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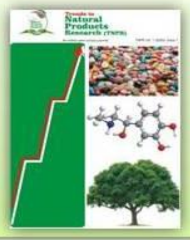

The Potentials of Biogas on Groundnut Shells and Rice Husk using Heterogeneous Catalysts

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Abstract: The main aim of this research work is to determine the potentials of biogas on groundnut shells and rice husk using heterogeneous catalysts. The result reveals yields produced in both groundnut shell and rice husk without catalyst were (650 cm^3) and (608.9 cm^3) respectively in comparison to the yield obtained through catalysts influence as (1418 cm^3). However, proximate analysis of the substrate to indicates its viability for biogas generation is encouraging as the groundnut shell has moisture content (40.5 ± 0.06 %) while rice husk has (32.0 ± 0.02 %), volatile matter of groundnut shell and rice husk were (80.5 ± 0.45 %) and (71.5 ± 0.031 %) respectively. The result reveals that heterogeneous catalysts ZnO and CaO have little significant effects in biogas generation using both groundnut shell and husk shell as substrates. However, proximate analysis of the substrate indicated its viability for biogas generation is encouraging as the groundnut shell has the highest moisture content fallowed by rice husk. Elemental composition of the substrates proves that the slurry could be used as bio fertilizer as the nitrogen content was (1.16 ± 0.003) which is higher than that of rice husk (1.06 ± 0.05), phosphorus (4.14 ± 0.006) and potassium content for rice husk were higher than that of groundnut shell (16 ± 353), on comprising groundnut shell is more viable as a candidate for biogas generation.

INTRODUCTION

The renewable energy sources play a vital role in an effort to solve the present and future energy problems. African countries require sustainable energy supplies for their economic development. Unreliable energy supply might end up with low of private investment in African continent. Therefore, improvement in the quality and magnitude of energy services in developing countries is needed to meet developmental objectives including the Millennium Development Goals (Ismail et al., 2016).

Energy is one of the key important resources for mankind. Today, energy crisis becomes one of the issues facing or throwing threat to the globe. Fuels on the other hand are of paramount importance because they could be burned to produce significant amounts of energy. Furthermore, the burning of conventional fuels (fossil fuels) is associated with the environmental consequence which eventually affects the ecosystem. The energy demands increase day by day along with world's population growth which eventually becomes more difficult to cover the entire demand considering the fact that the most of the used source of energy (fossil fuels), are likely to run out of existence in a near future for the reason that it is a non-renewable source of energy (Garba,1996).

Biomass in the form of mainly fuel wood and charcoal is the dominant energy source in Sub-Sahara Africa. Though it appears cheap, overexploitation of this biomass leads to serious negative environmental consequences. However, the increased prices of oil and increased awareness of climate changes is promoting the use of alternative environmentally friendly renewable energy sources such as biogas (Casebow, 1987). Among the alternative energy sources, biogas production from organic wastes has worldwide application as it yields good quality fuel and fermented slurry, which may be used as a manure or soil conditioner. In addition, it helps to a great extent in the abatement of pollution. Biogas has a very positive impact on the environment since less CO_2 is formed during its combustion than it is used for photosynthesis by the plants (Chernousova and Epple, 2013).

Biogas over the years has been known to be a combustible gas which is produced from organic and inorganic matter. This process usually occurs by anaerobic fermentation and decomposition of plant and animal material. This fermentation process occurs in the absence of air and at certain temperature in the presence of a substantial amount of water. The material used to produce this gas is made into slurry (a sort of paste by mixing with water at different rations) and is fed into a digester. Biogas is defined as a combustible gas (composed primarily of methane) produced when sewage or manure is fermented in the absence of oxygen. The solid material that remains in the digester after fermentation can be used as an organic fertilizer (Ismail *et al*., 2016). Biogas is a mixture, consisting of approximately 40 to 75 % methane (CH_4), 25 to 60 % carbon dioxide (CO_2) and approximately 2 % other gases (Hydrogen, hydrogen sulphide and carbon-monoxide) (Brunow, 2006).

