



## Characterization of Biogas Digestate for Solid Biofuel Production in Uganda

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### ABSTRACT

In this study, suitability of digestate from anaerobic digestion of cow dung, pig dung, and human waste feedstock as a solid fuel for thermal applications was investigated. The digestate was obtained at different retention times from laboratory scale and household digesters and later characterized. Carbonized briquettes were produced from the digestate followed by their physico-chemical characterization and assessment for combustion and mechanical properties. Results of the proximate analysis of the digestate were: moisture content (6.1 to 18.3%), volatile matter (27.9 to 47.7%), ash (15.0 to 48.9%), and fixed carbon (9.1 to 17.1%). The ultimate analysis results for the digestate were: carbon (19.5%), hydrogen (3.3%), oxygen (20.8%), and nitrogen (7.0%). The developed briquettes showed a moisture content, volatile matter, ash, and fixed carbon in the range of 3.7 to 8.9%, 9.9 to 21.5%, 45.6 to 76.4%, and 8.2 to 22.8%, respectively. Their ultimate analysis results were: carbon (21.1%), hydrogen (1.3%), oxygen (1.8%), and nitrogen (1.9%). The briquette combustion properties revealed an ignition time, burning rate, and water boiling time of 5.35 seconds, 0.16 g/min, 31.1 minutes, respectively with higher and lower heating values of 14.87 and 7.88 MJ/kg, respectively. The briquette ash compounds were sodium 1718.5 ppm, potassium 20017.8 ppm, copper 6.12 ppm, cadmium 1.22 ppm, and lead 25.6 ppm. TGA/ DTG analysis indicated high mass loss rates at 105°C and maximum energy release between 600 and 900°C. The mechanical compressive strength was between 19 and 50 MPa, with bulk density between 1.82 and 2.02 g/cm<sup>3</sup>. Thus, the briquettes from the biogas digestate demonstrate potential for domestic thermal applications in Uganda.

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### Statement of Novelty

There are few notable studies devoted on the conceptualization of valorization of biogas digestate, a byproduct from the anaerobic digestion processes. Moreover, in Uganda, there is no reported study on the alternative use of biogas digestate as an energy resource. Thus, the novelty of the present study is to ascertain the possibilities for the valorization of biogas

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digestate towards production of carbonized briquettes for thermal applications, going beyond its usage as a fertilizer in agriculture, composting, and/or disposal in landfills.

## Introduction

Anaerobic digestion is one of the renewable energy technologies that has been recognized as a cost-effective bio-chemical technology for converting various bio-wastes into a primary product biogas [1] and digestate as a byproduct. Biogas from waste and several other biomass sources plays a critical role in ensuring that the use of renewable energy is met. It is a renewable energy source that can replace conventional fuels such as oil, gas, and coal to produce heat and power [2]. During anaerobic digestion, the processed substrate from which the biogas has been extracted is released from the digester in form of a slurry referred to as digestate. This product is rich in nutrients and is traditionally used as a bio-fertilizer for agriculture [3]. However, the quantity of digestate produced often exceeds the consumption ability of the surrounding farmland, and digestate transportation from the point of surplus to distant farmlands is sometimes not economically feasible [1]. Additionally, because of the limited time of substrates in the digester and incorporation of stirring systems for maintaining uniform conditions in the digester, the digestate may in some cases come out of the digesters with a percentage of biomass still undigested.

A number of studies have investigated the alternative use of biogas digestate besides land fertilization. According to Černá et al. [4], it is possible to dry the separated solid part and compress it into densified biomass energy forms such as briquettes or pellets which can be used for several thermal applications. Moreover, the variability in the substrates and operational conditions in biogas digesters implies that the production of substrates with varying characteristics and consequently different requirements for densified biomass fuel production fall within these characteristics. This creates the need to identify several fuel capabilities of the digestate and procedures to be followed during conversion. In a study conducted by Brunerova et al. [5] to determine the mechanical durability of briquettes from digestate in different storage conditions, briquettes were discovered to be a secondary product of proper waste management and effectively a modified fertilizer. Further, they were found to be a suitable fuel with an average mechanical durability of 99.44%. Similarly, Černá et al. [4] conducted a study to evaluate the dependence of the durability of digestate briquettes and sorption properties on represented particle sizes. It was revealed that the rate of abrasion of digestate briquettes depended primarily on the material properties of the briquettes where the size and shape of particles in the uncompressed digestate had an influence on the durability of the briquettes.

According to Kratzeisen et al. [6], digestate can be recommended as a fuel for combustion due to its calorific value, ash properties, and the low emissions which allow its use in solid biomass combustion units. However, it is required to cover a broader range of digestates and combustion techniques. It is reported from a previous study conducted in 24 districts of Uganda that there is an increasing number of biogas digesters in the country (82% being fixed dome type, 9% floating drum and 7% tubular) [7]. This implies a simultaneous increase in the digestate quantities being produced from the digesters. The digestate has commonly been used as a fertilizer to enhance agricultural production in many parts of the country. However, the excess digestate is not properly managed and has resulted into wastage through release into the environment especially during dry seasons when farm activities are at stand still. While one of the solutions to this is to harness the remaining available energy in the digestate through production of briquettes before releasing the liquid component to be used as fertilizer, there is limited knowledge of the process requirements for production of the same in Uganda. This is based on the fact that the different substrates and operating conditions give rise to digestate with varying properties which require different pretreatment conditions [6] and hence the need for additives for briquette production. Thus, this research therefore sought to determine the process requirements for producing briquettes from digestate after anaerobic digestion. Moreover, to evaluate the suitability of the digestate as a solid biomass fuel for thermal applications in Uganda.

## Materials and methods

### *Digestate collection and preparation*

Different digestate samples were collected after mesophilic digestion from laboratory scale and household digesters. The samples were collected from two different laboratory scale digesters at different retention times. One in intervals of 5 days i.e., 15, 20, 25, 30, and 35 days and the other in intervals of 1 day i.e., 15, 16, 17, 18 and 19 days. One sample was collected from each digester. The substrates were cow dung obtained from zero grazed Friesian cows. The cow feeds were mainly pastures, stalks, leaves, cassava, potato, and banana peelings. The two digesters were operated at the same time with one operating under batch feeding method while the other under continuous feeding method. Ten digestate samples were equally collected from the different household digesters in Mityana and Mbarara districts that are located in the central and western regions of Uganda, respectively. The cow dung sources were from the continuous grazed long horned Ankole cattle that mainly feed on pastures, stalks, and leaves. The ten samples collected were intended at having representative samples of digestate. This was mainly due to the differences in the animal breeds, their age, type of feeds, limited time of substrates in the digester and the incorporation of stirring systems for maintaining uniform conditions in the digesters. The digesters that were near and easily identifiable were visited to represent the entire digester population in the region. The farmers that were readily approachable had their biogas digesters included as part of the population samples. Table 1 presents the

