Comparative study of calorific values and proximate analysis of biogas from different feedstocks

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Abstract. Wastes used in this research paper were Orange and plantain peels with the addition of catalyst (clay). From the experiment carried out, the co-digestion of orange and plantain peels was the first to become flammable. The retention time was 66 days during which the ambient temperature and slurry temperature fluctuated between 22°C to 42°C which is within the range of mesophilic temperature. Orange peel, plantain peel and Orange/plantain became combustible on 64th, 55th, and 31st day respectively. Calorific values of the samples were 15,213.85 KJ/Kg, 18,309.35 KJ/Kg, and 16761.60 KJ/Kg respectively. Biochemical demands and chemical demand were obtained. The biogas produced was analyzed using biogas analyzer. The methane composition of the biogas produced were 70.70 %, 82.99 % and 77.85 % respectively. The proximate analysis of the samples was done. The Ash content was 0.35% for Orange peel, 0.55% for plantain peel and 0.41% for Orange/plantain due to the co-digestion process. The total viable count of the samples was also done.

Keyword: Mesophilic. Combustible. Biochemical. Flammable. Analyzer.

1.1 Introduction

Access to energy is frequently taken for granted in developed countries where resources are abundant, however the over-reliance on non-renewable sources such as coal, oil and natural gas is an environmental issue that will negatively impact future generations if sustainable energy...
sources are not found. Anaerobic digestion is a process by which almost any organic waste can be biologically transformed into combustible gas, in the absence of oxygen. Biogas is a renewable energy which typically refers to a mixture of different gases produced by the breakdown of organic matter in the absence of oxygen. Biogas can be produced from raw materials such as agricultural waste, manure, municipal waste, plant material, sewage, green waste or food waste. Most studies about biogas indicate that methane (CH₄) and carbon dioxide (CO₂) are the main components, where the ratio of methane ranged between 50 - 80% and the ratio of carbon dioxide range is 20 - 50%. Other components of biogas that may be found in small amounts (traces) are: Hydrogen (H₂), Nitrogen (N₂), Hydrogen Sulfide (H₂S), Carbon monoxide (CO), Ammonia (NH₃), Oxygen (O₂) and water vapor (H₂O). Waste, on the other hand, is generated in large quantities every day. A large fraction of municipal solid waste (MSW) is biodegradable, and it degrades naturally under anaerobic conditions in landfill to release methane into the atmosphere. However, organic wastes such as this MSW can be used effectively as an energy source. By diverting organic waste from landfill to anaerobic digestion (AD) plants, the methane produced can be harvested and eventually used for energy generation at homes. This strategy of recycling waste for energy will have two major benefits to the society: Reducing greenhouse gas emissions by cutting the methane released from landfill sites and cutting reliance on fossil fuels, as organic waste is renewable and readily available all around the world.

Biogas can be produced by anaerobic digestion with methanogen or anaerobic organisms, which digest material inside a closed system, or fermentation of biodegradable materials. Moreover, biogas production from organic waste has an important role as a waste management system. In addition, anaerobic digestion of organic waste enables recycling of nutrients because the sludge from the biogas process can be used as fertilizer, substituting for artificial fertilizers on land that can be used for growing crops and the gas can effectively be utilized for generation of power through a biogas-based power generation system after scrubbing the gas. It has been found that temperature variation, pH, retention time, addition of nutrient, loading rate, size of digester and concentration of total solid, etc. are some of the factors that affected the volume yield of biogas production.
A complex microbiological process lies behind the efficient production of biogas. Microbes that initiate the degradation consist of a large group of complex and differently acting microbes’ species notable, the methane producing bacteria. The whole biogas process can be divided into three steps hydrolysis, acidification and methane formation. Three types of bacteria are involved which are fermentative bacteria, acetogenic bacteria and methanogenic bacteria. In addition to the substrate, the microorganisms require a suitable environment in order to thrive and function. Examples of important environmental factors for growth are: temperature, pH, and solid concentration.