MATERIALS AND METHODS

The chemical reagents used in this research are: Zinc carbonate (A.R, 97.0 Qualikems), Sodium Hydroxide (A.R, 97.0 BDH Limited), Hydrochloric Acid (A.C.S 37.0 BDH Limited), Sulphuric Acid (L.G, 97.0 BDH Limited), Potassium hydroxide (A.R, 97.0 Qualikems). L.G = Laboratory grade, B.D.H=British drug house. While instruments used in the research work are: Analytical balance (Aw320, Shimadzu Japan), Autoclave (SS316L, fabricated in India), FTIR Spectroscopy (Carry 630, Agilents Techn), XRT Machine (X-Supreme 8000, Oxford instrument), XRD Analysizer (Empyrean, PAN Analytical), Muffle Furnace (MF 207, Turkey), PM meter (M.C 400, Oxford).

The substrates used in this research work are groundnut shells and rice husk. Groundnut shells were obtained from hubbare area, Sokoto while rice husk was obtained from Kalambaina area. The substrates were sampled, sun dried, grounded to powder and subsequently used in the preparation of slurries. The Pre-treatment of the groundnut shell and rice husk was conducted with KOH aqueous solution to remove Lignin component. The groundnut shell was immersed in to a 6% KOH aqueous solution with a solid – liquid ratio of 1:20, then loaded in to a 50 cm^3Teflon lined stainless steel autoclave. The autoclave was heated to 120ºC and kept for 4 hours. After that, the autoclave was cooled to room temperature and black liquid mainly consisting of lignin and KOH was separated from the mixture by centrifugation. The collected residual solid material was subsequently washed repeatedly with distilled water till the filtrate became neutral, and then dried at 80ºC for 24 hours in oven (Jialei *et al*., 2017).

Three slurry concentration of each of the treated samples were prepared. The first series of the slurry was made by mixing 2g of CaO, 2g of ZnO, with 600g of the substrate and 3000 cm^3 of water in a beaker while the second series was made by mixing 2g of CaO, and 2g of ZnO with 600g of substrate and 3600 cm^3 of water also in a beaker the third series was made by mixing 2g of CaO, 2g of ZnO, with 300g of the substrate and 1200 cm^3 of water in a beaker. The substrate to water ratio are 1:5, 1:6 and 1:4 respectively. Rice husk slurry was labeled as R (1:5), groundnut shell slurry G 1:6 and mixed rice husk and groundnut shell slurry RG 1:4 (Ismail *et al*., 2016).

Determination of proximate composition of the substrates was carried out in accordance with A.O.A.C. methods (1990). The crude fibre was determined parameters determined by using 2.0g sample the sample was allowed to drain, dried overnight at 150ºC in the oven. Then the crude fibre was calculated using the following formula below.

$$\%\ \text{Crude fiber} = \text{W1} - \text{W2}/\text{W1 x 100} \ldots\ldots\ldots\ldots\ldots\ldots \quad (1)$$

The Sample was ashes at 500ºC for 90 minutes in a muffle furnace, then cooled in a desiccator and weighed in accordance to the Rabah *et al*. (2011). The carbohydrate content of the samples was determined as the difference obtained after subtracting the values of organic protein, lipid, ash, and fiber from the total dry matter (Abdulrazak *et al.*, 2014).

Total moisture of the sample collected will be estimated by the method of Zhang *et al.*, (2014). In determining the moisture content, evaporating dishes were dried in an oven at 105 ^{0}C cooled in a desiccator and weighed. 5g of fresh sample will be placed in each of the dried evaporating dishes and kept in an oven maintained at a temperature of 105ºC for 24 hours. After drying, the evaporating dishes were removed, cooled in a desiccator and weighed A.O.A.C. methods (1990). The percentage moisture content will be calculated as percentage loss in weight using the formula.

$$\%\ Moisture = Loss\ in\ weight\ after\ drying\ X\ 100 \ldots\ldots\ldots\ldots \quad (2)$$

The pH of the substrate was determined from each digester directly using digital pH meter. Similarly, the total solids represent the solid left after heating at 105ºC for 24 hours. The volatile solids (organic matter) of the sample were determined by difference. This was calculated as shown by the equation 3.

$$\text{V.S} = 100 - (\%\ \text{moisture} + \%\ \text{Ash}) \ldots\ldots\ldots\ldots \quad (3)$$

Determination of Potassium and Phosphorous

The flame photometry method by AOAC (1990) as modified by Brunow (2006) was adopted for the determination of potassium. The vanadate-molybdate method reported by AOAC (1990). In this method 5cm^3 of HCl Acid was added to each sample and sample was diluted to 50 cm^3 of distilled water, then the sample transferred to the bottles, 2cm^3 of it was taken for the determination of phosphorous inside 50ml volumetric flask. 2cm^3 phosphorous extraction (NH_4F+HCl) Solvent was added to the sample, also 2cm^3 of ammonium molybdate added to the sample and also distilled water to the three quarter of the 50 cm^3 of volumetric flask 1cm^3 of diluted stannous chloride was added to the sample, filled the bottle with water to the mark level of the bottle the sample was taken Inside curvet for the absorbent of the phosphorous the sample was placed in the spectrophotometer and closed.

