Chemical Science International Journal

29(1): 30-46, 2020; Article no.CSIJ.53969
ISSN: 2456-706X
(Past name: American Chemical Science Journal, Past ISSN: 2249-0205)

Determination of Compressive Strength and Combustibility Potential of Agricultural Waste Briquette

B. A. Thliza[1*], F. I. Abdulrahman[1], J. C. Akan[1], Z. M. Chellube[1] and B. Kime[2]

[1]Department of Chemistry, University of Maiduguri, P.M.B. 1069, Maiduguri, Borno State, Nigeria.
[2]Department of Chemistry, Modibbo Adama University of Technology, P.M.B. 2076, Yola, Adamawa State, Nigeria.

Authors' contributions

This work was carried out in collaboration among all authors. All authors read and approved the final manuscript.

Article Information

DOI: 10.9734/CSJI/2020/v29i130156
Editor(s):
(1) Dr. R. Rajalakshmi, Professor, Department of Chemistry, Avinash Lingam Institute for Home Science and Higher Education for Women, India.
Reviewers:
(1) Imtiaz Ahmad, University of Peshawar, Pakistan.
(2) C. Yonny Martinez Lopez, Federal University of Espírito Santo, Brazil.
Complete Peer review History: http://www.sdiarticle4.com/review-history/53969

Original Research Article

Received 14 November 2019
Accepted 24 January 2020
Published 25 February 2020

ABSTRACT

This study evaluates the development of a substitute and alternative solid fuel in the form of briquette from agricultural wastes (rice husk, sawdust and cotton stalk charcoal). Four sets of briquette with different grades were produced using a discontinuous briquette production technology, where a single briquette is produced at a time in a closed mould. Some physical properties such as; Length of briquettes (170 mm), diameter of briquettes (50 mm), mass of briquettes (140-160 g), volume of briquettes (133.5 cm^3), density of briquettes (1.05-1.20 gm^3), texture of briquettes (rough), and colour of briquettes (brown, light brown and mud black), were investigated using physical methods of evaluation. The result of the proximate analysis (moisture 10.5-10.8%, ash 30.3-33.8%, volatile matter 20.5-25.9% and fixed carbon contents 45.2-52.6%. The burning potential tests carried out on the formed briquettes compared to that of firewood showed that firewood boils 5 litres of water in 60 minutes, while Rice husk, Sawdust, Cotton stalk charcoal blended with rice husk, and Sawdust briquettes boils the same volume of water in 35, 30, 20 and 25, minutes respectively. Compressive strength of the briquettes was determined as 155.9, 155.9, 158.1

E-mail: edmondmoses63@yahoo.com;

and 158.1 KN/m2 for rice husk, sawdust, cotton stalk charcoal blended with rice husk and sawdust briquettes respectively. Energy dispersed x-ray fluorescence spectrometer (ED-XRF) analyses revealed the existence of minor and major elemental percentage compositions (Cl, Br, P, K, Ca, Ba, Cu, Zn, Fe, Mn and Si, Ti, V, Cr, Ni, Sr, Rb). Liebig's and Kjeldahl's methods of laboratory analyses confirmed the presence of organic elements (C, H, O, N and S) that contributes to the heating value, increase in ignitability, smooth combustion. It can, therefore, be concluded that briquettes produced in this research work provide a better alternative to firewood and charcoal energies, they are eco-friendly, having high heat intensity with smooth burning and are easy to handle, store and transport, they are very cheap, affordable to both rural and semi-urban dwellers.

Keywords: Cotton stalk charcoal; rice husk; sawdust; cassava starch.

1. INTRODUCTION

Global warming has become an international concern [1]. This is caused by greenhouse gases, it was shown that increased emission of carbon (iv) oxide have been drastically reduced owing to the facts that the rate of deforestation is higher than afforestation in search of wood and charcoal for fuel [2]. The decreasing availability of woods and charcoal fuels has necessitated that efforts be made towards efficient utilization of agricultural wastes in the form of briquettes that can be affordable for all.

Compaction of bulky combustible materials for fuel making purposes has been a technology widely used by many countries [3]. Among the common types of briquettes widely used in some countries are biomass briquettes, coal briquettes and charcoal briquettes etc. However, blending cotton stalk charcoal with biomass (Agro waste) gives rise to briquettes with better combustion properties and pollutant-free emission compare to the conventional firewood charcoal briquettes. These briquettes are known as Biomass briquettes which is a type of solid fuel prepared by compacting pulverized biomass (Agro-waste such as rice husk, and sawdust), and binder [4] provide better fuel for domestic heating and cooking for urban and rural areas. However, there have been several types of research carried out on the production of fuel briquettes for both domestic cooking and industrial application. One of the major driving forces behind these researches is the need to address the environmental consequences and health hazards associated with the use of solid fuels such as fuelwood and charcoal [5] and also effective means of recycling and managing agro wastes.