![Diagram of Anaerobic Digestion](image)

**Figure 1: Anaerobic Digester—Fundamental Steps**

- Methylo trophic methanogens that grow with methyl group containing substrates for eg. Methanol, methylamines, acetate
- The reaction for acetate is:
  \[ \text{CH}_3\text{COOH} \rightarrow \text{CH}_4 + \text{CH}_2 \]
- Organisms such as *Methanosarcina barkeri* grow on methanol or methylamines. Here, one fourth of the substrate has to be oxidised to CO₂ for reducing power generation.
  \[ \text{CH}_3\text{OH} + \text{H}_2\text{O} \rightarrow \text{CO}_2 + 6\text{H}_2 \]
  \[ 3\text{CH}_3\text{OH} + 6\text{H}_2 \rightarrow 3\text{CH}_4 + 3\text{H}_2\text{O} \]
  \[ 4\text{CH}_3\text{OH} \rightarrow 3\text{CH}_4 + \text{CO}_2 + 2\text{H}_2\text{O} \]
- Group 2 organisms produce methane directly from methyl groups and not via CO₂.
- Obligate chemolithotrophic methanogens do not contain cytochromes which are present in methylo trophic methanogens

Figure 1: Anaerobic Digester—Fundamental Steps
In order to function and grow, a microorganism needs access to an appropriate culture medium, i.e. a substrate. A substrate is "food" for the organism and must contain several different elements: a source of energy, electron acceptors, building blocks for building new cells, and different types of vitamins and trace elements (metals). With access to a substrate, microorganisms can metabolize, that is build up new cells (anabolism) and produce energy (catabolism) for this growth\textsuperscript{17}. Catalyst is a compound that increases the rate of a reaction by providing an alternative reaction mechanism for a chemical process. Catalysts can be homogeneous, heterogeneous or enzymatic in nature\textsuperscript{18}.

Co-digestion is a waste treatment method in which different wastes are mixed and treated together. It is also termed as “co-fermentation”. Co-digestion is preferably used for improving yields of anaerobic digestion of solid organic wastes due to its numeral benefits\textsuperscript{19} &\textsuperscript{20}. Temperature is a key component to the efficiency of anaerobic digestions. Being that organisms are responsible for the digestion process, it is vital to ensure that the entire process is kept within a certain temperature range to maximize reaction speed and organism livelihood. However, the range depends on the species of organism used. The two most widely used types of microorganisms are mesophiles, which undergo mesophilic digestion, and thermophiles, which undergo thermophilic digestion. Mesophiles are most efficient at moderate temperature ranges between 25-40\textdegree C. The words mesophile and thermophile are simply terms used to classify broad groups of organisms and just describe the temperature of their livable environment\textsuperscript{16\&21}. The constitution of different biogas production plants varies widely; hence, to determine the overall performance of a biogas production plant, not only biological performance but also energy performance must be determined and evaluated\textsuperscript{18\&22}. There are two modes of digester operation, batch and continuous, batch operation was used in this work\textsuperscript{23}. The main aim of this paper is therefore to: determine the calorific values of waste/feedstocks (Orange and Plantain peels); determine the percentage yield of biogas produced using gas analyzer, and determine the proximate analysis of the slurry.

\textbf{2.1 Materials and method}
2.1.1 Materials

The waste materials used for the study were: decayed plantain peels, decayed orange peels and clay. The following equipment were used in the study: Weighing balance, pH meter, mixing tank, three fixed dome biodigesters of volume 30254.72 cm$^3$ ($\approx$ 30 litres) (see figure 2), rubber hose and manual thermometer. All the chemicals used were analytical grade and purchased from Sigma Aldrich.

![Figure 2: Fixed-dome biogas digester](image)

2.1.2 Preparation of the slurry

Solid and granular wastes like decayed plantain peels and decayed orange peels were prepared in the ratio of 1:3, and the clay. This mixture was to combine feedstock with high caloric content and anaerobic microbes. The wastes were pounded until a soft pulpy texture was obtained, while 1 kg weight of clay was mixed with water thoroughly. The clay serves as a catalyst for anaerobic digestion. Orange peels charged (independently) was mixed with water in the ratio of 1:3 (6 kg of the waste was charged inside the digester with 18 kg of water) and fed into digester $X$ and Clay act as a catalyst to the orange peels. Plantain peels charged (independently) was mixed with water in the ratio of 1:3 and fed into digester $Y$. Orange peels/Plantain peels was mixed with water in the ratio of 1:3 and fed into digester $Z$.

Table 1: Mixing ratio and the temperature of samples
| Waste          | Mixing ratio (water/waste) | Ambient Temperature (AT) (°C) | Slurry Temperature range ST (°C) | Total volume of gas produced (litre) |
|---------------|--------------------------|-------------------------------|----------------------------------|-------------------------------------|
| Orange/Clay   | 3:1                      | 22 – 35                       | 25 – 42                          | 40.60                               |
| Plantain      | 3:1                      | 22 – 35                       | 25 – 41                          | 102.50                              |
| Orange/plantain | 3:1                  | 22 – 35                       | 25 – 42                          | 51.50                               |

### 2.1.3 Methods

In this study, biogas production was done in batch operation in which the slurry was added once in the digester for the whole duration of the process. The digester was provided with suitable arrangements for feeding, gas collection and draining residues. The digester was connected to a calibrated measuring cylinder with displacement arrangement to measure the volume of biogas produced. The biogas digester was built to maintain the anaerobic condition and the slurry was allowed to ferment anaerobically. The digesters and their contents were stirred thoroughly using an inbuilt stirrer and a little amount of the slurry was collected to measure the pH and the temperatures (ambient and slurry).