Preparation of Catalysts and Characterization

Both ZnO and CaO catalysts were prepared by calcination of the $ZnCO_3$ and Snail Shell respectively as their Precursors. Their fine powders were calcined at 800 ^{0}C for 3 hrs (Hadiyanto *et al.*, 2016; Ismail *et al.*, 2016). After that, two catalysts prepared were characterized using X-ray fluorescence (XRF), Fourier Transform Infrared Spectroscopy (FTIR) and X-ray diffraction (XRD).

Statistical Analysis

Proximate analyses of the substrates were calculated according to Tambuwal (2005) and Abdulrazak *et al.* (2014). FTIR Spectroscopic XRF and XRD analysis at 95 % significance level was used to compare the mean. Data obtained was subjected to one-way analysis of variance (ANOVA) and means were separated using least significant difference (LSD) at 0.05 probability level.

RESULTS AND DISCUSSION

Proximate analysis of undigested substrates carried out before and after digestion are shown below in table 1 and 2

Table 1 and Table 2 give the comparative mean value (%) of ash and moisture content of the samples before and after digestion used in biogas generation. Ash content can be defined as a non-volatile inorganic matter of a substance, which remains after subjecting it to a high decomposition temperature, while moisture content is simply the water content of sample. Ash content determination is significant in biogas production because it reveals the elemental content of N, P, K etc. present in the sample from table 3.2 the result shows that Rice Husk has the highest ash content of (25.5± 0.08 %) followed by groundnut shell with (16.0±0.38 %) respectively. The result is in congruence with the conclusion made by Grzegorz *et al.* (2015) that the high Ash content (50-55 %) reduces the biogas production potentials as only organic matter can be converted to biogas and in this work the result of obtained are not up to (50-55 %). Similarly, the high percentage of Ash indicates that most of the substrate would be viable for biogas production. However, moisture content value is favourable for biogas production potentials as recommended by Bagudo *et al.* (2008) the high moisture will help in early digestion. The result obtained in this research work shows that the moisture contributed to the early digestion and can be seen that all the substrate start production on first week.

Table 1 and 2 shows the result of proximate analysis of total solid content of the substrate in standard deviation, in this research work the percentage of total solid of groundnut shell are 70±0.6 before the digestion and 68.3±.0.04 after the digestion while for Rice husk the value obtained for total solid are 60.0±0.015 before the digestion and 52.01±0.012 after the digestion respectively, this result is in line with the work of Rabah *et al.* (2011) who reported a decrease in the value of total solid after anaerobic digestion. The values of volatile matter in table 1 and 2 were 80 05±0.045, 71.5± 0.031 for grandaunt shell and rice husk respectively before the digestion. After the anaerobic digestion the values of the volatile solids were 30±0.03, 25.5±0.017 respectively for groundnut shell and Rice husk the reduction in their values was as a result of the volatile solid that has been converted into biogas. This result is in agreement with the work of Abdulrazak. *et al*, (2014).

The crude fiber obtained was 14.5± 0.16 for groundnut shell and 27± 0.14 % for rice husk, crude fiber in food or waste is an indication of the level of non-digestible carbon hydrate and lignin. This high level is considered appropriate and shown that both groundnut shell and rice husk is non-edible material for human and can be used for the production of renewable of fuels and chemicals, hence sustainable production of chemicals can be achieved without affecting food supplies. Crude fiber is made up largely of cellulose together with a small quantity of lignin which is indigestible in Human (Ikbal *et al.*, 2018). Also, the result showed groundnut shells contained appreciable amount of the crude lipid (1.66± 0.02 %). For groundnut shell and 0.33± 0.01 % for rice husk. Crude protein was 7.29± 0.007 % for groundnut shell and 3.26± 0.011 % for rice husk respectively. This high value of nitrogen is an indication that the substrate could be serve as good source of biogas production (BoonSawang, *et al*, 2014). The high level of carbohydrate content 17.34± & 41.2± 1.26 % in both the substrates showed that groundnut shells have a relatively low nutritive value and can be used as an Alternative source for the production of bio energy. The finding of this work is in agreement with Abdulrazak, *et al*. (2014) who reported a similar range of proximate composition values.