**Table 1**  
Operating parameters of the digesters.

Samples	Type of reactor/ digester	Digester capacity	Feed stock	Feeding rate ( $\pm 200$ g)	Retention time (days)
<b>Batch feeding method (laboratory scale)</b>					
1	Floating drum	0.24 m <sup>3</sup>	Cow dung	20 kg/ batch	20
2	Floating drum	0.24 m <sup>3</sup>	Cow dung	20 kg/ batch	25
3	Floating drum	0.24 m <sup>3</sup>	Cow dung	20 kg/ batch	30
4	Floating drum	0.24 m <sup>3</sup>	Cow dung	20 kg/ batch	35
5	Floating drum	0.24 m <sup>3</sup>	Cow dung	20 kg/ batch	40
<b>Continuous feeding method (laboratory scale)</b>					
6- 10	Floating drum	0.24 m <sup>3</sup>	Cow dung	10 kg/ day	12
<b>Household digesters (continuous feeding method)</b>					
11	Floating dome	9 m <sup>3</sup>	Cow & pig dung	25 kg/ day	13
12	Floating dome	6 m <sup>3</sup>	Cow & human waste	20 kg/ day	15
13	Floating dome	6 m <sup>3</sup>	Cow dung	20 kg/ day	12
14	Floating dome	6 m <sup>3</sup>	Cow dung	25 kg/ day	14
15	Floating dome	9 m <sup>3</sup>	Cow dung	30 kg/ day	13
16	Floating dome	9 m <sup>3</sup>	Cow dung	30 kg/ day	12
17	Floating dome	9 m <sup>3</sup>	Cow dung	20 kg/ day	13
18	Floating dome	9 m <sup>3</sup>	Cow dung	25 kg/ day	15
19	Floating dome	9 m <sup>3</sup>	Cow dung	30 kg/ day	13
20	Floating dome	6 m <sup>3</sup>	Cow dung	20 kg/ day	14

operating parameters of the digesters maintained at temperatures between 25 and 30°C from which the digestate samples were collected.

In the preparation of the digestate, a laboratory experiment was conducted in both the batch and continuous feeding methods in a 0.24 m<sup>3</sup> capacity temporary digester. Also, ten samples of digestate were collected from the different household biogas digesters that were operating under the continuous feeding method. In the batch feeding method, 100 kg of fresh cow dung was collected from the kraal. The dung was measured using a Salter weighing scale of maximum measurable limit of 100 kg ( $\pm 200$  g). The cow dung was later diluted with tap water in the ratio of 1:1. The mixture was thoroughly stirred with a clean wood to achieve homogeneity. The fibers were screened through a sieve mesh of size 0.5 cm x 0.5 cm to remove foreign materials. The cow dung - water mixture was slowly fed into the 0.24 m<sup>3</sup> capacity digester occupying 0.2 m<sup>3</sup> of the volume thereby undergoing mesophilic digestion. 0.04 m<sup>3</sup> of the digestate was removed after 15 days as the first batch. This was an approximated volume selected to give enough digestate for characterization and briquetting. An equal volume of the digestate was removed for the next four batches in intervals of 5 days i.e., 20, 25, 30 and 35 days, making a total of 5 samples.

In the continuous feeding method, the above procedure was repeated but with 0.04 m<sup>3</sup> of digestate being removed after 15 days which was the second day after the start of biogas production. The sampled volume was approximated to give enough digestate for characterization and briquetting. The other four samples were removed after intervals of 1 day i.e., 16, 17, 18 and 19 days. Ten samples of digestate were collected from household biogas digesters of 6 m<sup>3</sup> and 9 m<sup>3</sup> capacities, respectively. Each sample contained 15 kg of solid digestate with 50% moisture content. Out of the 10 samples, there was one sample that was obtained from a digester whose substrates were a mixture of cow and pig dung, while the other being a mixture of human waste and cow dung. The other eight samples were purely from cow dung.

### Dehydration of Digestate

The digestate samples that were removed from the batch and continuous feeding methods were separated into solids and liquids using 75-micron filter. The solid samples were sun dried for 7 days to achieve a moisture content of 20% [7]. The moisture content was measured using a moisture meter (Humimeter BLL PCE Instruments, FL, USA). The digestate samples from the household digesters were skimmed from the surfaces of the slurry tanks. They had a lower moisture content that did not require separation. They were equally sundried for seven days to achieve drying to the resultant reduction in the volume (i.e., weight). The dry matter was then packed for characterization and carbonization.

### Characterization of Digestate

#### Proximate Analysis

An Eltra Thermostep Thermogravimetric analyzer was used to determine the proximate analysis of the digestate. The parameters determined were moisture content, volatile matter, analytical ash, and fixed carbon with errors of  $\pm 0.001$ ,  $\pm 0.001$ ,  $\pm 0.005$  and  $\pm 0.001$ , respectively.

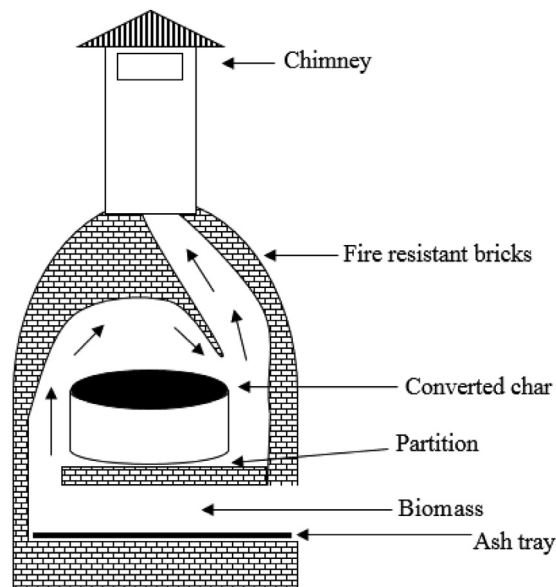


Fig. 1. Carbonizer.

#### Ultimate Analysis of digestate

Ultimate analysis to determine the carbon and nitrogen content in the digestate was done based on the ASTM standards. The ASTM standards D3179 using the Kjeldahl method and D3178 using the Liebig method were used to determine the nitrogen and carbon contents, respectively. Further, the hydrogen and oxygen contents were determined through correlations for predicting the elemental compositions. This was based on the proximate analysis results with average absolute errors of 4.79% and 3.4%, and bias errors of 0.15% and 0.49%, respectively. The following relations based on fixed carbon (FM), volatile matter (VM) and ash content (ASH) of raw digestate and briquettes were used.

$$H = 0.059FC + 0.060VM + 0.010ASH \quad (1)$$

$$O = 0.340FC + 0.469VM + 0.023ASH \quad (2)$$

[8]

#### Development of Briquettes from Digestate

##### Carbonization

The dried digestate sample weights were determined using an Electronic Digital Balance (model 11-D0630/6) followed by loosely packing into a wood fired carbonizer of base size 500 × 500 mm. Thereafter, pyrolysis started as the biomass burned slowly with the heat spreading to the digestate at temperatures between 350°C to 500°C [9]. The carbonizer shown in Fig. 1 was constructed with fire resistant bricks, one foot above the ground with stainless steel insulated door.

The temperature of the carbonizer was monitored using a 62 Max Mini Infrared Laser Thermometer, Washington, USA. The temperature was maintained between 350 and 500°C for 30 minutes after which the biomass supply to the carbonizer was cut off. The char was removed and cooled by sprinkling water followed by sun drying at an average ambient temperature of 30°C for 3 days to 15% moisture content wet basis [10]. The drying period was determined by the stable weather condition which favored the drying in 3 days. The samples were then weighed again. Bigger char pieces were reduced by milling until they could pass through a sieve size of 1 mm. The process was repeated for all the samples.