The downward displacement of water in the measuring cylinder was taken as a measure of the volume of biogas produced, and the volume of daily biogas production was recorded alongside the pressure and temperature for 66 days.

### 2.1.4 Measurement of parameters affecting biogas production

The pH of the slurry was determined electrometrically using glass electrode pH meter and sometimes with a litmus. The recorded pH from this experiment was within the range of 5.0 to 8.8 for orange, indicating that the orange waste is more of acidic than alkaline. The pH record for plantain is within the range of 6.0 to 9.2 and majorly above 7 indicating that the plantain waste is alkaline. Rate of gas generation is initially high and then, gradually, declines as the digestion approaches completion. Weighing balance was used to determine the weight of these wastes and water. Proximate composition of the slurries was carried out according to the method of AOAC\textsuperscript{24}.
The ambient and slurry temperature were both measured using a manual thermometer in degree Centigrade on daily basis to get the temperature of surrounding and that of the slurry. The ambient temperature for each day is the same for each waste (orange, plantain, orange and plantain combined). The recorded temperature for ambient and slurry was within the mesophilic range (22°C to 42°C). The flammability tests were carried out using a Bunsen burner connected to the discharge pipe of the anaerobic digester and cylinder.

**Table 2: Retention time, day of combustion and the volume of gas produced**

| Waste                   | Retention time (days) | Combustibility (days) | Volume of gas (Litres) |
|-------------------------|-----------------------|-----------------------|------------------------|
| Orange/Clay (Exp.X)     | 66                    | 64                    | 40.60                  |
| Plantain (Exp.Y)        | 66                    | 55                    | 102.50                 |
| Orange/Plantain (Z)     | 66                    | 31                    | 51.50                  |

**3.1 Characteristics of the biogas produced**

Each sample of biogas produced was analyzed using gas analyzer. A gas compressor is a mechanical device that increases the pressure of a gas by reducing its volume. The capacity of the compressor used was 1/5 horse-power. Biogas was scrubbed to refine it for use as a fuel using a scrubber which was connected to gas- chamber compartment of the digester. Each cylinder was able to compress biogas to 1.2 bars of pressure. Table 3 shows the percentage of the component of biogas produced from the three samples. From there, it was observed that the methane contents of Exp. X, Y, Z were 70.70 %, 82.99 %, and 77.85 %, respectively. It was observed that Exp. X yielded the highest value of poisonous gases (CO₂, H₂S, CO). Consequently, the biogas component for methane and other gases in X was the lowest which could possibly be the reason why X had the lowest calorific value. The percentage of methane is most important when we talk about the overall calorific value of the substrate/feedstock mixture. Higher the methane content, higher the calorific value.
Table 3: Percentage of the component of biogas produced

| Waste                  | Carbon dioxide (CO₂) (%) | Hydrogen sulphide H₂S (%) | Carbon monoxide (CO) (%) | Methane and other components (%) |
|------------------------|--------------------------|---------------------------|--------------------------|---------------------------------|
| Orange/Clay (Exp.X)    | 24.5972                  | 0.0028                    | 0.0002                   | 70.7000                         |
| Plantain (Exp.Y)       | 17.0000                  | 0.0025                    | 0.0001                   | 82.9955                         |
| Orange/Plantain (Z)    | 22.1449                  | 0.0010                    | 0.0001                   | 77.8541                         |

3.1.1 Determination of biochemical oxygen demand (BOD)

The biochemical oxygen demand (BOD) determination is an empirical test in which standardized laboratory procedures are used to determine the relative oxygen requirements of wastewaters, effluents, and polluted waters. The test measures the molecular oxygen utilized during a specified incubation period for the biochemical degradation of organic material and the oxygen used to oxidize inorganic material such as sulfides and ferrous iron. The test measures the molecular oxygen utilized during a specified incubation period for the biochemical degradation of organic material and the oxygen used to oxidize inorganic material such as sulfides and ferrous iron. The test measures the molecular oxygen utilized during a specified incubation period for the biochemical degradation of organic material and the oxygen used to oxidize inorganic material such as sulfides and ferrous iron. The test measures the molecular oxygen utilized during a specified incubation period for the biochemical degradation of organic material and the oxygen used to oxidize inorganic material such as sulfides and ferrous iron. The test measures the molecular oxygen utilized during a specified incubation period for the biochemical degradation of organic material and the oxygen used to oxidize inorganic material such as sulfides and ferrous iron.