Table 3 gives the comparative value of the carbon to nitrogen ratio for both rice husk and groundnut shell obtained in this research work. The quantity of biogas yield depends on the number of total solids present in the slurry and their digestively, in this work C/N ratio was 36.74 and 33.90 for groundnut shell and rice husk before digestion respectively. C/N ratio is one of the major criteria for a better biogas production. The closer to the range of the recommended value of C/N ratio 30:1 the high the biogas yield, reason could be linked to the fact that low level of Nitrogen limits the growth of bacteria action while excessively high level could be toxic to the bacteria as noted by the work of (Bagudo *et. al*., 2008). According to Casebow (1987) who had reported that anaerobic bacteria use up to 30 times carbon faster than Nitrogen. The fact bacteria use carbon as food and Nitrogen to replenish it tissues.

Table 3 shows the result of carbon content of the substrates used in this research work, organic matter content of the substrates influences their biogas yield since microbes Act on the organic matter of the substrates to produce biogas, it would imply that low organic matter content should result in low biogas yield and high organic matter content result in more yield of biogas. Hence this is because microbes acquired carbon for their food while acquired nitrogen for their enrichment of their tissues. (Tambuwal, 2005; Bagudo *et al*., 2008). The result obtained for the total carbon content were 42.62±0.68 % and 35.6±0.13 % for undigested groundnut shell and for rice husk before digestion respectively. Never the less the value obtained revealed a high yield of carbon which boost biogas production.

XRF Analysis of CaO

The chemical composition of calcined snail shell catalyst was determined using XRF. The CaO was found to be the major composition with mass percent of 96.70wt % in addition to the trace number of other oxides such as Al_2O_3 SiO_2, SrO, and Fe_2O_3, were also observed, the composition of the catalyst is shown in appendix I.

XRF Analysis of ZnO

The chemical composition of zinc carbonate catalyst was determined using XRF. ZnO was found to be the major composition with mass percent of 69.28wt% in addition trace number of other oxides such as Al_2O_3 SiO_2, MgO, and Na_2O, were also observed, the composition of the catalyst is shown in appendix II.

FTIR Analysis of CaO

Appendix III: shows the FTIR spectra of raw and calcined snail shells and high peak were recorded at 650-4000 (cm-1) the sharp peaks at 1735 cm-1, 859 cm-1, 1468 cm-1 and 711 cm-1 were characteristics peaks of C-O stretch and bending mode of CaCo3. The broad band at 1420 cm-1 is characteristic absorption of CaO and sharf band at 3641 cm-1 is associated with OH stretching vibration mode of H_2O physio bed on the surface of the CaO. This is related to OH in Ca $(OH)_2$ the disappearance of moderate to weak signals and shifting of absorption band corresponding to CO32- after calcination of the snail shells confirmed the decomposition of $CaCO_3$ to CaO. IR absorption band in the raw and calcined spectra was comparably with the result of Ikbal *et al.* (2018).

XRD Analysis of CaO

The peaks that are specific pattern of CaO were observed at 2 θ = 33.2º, 37.3º, 64.1º, 67.30º. Peaks of $CaCO_3$ also appeared at 2θ = 23.0º, 29.4º and 39.4º. However, peaks of Ca $(OH)_2$, were observed due to interaction between CaO with water vapour in the air after sample decomposition. This phenomenon was also reported by Serris *et al.* (2011) the numerous of open pores exist inside the aggregate of CaO sample, the conversion of CaO into Ca $(OH)_2$ reaction takes place in each particle on the water vapour was easily circulate inside the porosity which is in favour of conversion of CaO into Ca $(OH)_2$. As shows in appendix IV

Analysis of Variance (ANOVA) for Biogas Yield

Table 4: shows the analysis of variance (ANOVA) for Digester D1, D3, and D4 for biogas production. P-values from the table indicate which factors are significant and which one are not significant. The factors with P-value greater than 0.05 is not significant while factors with P-value less than 0.05 is significant. Therefore Table 3.4 result shows that P-value for digester D1, D3, D4 is (0.040) indicate that there is significant difference. While R2 low predicted R2 (6.21) suggested that the model will not predict new observation.