##### Binder Preparation and Mixing

Cassava starch was used as a binder due to its high viscous bonding media [10]. Moreover, it is commercially available and cheap. In the binder preparation, cassava flour was mixed with water in the ratio of 20, 40, 60, and 80 g of cassava flour for every 1000 g of digestate biochar with water being at room temperature followed by stirring to achieve a uniform mixture. The starch was then gelatinized by cooking in water at temperatures between 55 to 85°C to make porridge followed by cooling. Char powder was then mixed with porridge in a basin until all the charcoal was coated. To confirm whether the material was ready for briquetting, a handful of the mixture was crushed in the palm to make a rough ball. The balls that fell apart when released needed more binder. The balls that kept the shape of the hand were ready to make briquettes [5].

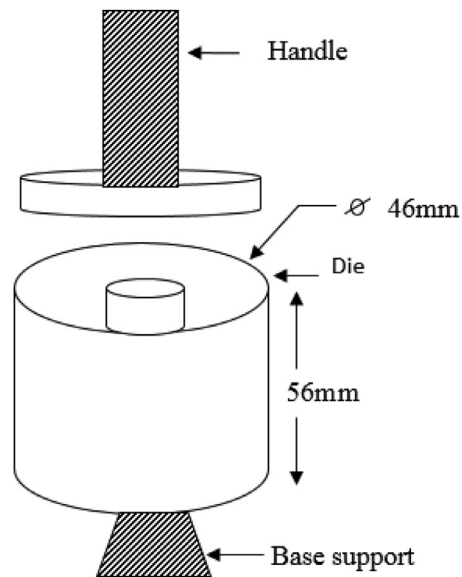


Fig. 2. Stainless steel briquetting mold.

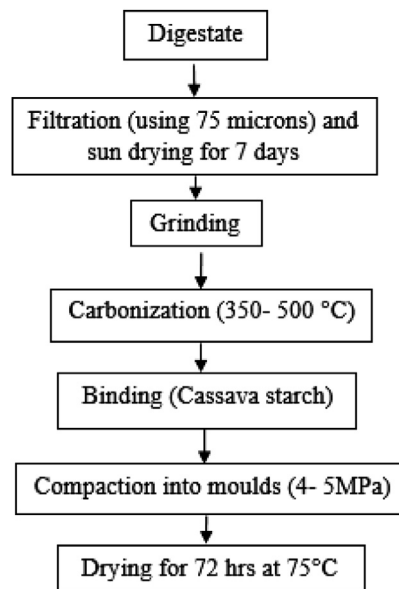


Fig. 3. Digestate conversion into briquettes [11].

### Briquetting and Drying

A hand-held single die cylindrical stainless-steel mold (Fig. 2) with the size 56 mm × 46 mm was used to prepare the briquettes.

In the briquetting process, a handful of charcoal porridge mixture was squeezed into the mold until it flushed with the top surface. The round top was used to enclose and compress the material manually at a pressure ranging between 4 - 5 MPa. The procedure to prepare more briquettes was repeated after which they were sun dried for 3 days at an average ambient temperature of 30°C. The briquettes were weighed after drying. It was observed that the briquettes had no visible cracks indicating that the amount of cassava starch was enough to evenly coat the surface. The procedure used for conversion of the digestate into briquettes is shown in the Fig. 3.

### Characterization of the Developed Briquettes

Characterization of the briquettes was conducted through proximate and ultimate analysis using an Eltra Thermostep Thermogravimetric analyzer. The proximate analysis included moisture content, ash content, fixed carbon, and volatile matter content while the ultimate analysis included determination of carbon, hydrogen, oxygen, and nitrogen with average bias errors of 0.25%, 0.34%, 0.19%, and 0.12% respectively. Also, a Flash 2000 Elemental Analyzer was used for ultimate analysis of the briquettes.

#### Evaluation of the Combustion Properties of Developed Briquettes

##### (a) Ignition Time

To determine the ignition time, the briquette samples were ignited at the edge of their bases with a bunsen burner. The burner was left on until all the briquettes were well ignited and had entered into their steady state burning phase. Using a stopwatch, the time taken for each briquette to ignite was recorded.

##### (b) Burning Time/ Burning Rate

Using match sticks, the briquette samples were ignited one-by-one. The time it took to burn a certain number of briquettes was recorded. At the end of approximately 30 minutes most of the briquettes were about half of their initial size and if disturbed would break apart easily. The only briquettes that still had solidity at their center were those that were obtained from a mixture of cow dung and human waste. After 75 minutes, the briquettes that were poked fell apart indicating that they were completely burned to ashes.

##### (c) Water Boiling Test

This was carried out to compare the cooking efficiency of the briquettes obtained at the different retention times. It measured the time taken for each set of briquettes to boil an equal volume of water under similar conditions. Initially, the briquettes were sprinkled with a little amount of kerosene followed by ignition. 100 g of each briquette sample was used to boil 250 mL of water using small stainless cups and a domestic briquette stove [9,12]. The temperature reading was taken after every minute with the Infrared Laser Thermometer until the water started boiling. During this test, other fuel properties like the burning rate and specific fuel consumption were also determined including the observation of the smoke level. The time taken by each sample to burn to ashes was monitored using a stop watch. The specific fuel consumption during the water boiling test was calculated using the equation

$$\text{Specific fuel consumption} = \frac{\text{Mass of fuel consumed (kg)}}{\text{Total mass of boiling water (litre)}} \quad (3)$$

[13]

#### Determination of the Calorific Values

A Gallenkamp Autobomb calorimeter was used to determine the heating value of the briquettes. The net calorific value (NCV) was calculated from gross calorific value (GCV) according to the water content as below:

$$\text{NCV} = \text{GCV} - \text{Water} \times 24.41 \quad (\text{MJ/kg}) \quad (4)$$

where NCV corresponds to the net calorific value of the dry sample with an error of  $\pm 1\%$ , GCV was the gross calorific value of the dry sample, and water was the percentage of the total water.

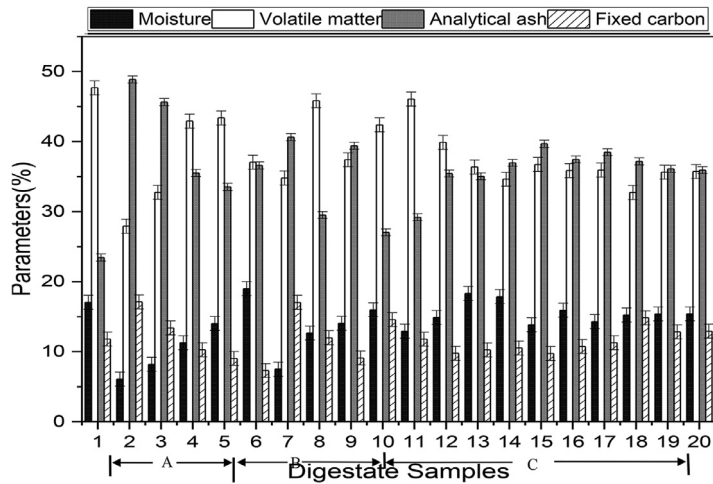
#### Ash compositional analysis

The 20 briquette and digestate samples were ground using a laboratory ceramic motor and pestle to powder for homogenization and later burnt to ashes in a High-temperature Box Muffle Furnace-27L (Model: KJ-M1700-27LZ). This was followed by mixing with an aqua regia solution. The resultant solution was analyzed using an Atomic Absorption Spectrophotometer (Agilent 240FS AA). The parameters analyzed were Na, K, Cu, Cd, and Pb with absolute errors of 0.01% for each element.