Table 4: Biochemical Oxygen Demand for Samples

| Biochemical oxygen demand (BOD) (mg/l) | Orange | Plantain | Orange/plantain |
|---------------------------------------|--------|----------|-----------------|
| First Time                            | 35.20  | 25.60    | 30.40           |
| Second Time                           | 43.20  | 30.40    | 35.20           |
| Third Time                            | 27.20  | 16.00    | 20.80           |

3.1.2 Chemical oxygen demand (COD)

The chemical oxygen demand (COD) is an indicative measure of the amount of oxygen that can be consumed by reactions in a measured solution. Chemical oxygen
demand (COD) is a measure of the capacity of water to consume oxygen during the decomposition of organic matter²⁶.

Table 5: Chemical oxygen demand for the samples

| Chemical oxygen demand | Orange | Plantain | Orange/plantain |
|------------------------|--------|----------|-----------------|
| Volume of sample (V)   | 20.00  | 20.00    | 20.00           |
| Blank titre (Vₐ)      | 40.50  | 40.50    | 40.50           |
| Sample titre (Vₙ)     | 25.80  | 29.80    | 27.80           |
| COD (mg/l) = \((Vₐ-Vₙ)\times0.01\times1600/V\) | 117.60 | 85.60    | 101.60          |

Table 6: Calorific values of the wastes

| Calorific Values       | Orange | Plantain | Orange/Plantain |
|------------------------|--------|----------|-----------------|
| Weight of sample (g) = | 1.001  | 1.003    | 1.002           |
| Change in temperature (ΔT)= | 1.169  | 1.410    | 1.289           |
| Burnt wire = (B)       | 4.300  | 6.500    | 5.400           |
| \(\Phi=\text{B}\times2.3\) | 9.890  | 14.950   | 12.420          |
| Volume of alkali (V)   | 4.000  | 6.200    | 5.100           |
| Calorific value (kj/kg) = \(\frac{\Delta T-\Phi-V}{g}\) | 15213.850 | 18309.350 | 16761.600 |

Table 7: Proximate Analysis for Orange peels, Plantain peels and Orange/Plantain peels

| Parameter         | OP       | PP       | OP/PP    |
|-------------------|----------|----------|----------|
|                   | During digestion (%) | During digestion (%) | During digestion (%) |
| Nitrogen          | 0.06     | 0.17     | 0.13     |
| carbon content    | 1.60     | 3.99     | 3.19     |
pH | 6.00 | 7.5 | 7.25  
---|---|---|---
Ash | 0.35 | 0.55 | 0.41  
Moisture | 95.70 | 96.10 | 96.00  
Phosphorus | 0.06 | 0.17 | 0.13  
Volatile solid | 3.71 | 4.10 | 3.89  
Total solid | 1.28 | 0.60 | 0.92  
Potassium | 0.07 | 0.16 | 0.14  

Tables 8 shows that there were decreases in the values of microbial total viable count (MTVC) before digestion and after digestion. From the data obtained, it was observed that in samples (XYZ) orange peels, plantain peels and orange/ plantain peels as plant waste, have lower values of MTVC in the first and third analysis, while there was an increased value of MTVC in the second analysis and this could be attributed to the nature of the material, pre-treatment measures and large surface area. Also, during the anaerobic digestion many numbers of microbes acted on the waste to cause the degradation and the production of biogas. Through a process called biodegradation, microbes use nutrients and chemical substances found in the environment for their own survival.

Table 8: Microbial Total viable count (MTVC)

| Total Viable Count | Orange (cfu/ml) | Plantain (cfu/ml) | Orange/Plantain (cfu/ml) |
|-------------------|----------------|------------------|-------------------------|
| First Time        | 2.4×10^4       | 4.0×10^4         | 2.9×10^4                |
| Second Time       | 1.8×10^5       | 2.5×10^5         | 2.0×10^5                |
| Third Time        | 1.2×10^5       | 2.8×10^5         | 1.6×10^5                |