Likewise, Table 5: shows the analysis of variance (ANOVA) for Digester D2, D5, D6 in the Anova table the P-value 0.065 for the digesters and indicates that all the mean are equal when Alpha value is set at 0.05 and predicted R2 (0.00) reveal that the model will not predict the new observation.

However, in Table 6: shows the analysis of variance (ANOVA) P-value (0.082) for the digester indicates that not all mean is equal when alpha value is set at 0.05, the predicted R2 (0.00%) reveal that the model will not predict the new observation. However, from the turkey comparison all the digesters D7, D8, D9 do not share a latter and their means are significantly different.

Result for the biogas produced from the groundnut shell and rice husk are presented as shown in Figure 1 to figure 9. The highest yield of biogas was obtained at week 2 for groundnut shell with (413 cm3) followed by rice Husk/groundnut shell with CaO (317cm3), groundnut shell with zinc oxide (200cm3), pure Rice Husk (238 cm3), groundnut with calcium oxide (I67.1 cm3), Rice Husk/groundnut shell (195.71cm3), Rice Husk/groundnut shell with ZnO (82.8 cm3), and Rice with CaO (78 cm). Going by this trend, it implies that the production potential of the sample differs and thus may be due to the lignocellusic content of the substrates. The content makes it not smooth for the bacteria to act upon in some of the substrates (Ismail *et al.,* 2016). Moreover, in a related work conducted by Abdulrazak *et al.,* (2014) the low yield of biogas from rice husk was ascribed to high lignin content and waxy nature of the substrate which prevented moisture to penetrate into them for a better anaerobic digestion.

However, low yield of biogas was observed at the first week of production for all of the substrates. The low yield of biogas production observed at first week may probably attributed to the fact that production depends on the activity of the methanogenic bacteria at first week the development of such bacteria was not completed due to the slower rate it has taken to grow.

Similarly, at 6th week of the production, a declined in the yield was observed and this may be due to the lower activity of the bacteria which result from shortage of ingredient needed for its survival Carbon/Nitrogen ratio (Ismail *et al.,* 2016)

Moreover, the overall yield of the substrates was found to be high for the groundnut shell having a yield of 1418 cm^3, Rice husk mixed with groundnut shell, 855.5 cm^3, groundnut shell with calcium oxide (658 cm^3), Rice husk/Ground nut shell (648cm^3), Rice husk (608 cm^3) and Ground nut shell with ZnO (1614 cm^3) respectively. Though the synthesized catalysts used in this research work were found to be ineffective in enhancing the yield of biogas production individually or combined based on the total cumulative gas produced by all the digesters, but they exhibit excellent behaviours in terms of early digestion and eliminating the lag phase period, this conforms the result obtained by Mu and Zheng (2011).

Table 1: Proximate analysis of the substrates before the digestion

Parameters	**Groundnut Shell**	**Rice Husk**
Total Solid (%)	70.0±0.06	60.0±0.15
Volatile matter (%)	80.5±0.045	71.5±0.031
Ash Content (%)	16.0±0.38	25.16±0.08
Moisture Content (%)	40.5±0.06	32.0±0.02
Crude Fiber (%)	14.5±0.16	27±0.41
Crude Lipid (%)	1.66±0.02	0.33±0.01
Carbohydrate (%)	58.04±0.03	41.2±1.26
Crude Protein (%)	7.29±0.007	3.26±0.011

Results are mean of triplicate determination ± SD

Table 2: Proximate analysis of digested substrates after digestion

Parameter (%)	Ground nut shell	Rice husk
Total solid	22.3±0.004 1.01±0.1	1.01.±0.1 1.01±0.1
Volatile matter	30±0.003	25.5±0.017 1.01±0.2
Total carbon	39.72±0.08	31.0±0.03
Ash content	10.0±0.14	19.4±0.09
Moisture content	17.0±0.02	

Results are mean of triplicate determination ± SD

Table 3: Elemental Composition of the Substrates

Properties	**Ground Nut Shell**	**Rice Husk**
Nitrogen content	1.16 ±0.003	0.53±0.005
Phosphorus content	4.14±0.0006	5.07±0.001
Potassium content	1.33±688	16±353
Carbon content	42.62±0.68	35.6±0.13
C/N Ratio	36.74	33.9

Table 4; Analysis of Variance (ANOVA) for Digesters (D3, D5, D6)