#### Mechanical properties of the briquettes

##### (a) Drop test

The drop test method was performed to determine the compaction reliability of the briquettes. Initially, the briquettes were raised up to a height of 2 m followed by dropping them onto a concrete floor. Thereafter, the ratio of the weight after dropping to the weight before dropping was recorded as the drop strength. After each drop, the amount of the remaining briquette fraction (over 10 mm in diameter) was determined where a 10 mm sieve was used to filter fractions less than 10 mm.



**Fig. 4.** Proximate analysis of the digestate samples. (A- Batch feeding method (laboratory scale), B- Continuous feeding method (laboratory scale), and C- Household digesters).

### (b) Bulk density

In the determination of the bulk density, the briquette samples were initially weighed using an Electronic Digital Balance of maximum weight of 8200 g ( $\pm 0.1$  g). Also, the volume of the briquettes was equally determined. Since the briquettes were cylindrical, geometric measurements were done followed by determining their volume using Eq. (5):

$$V = \frac{\pi dh}{4} \quad (5)$$

The bulk density was then determined using Eq. (6):

$$\rho = \frac{W}{V} \quad (6)$$

where;

- $\rho$  = bulk density of the sample ( $\text{g}/\text{cm}^3$ ),
- $W$  = average weight of the briquette (g),
- $V$  = average volume of the briquette ( $\text{cm}^3$ ),
- $d$  = diameter of the briquettes (cm),
- $h$  = height of the briquettes (cm).

### (c) Compressive strength

The compressive strength was measured by breaking the cylindrical briquettes in a M500- 25 kN universal compression testing machine. A vernier caliper (Mitutoyo 530-119) was used to measure the length and diameter of the samples. Thereafter, a sample of the briquette to be tested was placed horizontally in the compression test fixture followed by applying a load of 10 N at a constant rate of 1 mm/min until the briquette failed by cracking.

### Thermal Analysis

The briquettes thermal behavior during combustion was analyzed using thermogravimetric analysis under an oxidizing atmosphere (synthetic air) at a temperature of 750°C and a heating rate of 16°C/min.

## Results and discussion

### Characterization of the Digestate

Fig. 4 presents the results of the proximate analysis of the raw digestate samples. As observed, the moisture content varied between 6.1 and 19.0 wt%, with the highest value due to the fibrous nature of the digestate samples where they tended to retain moisture for longer periods of time [13]. Furthermore, the volatile matter ranged between 27.9 and 47.7 wt% resulting into production of briquettes that were easily lit with higher emissions. Analytical ash content on the dry basis varied from 22.8 to 47.9 wt%, implying that the digestate contained a high ash content. Moreover, the ash content is essential for the choice of appropriate combustion equipment. Thus, fuels with low ash content are desirable. The fixed carbon varied between 11.8 and 17.1 wt% which implied that the carbon that remained in the digestate was moderately small.

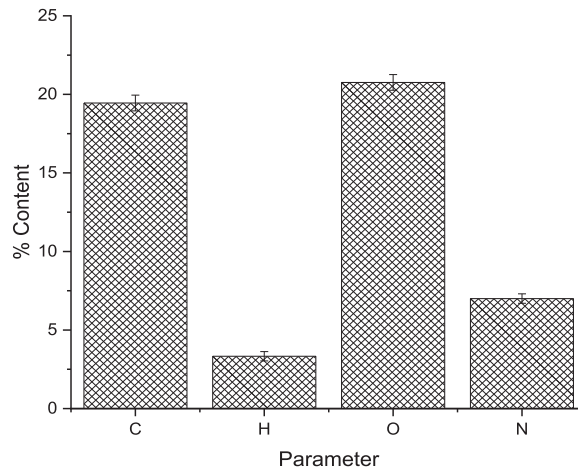


Fig. 5. Ultimate analysis of the digestate samples.

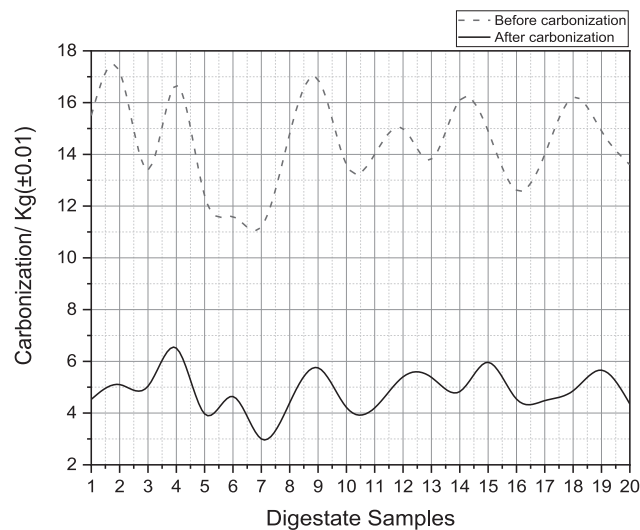


Fig. 6. Weights of the digestate before and after carbonization.

Fig. 5 shows the results of the ultimate analysis of the digestate samples. As can be seen, carbon and nitrogen were 19.5 and 7 wt%, respectively. Further, the hydrogen and oxygen content in the digestate were 3.3 and 20.8 wt%, respectively. The carbon content in the digestate was sufficient for production of a better quality of briquettes for thermal applications. Moreover, the higher the carbon content, the higher the calorific value [14]. The nitrogen content in the digestate may be due to the plant materials having alkaloids, chlorophyll, and other porphyrins that contain nitrogen in the cow dung feedstock. This resulted into better combustibility upon conversion of the digestate into solid char. Furthermore, the low nitrogen content in the digestate is welcomed because it is associated with minimal release of nitrogen oxides [15] into the atmosphere, an indication of the expected low pollution levels during combustion of the solid fuel extract. Also, the presence of hydrogen and oxygen in the digestate is significant for the improvement in the combustibility of the solid fuel extract hence easier burning.

#### Development of Briquettes from Digestate

##### Carbonization

The weights of the digestate before and after carbonization are presented in Fig. 6. It is observed that there was a loss in weight of the digestate after the carbonization process. The weight loss can be explained by the escape of the volatile matter through emissions during carbonization. Further, it is also possible to attribute the weight reduction due to the pre-treatment process of drying prior to feeding of the raw digestate into the carbonizer. As a result, the produced briquettes were lighter and took a few seconds to be ignited since most of the volatile matter was lost. Moreover, carbonization temperatures ranging from 350 to 500°C are expected to result into a lower volatile matter content in the resultant solid char.



Fig. 7. Samples of the developed briquettes.

Thus, to achieve a higher yield of carbon in the char, it is significant that a higher carbonization temperature is considered [16]. According to Lubwama and Yiga [16], the carbonization of raw coconut shells ranged between 400 and 850°C. In this process, volatiles amounting to 70% of the mass of coconut shells on dry weight basis were released to the atmosphere, yielding 35% of coconut shell mass of char.

#### Briquetting and Drying

Fig. 7 shows the briquette samples developed using a hand-held single die cylindrical stainless-steel mold. It was observed that the weights of the briquettes differed due to the variation in the briquetting pressure that ranged from 4 to 5 MPa. The average weight of the briquettes was 41.1 g. It was observed that the briquettes had no visible cracks indicating that the amount of cassava starch binder was enough to evenly coat the surface. Also, they had a coarse appearance with a firm and stronger surface finish attributed to the presence of unburned matter in the coarse grain fractions of the digestate. Moreover, they had small pores which is an indication of the low pressures exerted during their formation. This shows that the micro spaces between the feedstock materials were not completely filled with the binder material. This bonding is characterized by the short range forces due to the intermolecular hydrogen bonds between amylose and amylopectin components of starch, van der Waals' forces, and mechanical locking [17]. Also, the fibrous particle structure in the digestate strengthened up the mechanical bonding in the briquettes [18].