Biomass or agricultural wastes has a lot of energy abounding in them that can be properly recycled and processed into useful products or used directly by burning them to produce heat or indirectly by converting it to various biomass fuels [3]. In most developing countries like Nigeria recycling of waste products (an agricultural waste product) into a useful product is rarely practice. This has led to environmental problems such as pollution resulting in a refuse heap on our streets, drainage system and waterways, which has resulted in flooding on rainy days due to the blockage of the waterways. This attitude has also resulted in the outbreak of the epidemic in our societies today [6]. The availability of energy in the rural, as well as urban areas globally, is fast becoming a great challenge with the high cost of cooking gas and kerosene and environmental problems associated with firewood [7]. Electricity is erratic and unreliable. Other sources of energy that include sunlight, geothermal power, wind and tidal movements, are becoming not economically now for large-scale uses, and cannot be affordable especially for the rural and semi-urban dwellers [8]. This has drawn attention to the need for an urgent transition to a more sustainable energy system that would be affordable and eco-friendly [9]. As such this research is on the prospects of using agro wastes and other biomass for the production of solid fuels called briquettes which would serve as substitutes and alternative to the depleting non-renewable energy sources.

2. MATERIALS AND METHODS

2.1 Raw Materials

Agro-waste which comprises of (rice husks, sawdust, and cotton stalk charcoal), and binding materials comprising of prepared cassava starch and Calcium hydroxide (Ca $(OH)_2$.

2.2 Apparatus

Compression machine model EL-31-34010, Locally fabricated briquetting machine, Laboratory thermometer, stopwatch, Corona hand grinder, Plastic basins, Aluminum kettle (10 litres), Measuring cylinder (1000 ml), Analytical balance sensitive to 0.5 g, Siever, Aluminum pots (10 litres), Locally fabricated stoves, locally fabricated cylindrical moulds, ED-XRFS Minipal-4 machine, Kjeldahl's, and Liebig's apparatus, Carbonizer (earth kiln), and triple beam balance, model: MB-2610 g.

2.3 Methods

2.3.1 Biomass collection

Rice Husk, sawdust, and cotton stalk charcoal are selected as raw materials because of their availability, low cost and abundance. The rice husk sample was collected from Rice mill house at Zabarmari ward. The sawdust was collected from timber shade at Baga road area, while cotton stalk was obtained from Biu all in Borno state.

2.3.2 Preparation of samples

Rice husks and sawdust were sorted and made into smaller sizes and sun-dried for ten (10) days until its moisture contents found to reduce to the original contents. Later, the samples were pulverized (ground) using corona hand grinder to pass through 1 mm sieve and stored for use (Plates 1 and 2).

Cotton stalk which is traditionally considered as wastes were collected from Biu area. The cotton stalk was sorted and dried to reduce the moisture content of the feedstock to ensure effective carbonization. The dried cotton stalk was shredded to small sizes to provide more surface area for the carbonization. The cotton stalks biomass was carbonized using the conventional earth kiln method as adopted by Abdu, et al. [10] and was modified as traditionally used for wood carbonization (Plate 3). The Carbonizer is a simple earth dome design to provide a means of creating low oxygen environment, it was built using a silt soil of about 500 cm in height and 200 cm diameter with no opening at all sides. The biomass (cotton stalks) was sized such that they will be fed into the reactor (earth kiln) at a manageable batch of 500 kg. A fire port was provided at the bottom of the earth and was light through wicks. At the start of the carbonization process, an open was left for approximately 10 minutes for the volatile gases to escape. The open was then closed thereafter; properly sealed to prevent air from entering (Plate 3). The biomass material was left to carbonize for 10-24 hours. The fully carbonized material was collected, air-dried for another 2 days (Plate 4), after which they were pulverized for making briquette.