4.1 Results and Discussion
Table 1 shows the mixing ratio, temperature range and the total volume of gas produced. From the table, it was observed that the materials associated with anaerobic digestion perform best in the mesophilic temperature range and Exp. Y produced the highest biogas 102 litres. Figures 5 and 6 shows the variation of the volume of biogas produced and the cumulative biogas with retention time. From table 1 ambient temperature varies from 22°C to 35°C, while the slurry temperature varies from 25°C to 42°C, which were within the mesophilic range which is in close agreement with previous reports in journals 27 & 28. Table 2 shows the days of flammability for all the samples. Exp. X, Y, and Z became flammable on the 64th, 55th, and 31st day, respectively. The addition of clay optimised the methane production in Exp. X. It was established that clay was more active in quickening the gas combustibility day in Exp. X. It was also observed that when the percentage of carbon dioxide produced was higher in proportion than the methane gas, no combustibility took place. The volatile solids concentration in the digester determines the rate of gas production 29&30. Table 3 shows the percentage of the component of biogas produced from the three samples. From there, it was observed that the methane contents of Exp. X, Y, and Z were 70.7000%, 82.9955% and 77.854182% respectively. It was observed that Exp. X yielded the highest value of CO₂, 24.5972%, which delayed the combustibility of the gas produced. Most of the biogas is produced during the middle of the digestion, after the bacterial population has grown. It was observed that Exp. Y yielded the lowest value of poisonous gases (CO₂, H₂S, CO) which could possibly be why Exp. Y has the highest calorific value.

From tables 4 and 5 it was also observed that Exp. Y required the least of dissolved oxygen which possibly could be a factor that contributed to the high yield of biogas that is, at low BOD
the micro-organism proceeds with the digestion process. Both BOD and COD are commonly used to measure organic matter content.

Proximate analysis for each of the samples was carried out in the laboratory section of the National Center for Energy Research and Development (NCERD), University of Nigeria. During the digestion processes, samples from the digesters were collected and then characterized and synthesized for total solids, volatile solids, carbon, nitrogen, lignin and cellulose content. Moisture content was 95.70% for orange, 96.10% for plantain, and 96.00% for Orange/Plantain. Crude fiber has 0.20% for Orange peel, 0.40% for plantain, and 0.30% for Orange/Plantain. Nitrogen was 0.056% for Orange peel, 0.168% for plantain, and 0.126% for Orange/Plantain. Carbon Content was 1.60% for Orange peel, 3.99% for plantain, and 3.19% for Orange/Plantain. Phosphorus was 0.06% for Orange peel, 0.17% for plantain, and 0.13% for Orange/Plantain. Potassium was 0.07% for Orange peel, 0.16% for plantain, and 0.14% for Orange/Plantain. Table 7 shows the percentage of each parameter. We can deduce that discharged slurry can serve as a good biofertilizer since there was nitrogen, phosphorus and potassium (NPK), which are the main constituents of a fertilizer. This result is in line with the result obtained by 28&31. The common parameters such as pH and temperature of fresh substrate mixture and digested slurry were examined through the digital pH meter and thermometer. While total viable count determination for microbes in each sample were carried out in the pharmaceutical laboratory, University of Nigeria, Nsukka. Table 8 shows that the analysis of Microbial Total viable count (MTVC) done at the second time has many numbers of microbes acting on the substrate which quicken the biodegradation of wastes32. From figure 4 the study revealed that the pH decreased as the bacteria produced acids in the digester. The orange peels recorded more acidity than the co-digestion waste slurry which recorded a little acidity and alkalinity which is in agreement with the result reported by Dong and Pan 33. Clay acted as a catalyst which quickened Exp. X to produce biogas. The result supported the observation that acid concentration greatly affects the biogas production and is fairly in agreement with the result reported by Tabatabaei5 Figure 5 shows the variation of
pressure with retention time, Exp. Z (or 3) build the highest followed by Exp. Y (or 2) and then Exp. X (or 1)

Figure 3: Slurry temperature (degree) for the three samples

Figure 4: pH for the three samples versus retention time

Figure 5: Pressure of the gas versus retention time

Figure 6: Volume for the three samples versus retention time
5.1 Conclusion

This study investigated comparative study of calorific values and proximate analysis of biogas from different feedstock using anaerobic digester. The investigated substrates orange peels, plantain peels and clay as catalyst were anaerobically digested and biogas produced was measured and analyzed for methane and carbon dioxide content. The result shows that biogas produced from high caloric feedstock with high carbohydrates content such as plant waste combined with clay could yield biogas. Proximate composition of the slurries was carried out according to the method of AOAC. The result shows that sample with high volatile solid yielded the highest biogas. The result also shows that acid concentration greatly affected the biogas production. The three digesters set-up in the experiment anaerobically produced combustible gas which is renewable energy and can find many applications in our society.

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