#### Characterization of the Digestate Briquettes

Fig. 8 shows the proximate analysis results of the digestate briquette samples.

As can be observed in the Fig. 8 above, the analytical ash content had values ranging between 55.0 and 79.3 wt%, an indication that the briquettes contained a high inorganic material content. Furthermore, ash is an impurity that does not burn as it contains salts that may be compounds of sodium, vanadium, calcium, magnesium, silicon, iron, aluminum, and nickel. Moreover, excessive ash in fuels can cause fouling deposits in the combustion equipment which causes damage to the refractories at high temperatures thereby giving rise to high temperature corrosion. Also, it reduces handling and burning capacity, increases handling costs, and affects combustion efficiency [19]. According to Falemara et al. [18], ash is expected to have values for commercial fuels ranging from 0.60 to 9.80%, energy crops from 1 to 9.60%, cereals from 1.80 to 4.80%, and industrial waste from 0.40 to 22.60%.

The low amount of moisture in the digestate briquettes that ranged between 3.70 to 8.90 wt% is attributed to the removal of moisture from the bound char due to compression during briquetting process. Moreover, this moisture content in the briquettes could also be due to the storage conditions under room temperature for two weeks prior to characterization. Furthermore, moisture content facilitates starch gelatinization, protein denaturation, and fiber solubilization during briquetting. This means that feedstock with high moisture content is expected to lower the compressive strength and bulk density of the final briquettes [13]. The fixed carbon content ranged between 8.25 and 22.80 wt% which gives an indication

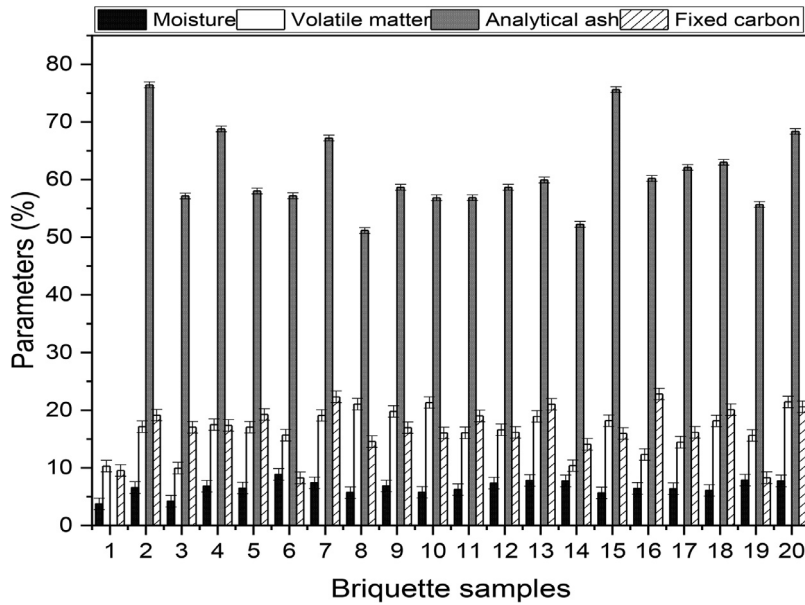


Fig. 8. Proximate analysis of the various compositions of briquette samples.

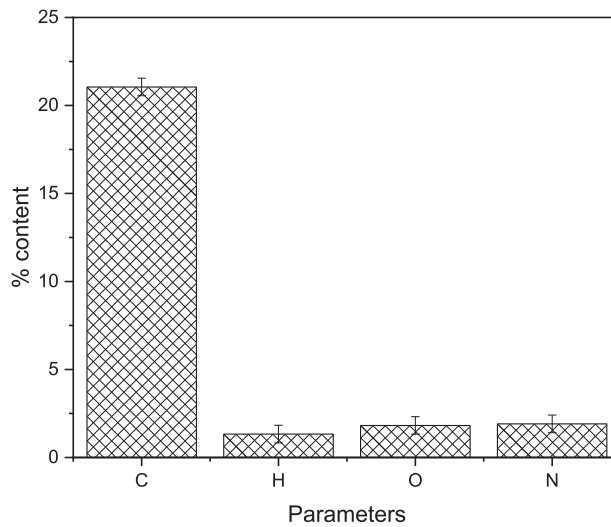


Fig. 9. Ultimate analysis of the briquette samples.

of the proportion of char that remained in the final briquettes. Further, the char consists of mostly carbon but can also contain some hydrogen, oxygen, sulfur, and nitrogen which is not driven off with the gases [20]. According to [19], it is expected that the high fixed carbon and its smokeless flame will enhance the heating value and combustion duration of the briquettes.

The volatile matter content ranged between 9.90 and 21.40 wt%. Moreover, a good quality and efficient fuel briquette is dependent on a low volatile matter and ash content with a high fixed carbon content. Also, the volatile matter is an index of the gaseous fuels present in the fuel and affects its combustibility. The typical range of volatile matter in most fuels is 20 to 35 wt% [1]. Furthermore, it has been observed that briquettes with higher volatile matter possess higher specific heat of combustion and lower fixed carbon. In contrast, the higher the volatile matter of briquettes, the higher the amount of emissions during burning. Hence, this implies that a low volatile matter content is required for good quality briquettes [19].

Fig. 9 presents the results of the ultimate analysis of the digestate briquettes. It can be observed that carbon, nitrogen, and oxygen are the main components of solid fuels. During combustion, carbon and nitrogen react in an exothermic reaction to generate CO<sub>2</sub> and H<sub>2</sub>O. Although internal oxygen forms part of the fuel fraction, only carbon influences the higher heating