Sources	DF	Adj SS	Adj MS	F value	p-value
Digester	2	20549	10274	3.3	0.065
Error	15	46718	3115		
Total	17	67267			

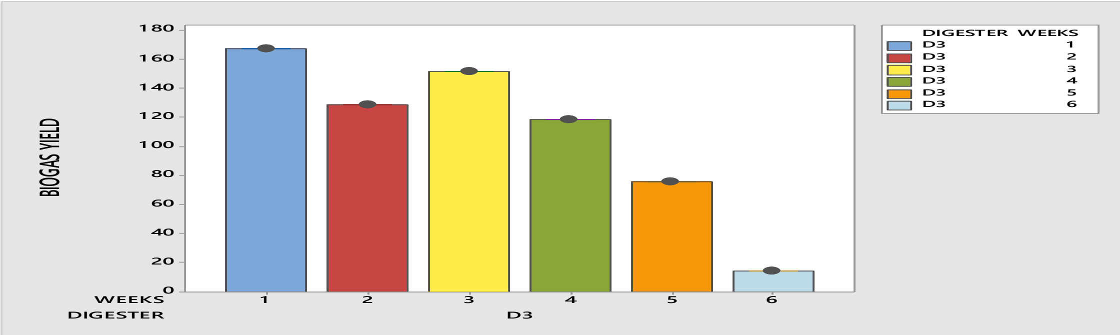

Figure 1: Weekly volume of biogas produced by groundnut shell mixed with CaO.

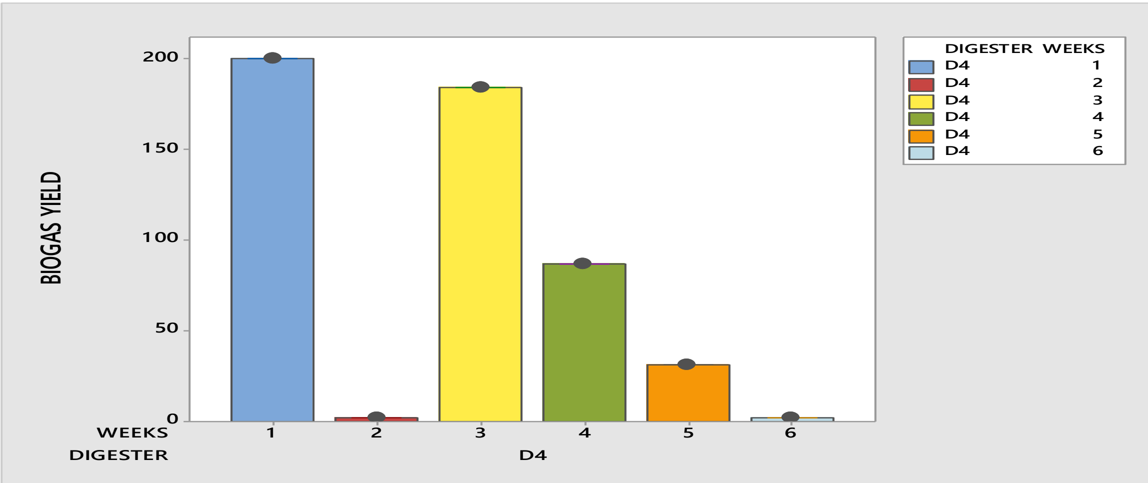

Figure 2: Weekly volume of biogas produced by groundnut shell mixed with ZnO.

