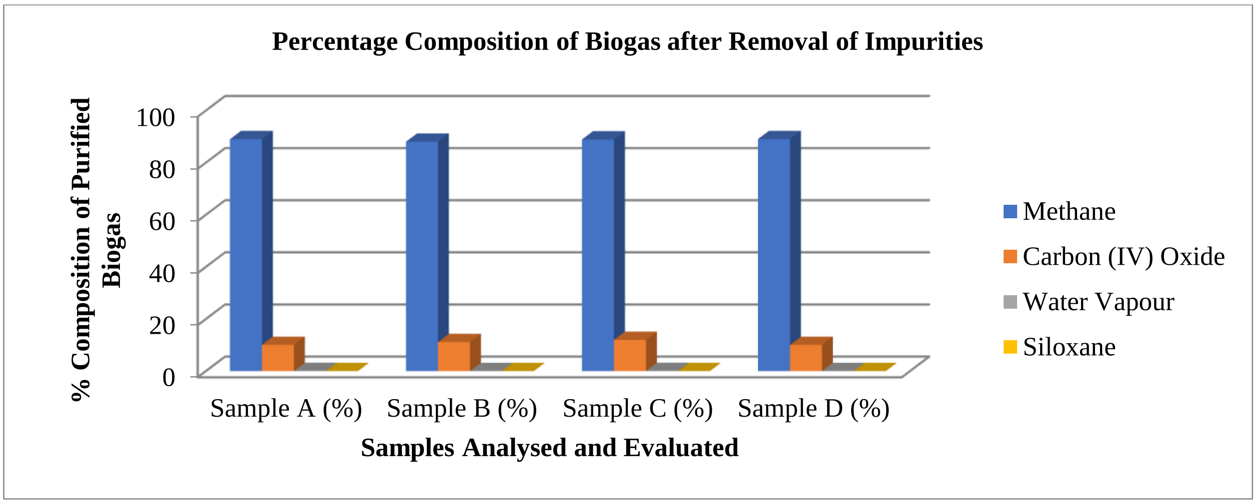

Figure 12. Biogas Percentage Composition of Purified Biogas.

As shown in Figure 12, there was a decreased in the percentage composition of all gaseous components present in biogas. The comparative analysis of percentage (%) composition of methane after purification showed improvement in percentage composition of methane from range of 58.10%-67.45% to range of 88.01%-89.99%. Also, there was huge drop in percentage composition of carbon (IV) oxide. Therefore, it can be deduce that the calcium oxide used in purification filter A was able to purify it, thus the drop in percentage composition from range of 30%-38.36% to 10.01%-12.84%. The other impurities were totally removed except for water and siloxanes (other gases) that was left with negligible percentages.

4. Conclusion

In this study, determination and removal of siloxane in energy recovery of biogas from biodegradable portion of municipal solid waste collected from Okada open dumpsite in Nigeria was successfully carried out. This was done mainly to safely use biogas for energy generation in internal combustion engines and power plants. A purification filter was developed for the removal of siloxane and other gaseous impurities from biogas. It can be deduced from the study that the percentage composition of methane (CH_4) increases with continuous anaerobic digestion of substrates while the percentage composition of siloxanes, carbon dioxide (CO_2) and other impurities decrease with duration and extent of biomethanation over hydraulic retention time. It was also revealed that siloxanes can be removed from biogas with chemical activator produced from the activated charcoal of palm kernel shell. Therefore, biogas containing siloxane impurities can be purified and utilized safely as fuels for engines and power plants.

Acknowledgment

The researchers acknowledge the support of the staff and students of the Department of Mechanical Engineering, College of Engineering, Igbinedion University, Okada, Nigeria for their professional criticism and support towards the successful completion of this study.

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