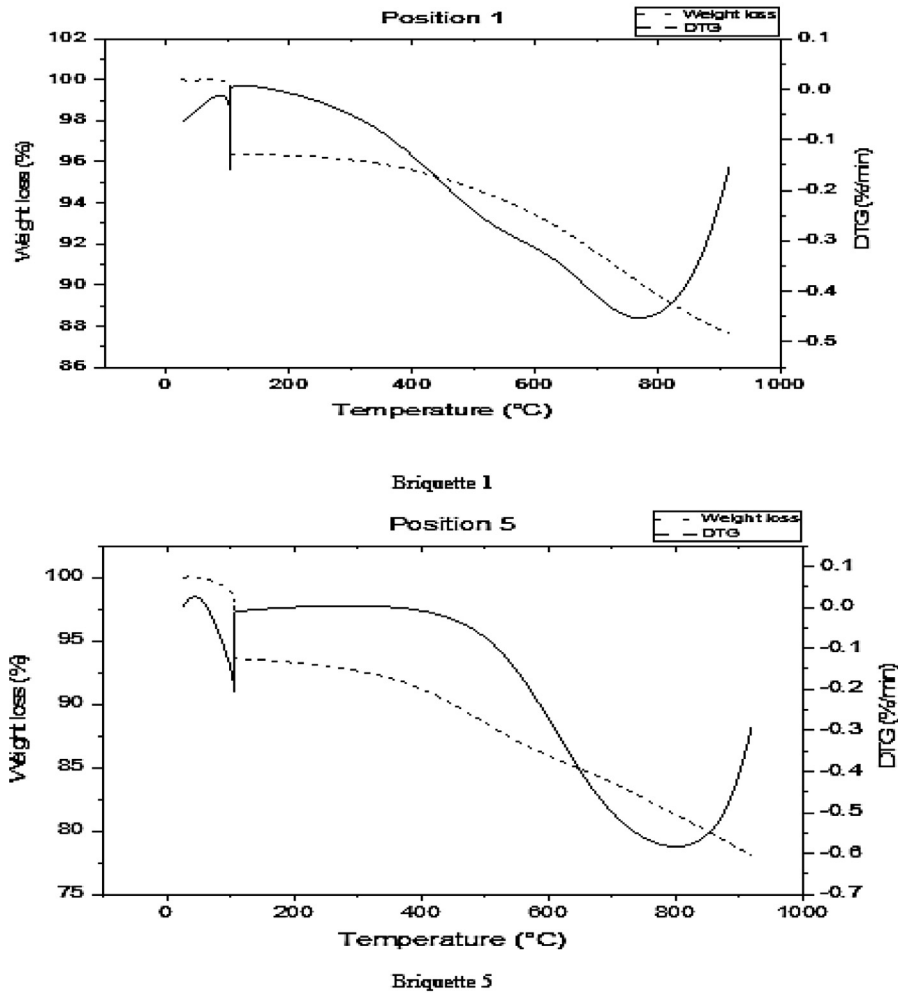


Fig. 10. Weight loss and derivative thermogravimetry curves for briquette samples 1 and 5.

value of the fuel. According to the results, the carbon, hydrogen, oxygen, and nitrogen content (% wt) in the briquette samples were; 21%, 1.3%, 1.8%, and 1.9%, respectively. The low nitrogen content in the briquettes is a requirement of a good fuel because it is associated with minimal release of nitrogen oxides [15].

#### Thermal Analysis Using TGA/DTG

Figs 10 and 11 show the weight loss and derivative thermogravimetry (DTG) of briquette samples 1, 5, 10, and 15 under an oxidizing atmosphere as a function of temperature.

The results indicate that the incorporation of different cow feedstock resulted in a thermogravimetric behavior that is different from typical biomass materials. Moreover, the clear peaks for volatilization are due to the presence of hemicellulose, cellulose, and lignin. This is attributed to the fact that each individual briquette material exhibited its own degradation pattern. Furthermore, the TGA thermograms of the developed briquettes remained at a plateau from combustion at room temperature to 104°C, followed by undergoing a major weight loss at about 105°C. This weight loss occurred due to the evaporation of moisture from the composite briquettes. The low peaks in the de-watering process are related to lower amounts of internal moisture in the bio-composite briquettes which is a result of the carbonization treatment process [21]. Between 600°C and 900°C, the last decomposition phase was observed. This stage involved the thermal decomposition of inorganic minerals like carbonates, owing to the degradation of lignin in the developed briquettes. Moreover, at approximately 900°C lignin in the developed bio-composite briquettes had decomposed off, implying that the remaining weight percentage was mainly composed of residues, including, ash, tars and fixed carbon [21].

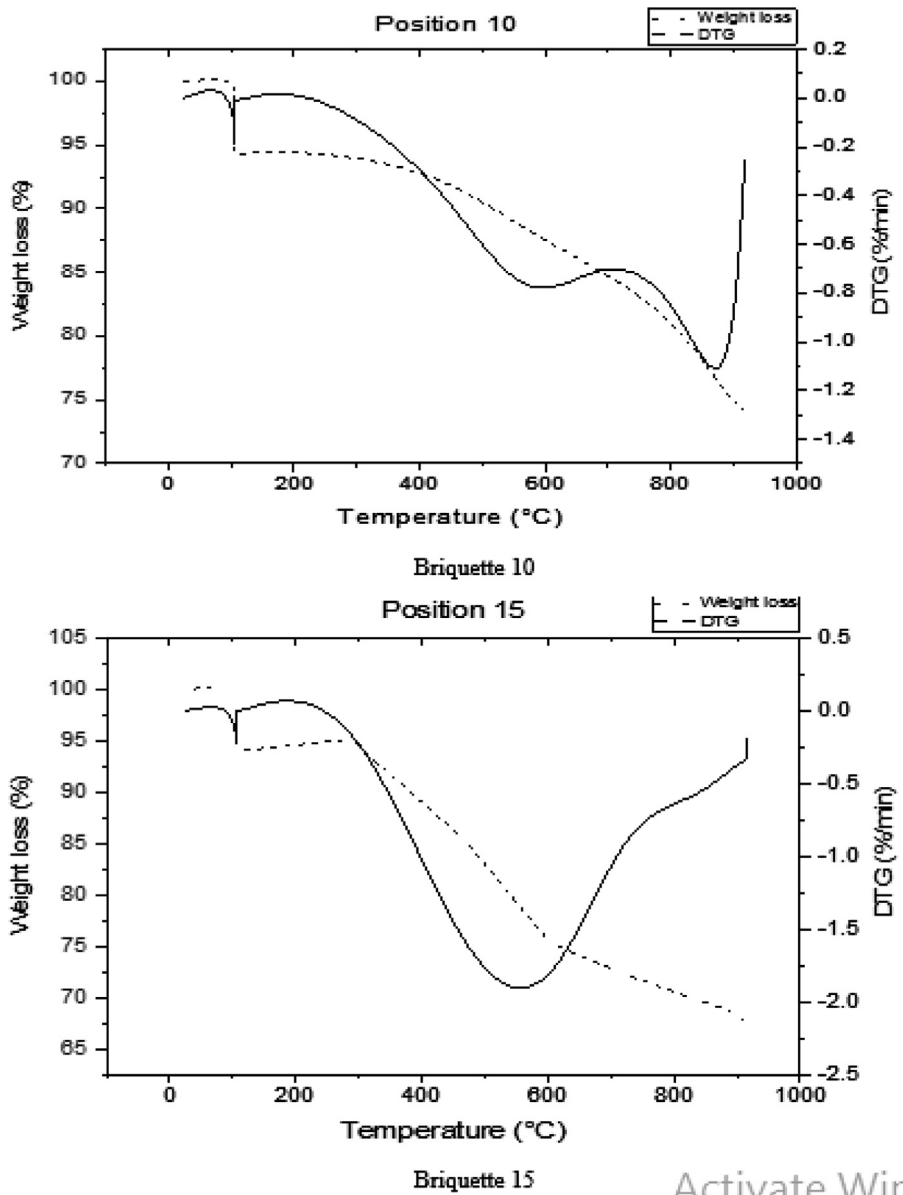


Fig. 11. Weight loss and derivative thermogravimetry curves for briquette sample10 and 15.

*Properties of the Developed briquettes*

(a) Ignition Time

The highest and lowest ignition time was 55 and 45 seconds, respectively, as shown in Table 3. The ignition time is a function of the volatile matter. A higher volatile matter leads to a high ignition time as more time is taken to burn off the volatiles before combustion. It was observed that the ignition time of the briquettes decreased with increase in the digestate concentration.