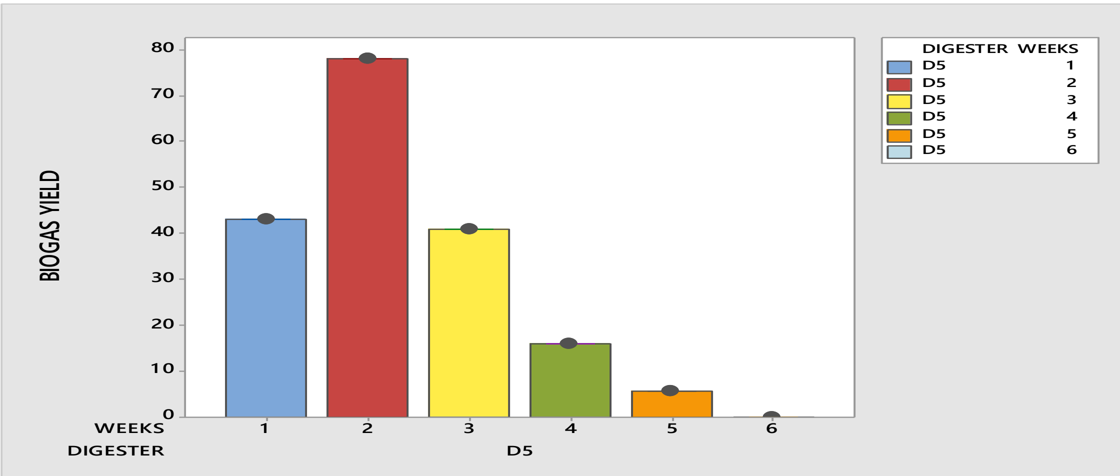

Figure 3: Weekly volume of biogas produced by rice husk mixed with CaO.

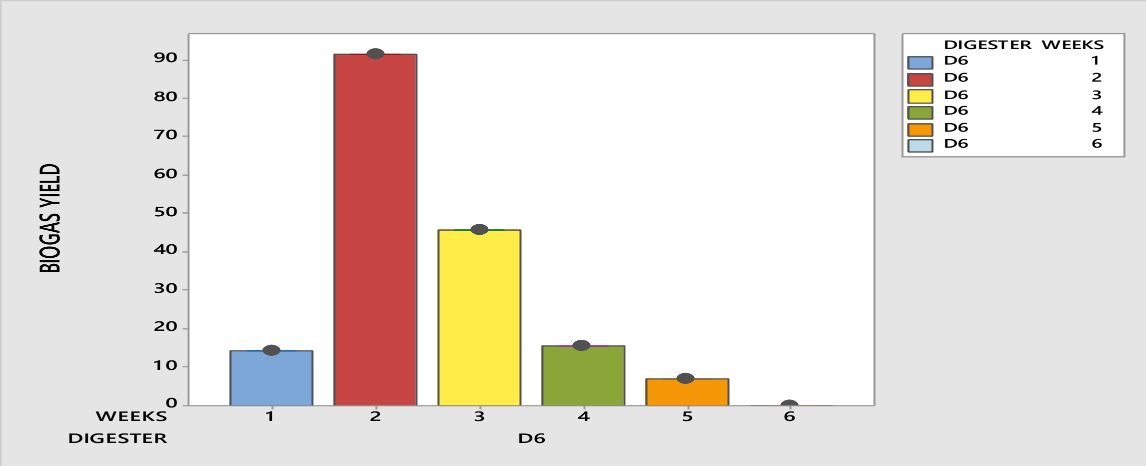

Figure 4: Weekly volume of biogas produced by rice husk mixed with ZnO.

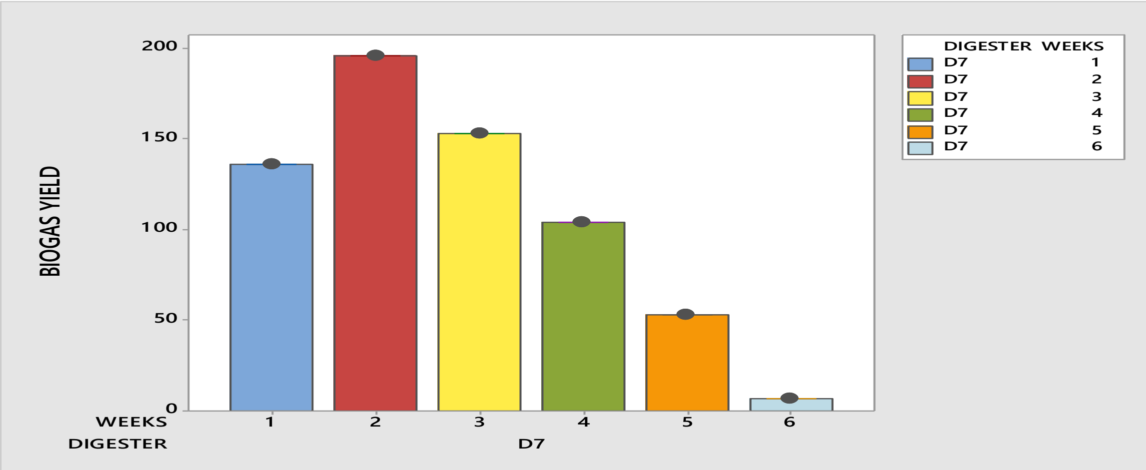

Figure 5: Weekly volume of biogas produced by rice husk mixed with groundnut shell.