(b) Burning Time/ Burning Rate

At the end of approximately 30 minutes, most of the briquettes were about half of their initial size and if disturbed would break apart easily. The only briquette that still had solidity to its center was one that was obtained from a mixture of cow dung and human waste. After 75 minutes, the briquettes that were poked, fell apart, an indication that they were completely burned to ashes. In the burning time test, the briquette samples lit quite fast thereby producing sustained heat which was

**Table 2**  
Chemical analysis of ash.

Samples	Na( $\pm 0.01$ )	K( $\pm 0.01$ )	Cu( $\pm 0.01$ )	Cd( $\pm 0.01$ )	Pb( $\pm 0.1$ )
Digestate	2102.30	8543.10	6.27	1.61	29.10
Briquettes	1718.50	20017.80	6.12	1.22	25.60

**Table 3**  
Energy analysis results of briquettes.

Briquette samples	Weight of briquettes (kg) ( $\pm 0.001$ )	Initial weight of briquettes (g) ( $\pm 0.1$ )	Final weight of briquettes (g) ( $\pm 0.1$ )	Ignition time (sec) ( $\pm 1$ )	Burning time (min) ( $\pm 1$ )	Burning rate (g/min) ( $\pm 0.01$ )	Water boiling time (min) ( $\pm 1$ ) ( $\pm 1$ )	Specific fuel consumption (kg/L) ( $\pm 0.001$ )
1	0.036	36.7	28.3	50	75	0.112	32	0.144
2	0.044	44.8	30.1	49	74	0.199	31	0.176
3	0.044	44.7	30	46	70	0.21	33	0.176
4	0.041	41.6	29.5	45	76	0.159	33	0.164
5	0.041	41	29	55	70	0.171	32	0.164
6	0.036	36.4	28.8	53	74	0.103	31	0.144
7	0.042	42.2	29.5	54	73	0.174	28	0.168
8	0.045	45	32	52	72	0.181	32	0.18
9	0.039	39.6	29.6	47	75	0.133	33	0.156
10	0.042	42.1	30	50	74	0.164	31	0.168
11	0.041	41.6	29.6	54	73	0.164	29	0.164
12	0.037	37	27.5	48	75	0.127	31	0.148
13	0.044	44	30.2	54	70	0.197	29	0.176
14	0.037	37.3	29.3	53	75	0.107	32	0.148
15	0.044	44	29.9	51	74	0.191	32	0.176
16	0.041	41.3	29	49	75	0.164	29	0.164
17	0.0362	36.2	27.3	46	73	0.122	29	0.1448
18	0.0424	42.4	29.9	53	70	0.179	31	0.1696
19	0.0452	45.2	30.2	48	73	0.205	32	0.1808
20	0.0398	39.8	29	50	75	0.144	32	0.1592

sufficient to boil water. It appeared that the whole carbonized briquette having a lower calorific value and higher volatile matter produced a higher ash residue. Subtracting the time the briquettes turned to ashes completely from the ignition time gives the burning rate [20] as presented in the Table 3.

#### (c) Water Boiling Test

In the water boiling test, it was observed that the briquettes burnt completely with a uniform flame. The burning rate and the calorific value are two combined factors that control the water boiling time. The recorded duration for the water boiling tests ranged between 28 to 33 minutes. Table 3, summarizes the ignition time, burning rate, water boiling test, and specific fuel consumption test results obtained for the 20 samples of briquettes. In the water boiling test, the briquettes that had high volatile matter burned with higher temperatures (250°C – 300°C), hence boiling water faster. According to [12], factors that could be responsible for burning rate of briquettes are volatile matter content and geometry of the briquettes. The lower the specific fuel consumption, the more economical the fuel source [12].

#### (d) Ash compositional analysis

The samples were subjected to mineralization for pre-concentration of trace substances prior to chemical analysis. The ash compositional analysis was done on both the digestate and briquettes. This was purposely aimed at measuring the amount of specific inorganic components present such as Na, K, Pb, Cu, and Cd with the intentions of identifying the presence of heavy metals and possibility of using the ashes as fertilizers. The presence of Na and K in both the digestate and briquette ashes implies that they can still be used as fertilizers. The presence of heavy metals like Cu, Cd, and Pb were in small amounts. This poses very little threat to the end users when the briquettes are used as fuel. The Table 2 shows the chemical composition of ash obtained from digestate and briquette samples.

Table 3 below shows the specific fuel consumption, burning rate, ignition, burning and water boiling time for the 20 samples of briquettes. Briquette samples 1-5 were obtained from batch feeding method under laboratory scale conditions while samples 6-10 were obtained from the continuous feeding method under laboratory scale conditions. Samples 11-20 from household digesters operated under continuous feeding method.

It should be noted from the Table 3 above that briquette samples 1 to 5 were obtained from batch feeding method (laboratory scale), 6 to 10 from continuous feeding method (laboratory scale) and 11 to 20 from Household digesters (Operated under continuous feeding method).

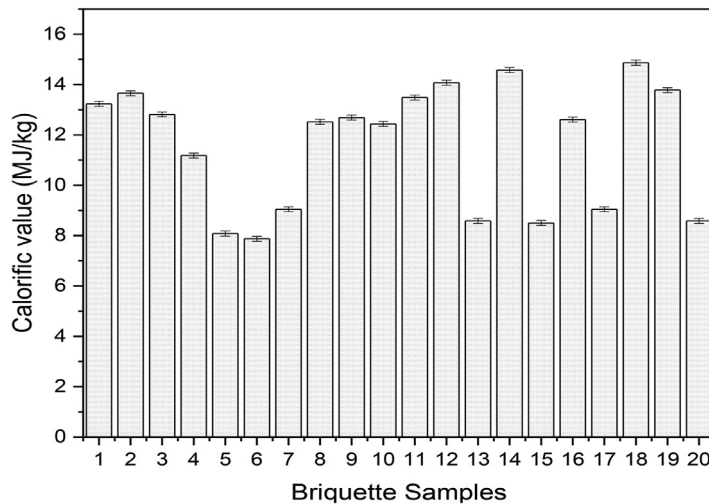


Fig. 12. Calorific values against the briquette samples.

#### (e) Determination of Calorific Value

Fig. 12 shows the results of the calorific value of the briquette samples. As can be seen, the calorific values of briquette sample 18 was higher than the rest of the briquette samples with a value of 14.87 MJ/kg. Briquette sample 6 reported the lowest value at 7.87 MJ/kg. The average calorific value obtained was 11.73 MJ/kg.

The lower calorific value may be due to the addition of a non-combustible substance which does not contribute to the total heat value released. The highest calorific value reported from briquette sample 17 was probably due to the moderate carbon content. Also, the particle sizes of the raw materials and the uniform formulation favors abrasions and a high degree of conditioning due to its ability to absorb moisture. The low calorific values of briquette samples 5 and 6 at 8.09 MJ/kg and 7.87 MJ/kg, respectively meant that they had a low carbon content. Moreover, the cassava starch used as binder influenced the properties of these briquette samples. According to Sani et al. [11], cassava starch had a moisture content of 13.09% on dry basis, ash content of 0.24%, a pH of 5.34% and a calorific value of 0.67 MJ/kg. This made it an excellent binder for use. The results also indicate appreciable amounts of heating value that can support heating. The higher heating value of the briquettes obtained from the present study is lower than the 16.34 MJ/kg obtained by Hasan et al. [9]. Also in comparison, [22], obtained the higher heating value of wood charcoal as 31.01–33.10 MJ/kg and of char briquettes as 22.21–28.91 MJ/kg. The highest value of 14.87 MJ/kg reported in the present study is low compared to the above-mentioned fuels due to the high levels of ash content which negatively affects the energy content.

#### Mechanical Characterization

##### Drop test

Table 4 shows the drop strength test results for the briquettes. The strength is presented as a fraction of briquettes of over 10 mm in diameter in relation to the total briquette bulk. The average drop test result was 94.54%.

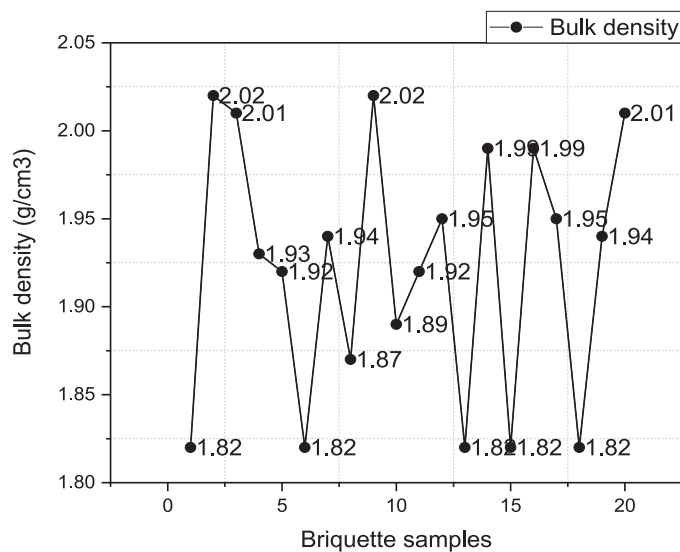
From Table 4 above, it can be seen that the briquettes obtained from the household digesters were slightly weaker than those obtained from the batch and continuous feeding methods under laboratory conditions. This may be attributed to a long overdue accumulation of unprocessed digestate. Moreover, the briquettes had the same shock and impact onto the concrete floor. The final result values showed a high level of mechanical durability. The overall average value was equal to 94.53% (minimum 86.06%, maximum 100%). This corresponds to the highest grade of the quality indicator (DU B 95%) given by the International standard for solid biofuels EN ISO 17225-1:2015. This therefore implies that the use of cassava starch binder allowed a better compaction based on the fact that the viscosity of hydrated starch increased its shear and tensile strengths thereby giving it the ability to occupy void spaces present within and between the char particles. This led to the formation of solid bridges that increased in strength during air cooling and storage [17].

##### Bulk density

The bulk density results of digestate briquettes are presented in Fig. 13. The results revealed low bulk densities with the highest being 2.02 g/cm<sup>3</sup> and the lowest 1.82 g/cm<sup>3</sup>. Due to the heterogeneous nature, digestate material possessed slightly higher bulk densities than the briquettes, an indication that the briquettes were able to withstand handling and storage procedures without damage. This helped to preserve the integrity of the briquettes with their average bulk density being reported at 1.93 g/cm<sup>3</sup>. According to Lohri et al. [23], in the determination of bulk density of biomass using measuring cylinders, they concluded that powdered biomass had bulk densities in the range of  $9.89 \times 10^{-2}$  to  $1.32 \times 10^{-1}$  g/cm<sup>3</sup> by

**Table 4**  
Average drop strength test results for the briquettes.

Briquette samples	Weight before (Wb)/g ( $\pm 0.1$ )	Weight after (Wa)/g ( $\pm 0.1$ )	Fraction over 10 mm (Wa/Wb)X100%( $\pm 1$ )
1	36.7	36.5	99.46
2	44.8	44.3	98.88
3	44.7	42.2	94.41
4	41.6	39.9	95.91
5	41	39.9	97.32
6	36.4	35.4	97.25
7	42.2	38.8	91.94
8	45	44.9	99.78
9	39.6	39.7	100
10	42.1	41.9	99.52
11	41.6	39.8	95.67
12	37	34.9	94.32
13	44	43.3	98.41
14	37.3	32.1	86.06
15	44	42.1	95.68
16	41.3	35.4	85.71
17	36.2	33.4	92.27
18	42.4	38.3	90.33
19	45.2	41.5	91.81
20	39.8	34.2	85.93



**Fig. 13.** Bulk density of digestate briquettes.

a rectangular container and from  $9.02 \times 10^{-2}$  to  $1.27 \times 10^{-2}$  g/cm<sup>3</sup>, by a cylindrical container. Further, cumbu napier grass biomass had the highest bulk density of  $1.32 \times 10^{-1}$  and  $1.18 \times 10^{-1}$  g/cm<sup>3</sup> by rectangular and circular container, respectively. Ground nut shells had the lowest bulk density of  $9.89 \times 10^{-2}$  and  $9.02 \times 10^{-2}$  g/cm<sup>3</sup>, respectively. Ground nut shells were observed to possess the lowest bulk density due their larger particle sizes and shapes.

It can also be noted from the results in the Fig. 13 above that the bulk density of the briquettes exhibited random values regardless of the retention time and feeding methods. Also, the briquettes with higher bulk densities were due to the smaller particle sizes that reduce the number and size of air pockets thereby facilitating compaction. On the other hand, the reported low bulk densities were due to the enclosure of some air pockets.

#### Compressive strength

Fig. 14 shows the results of the average compressive strength of the briquette samples. The highest and lowest strength attained was 50 MPa and 19 MPa, respectively with an average drop test reported at 30.55 MPa. Therefore, the briquettes' compressive strength showed that they were able to withstand compressive forces when packed by stacking one onto the other and being transported without breaking. Also, observations showed that the briquettes had 45 mm height and 44 mm diameter sizes. According to Singaravelu [31], the compressive strength varied from 6.26 to 20.18 N/mm<sup>2</sup> in production and quality testing of fuel briquettes made from pongamia and tamarind shell. They concluded that the briquettes' surface compressive strength of 19.60 N/mm<sup>2</sup> was reasonably adequate for handling and considered an appropriate fuel for domestic

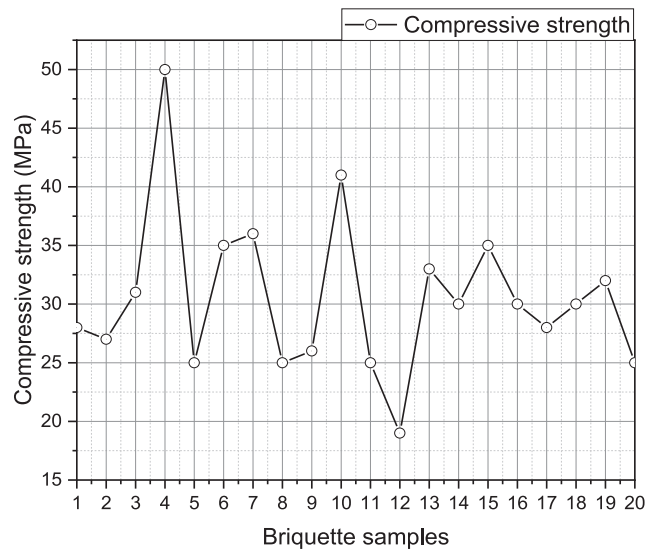


Fig. 14. Compressive strength of briquettes.

purposes. In general, when the raw material is finer, it gives a larger surface area for bonding which results in the production of briquettes with higher density.

## Conclusion

From the present study, the following conclusions can be drawn;

- The digestate compositions exhibited differences which had minor effects on the combustion properties of the briquettes despite varying retention times.
- The developed briquettes had a higher percentage of ash, an indication that they had much inorganic matter, hence lowering the heating value. They also had lower fixed carbon which gave insufficient carbon for briquetting.
- The practicability of domestic production of solid fuel for thermal applications from the digestate is feasible.
- Therefore, the moderate calorific value of the developed briquettes strongly provides an alternative energy source for thermal applications despite the 15% of ash residue.

## Declaration of Competing Interest

Conflict of interest; On behalf of all authors, the corresponding author states that there is no conflict of interest

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