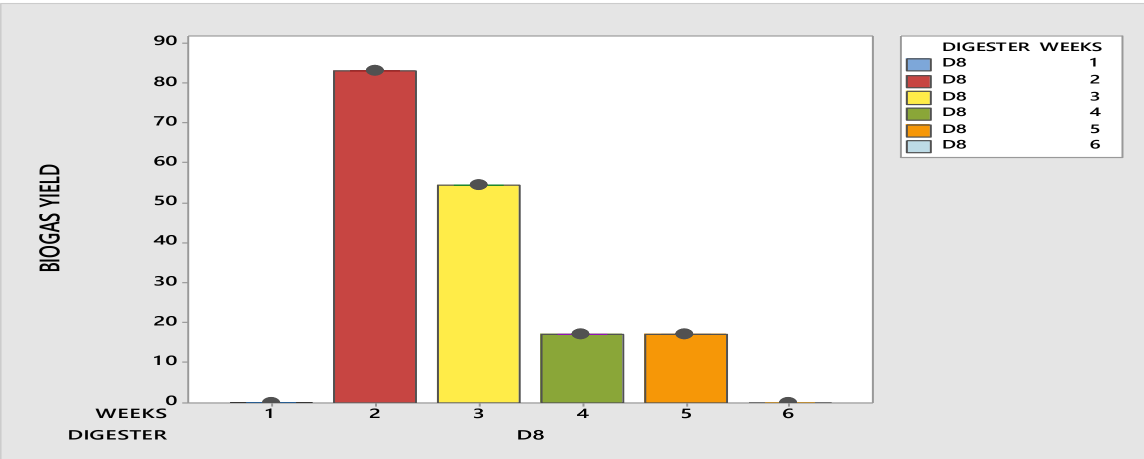

Figure 6: Weekly volume of biogas produced by rice husk mixed with g/shell and CaO.

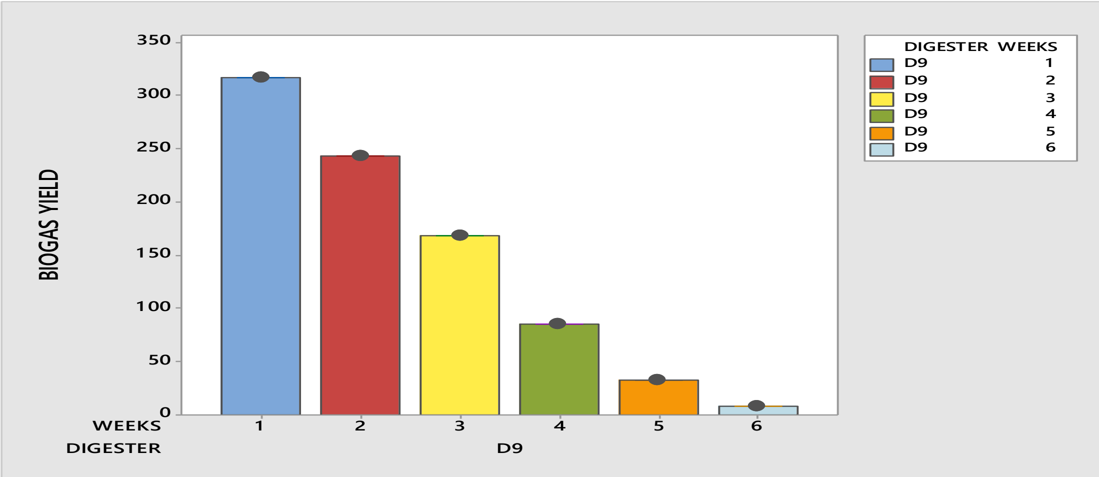

Figure 7: Weekly volume of biogas produced by rice husk mixed with g/nut shell and ZnO.

CONCLUSION

The result indicates the potentials of using ZnO and CaO has no much significance effect in biogas generation due to the quantity of biogas produced were high in substrates that catalysts were not used. However, proximate analysis of the substrate indicates viability of the substrate for biogas is encouraging as the groundnut shell has the highest moisture content (40.5 ± 0.06 %), and elemental composition of the substrates also proves that the slurry could be used as bio fertilizer as the nitrogen content was (1.16 ± 0.003) which is higher than rice husk (1.06 ± 0.05) and another element. On comprising groundnut shell is more viable as a candidate for biogas generation.

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