Plate 1. Sorted rice husks

Plate 2. Sorted sawdust

Plate 3. Earth dome kiln

2.3.4 Briquettes production

The collected rice husk and sawdust samples were screened from stones and other impurities that might inhibit proper briquette formation, the cotton stalk charcoal was powdered properly. After boiling, the hot water was then mixed with the starch until a sticky gel was produced. Rice husk, sawdust, cotton stalk charcoal briquettes, cotton stalk charcoal briquettes blended with rice husk, cotton stalk charcoal briquettes blended with sawdust, are produced using the cassava starch binder; four sets of briquettes were produced at the ratio of 6:2,6:2, 6:6:4, and 6:6:4 (rice husk: cassava starch paste wt/wt, sawdust: Cassava starch paste wt/wt, cotton stalk charcoal: Rice husk: Cassava starch paste wt/wt, and cotton stalk charcoal: Sawdust: Cassava starch paste wt/wt). Using the discontinuous process, in which a single briquette is produced at a time in a closed mould the mixtures were loaded into a fabricated mould and compressed by weight to the mould for some time, then extruded and sun-dried. The drying process was continued for proper drying to be achieved before the various analysis.

Plate 4. Cotton stalks charcoal

2.4 Proximate Analysis

Proximate analysis, which is a standardized procedure that gives an idea of the bulk components that make up a fuel, was done to determine the average of the percentage volatile matter content, percentage ash content, moisture content, physical properties and percentage content of fixed carbon of the briquettes obtained from four replicates. The procedures of the [11] were adopted to obtain the following parameters:

Determination of physical properties of briquette samples: The method used by Andrew, et al. [12] was adopted. four briquettes were selected for evaluation of the physical properties such as colour, texture, volume height, mass, density and diameter. The mass was obtained by using a digital weighing scale, while the volume was calculated by taking the dimensions of the cylindrical briquettes (radius and height). By applying the formula for the volume of a cylinder ($\pi D^2 H$), the volume of the formed briquette was obtained, the density was obtained by evaluation, D = m/v, while the colour and texture wawereone by physical observations.

The determination moisture content of briquette samples: The initial moisture content of each briquettes samples was determined according to the method Used by Andrew, et al. [12], with slight modification. 10 g each the samples were weighed and dried at a temperature of 150°C in a hot air oven until constant weights of the samples were recorded. Using an analytical weighing balance, the weight of each sample was recorded after and before putting them in the oven. The dried samples weight represents the weight of dry matter present in grams. The initial moisture content of each sample was calculated using Eqn. (1) and is expressed in percentage wet basis (% wb).

$$\text{MC (\% wb).}= \frac{w1-w2}{w1} \text{ x } 100 \qquad (1)$$

Where W1 is the initial weight of the sample and W2 is the final weight or dry weight of the Sample.

Determination of ash content of briquette samples: Six grams (6 g) portion of each sample were placed in a preweighed porcelain crucible and transferred into a preheated oven set at a temperature of 600°C for 5 hours after which the crucible and its content were transferred to a desiccator and allowed to cool. The crucible and its content were reweighed and the new weight noted, the method used by Ikele, et al. [13] was adopted. The percentage of ash content was calculated thus:

$$\text{AC (\%)}= \frac{W2}{W1} \text{X}100 \qquad (2)$$

Where, W2= weight of ash cooling, W1= Original weight of dry sample AC = Ash content

Determination of volatile matter of briquette samples: A Portion (18 g) of the sample was heated to about 300°C for 30 minutes in a partially closed crucible in an oven. The crucible and its content were retrieved and cooled in a desiccator. The difference in weight was recorded and the volatile matter was calculated thus.

$$\text{VM} = \frac{W1-W2}{W1} \text{ X } 100 \qquad (3)$$

Where, VM = Volatile Matter. W1 = Original Weight of sample. W2 = Weight of the sample after cooling this method was used by Agu, et al. [14] and was adopted.

Determination of percentage fixed carbon of briquette samples: The method used by Andrew, et al. [12] was adopted. The percentage fixed carbon (PFC) was computed by subtracting the sum of PVM (percentage volatile matters) and PAC (percentage ash content) from 100 as shown in Equation 4:

$$\text{Fixed Carbon} = 100\% - (PAC + PVM) \qquad (4)$$

Determination of water boiling points of briquette samples: The water boiling test is a measure of time it takes a given quantity of fuel to heat and boils a given quantity of water. In line with the method adopted by Yahaya and Ibrahim [6], with modification a known quantity each of both briquettes and firewood was measured. The first briquette sample was stacked in a 1st local fabricated stove while the firewood was stacked in a 2nd fabricated local stove. The stoves were ignited and as soon as the flames were stabilized for 2 minutes, two aluminium pots containing 5 litres of water each were mounted on the stoves. A stopwatch was activated. The initial temperatures of the water were noted and thereafter readings were obtained at 5 minutes' interval using a thermometer. This was terminated after attaining boiling point. Similarly, a known quantity of the second sample was stacked in the stove while firewood was stacked in the second stove and the procedure repeated for the remaining two briquette samples.

Plate 5. Briquette production process

Plate 6. Formed briquettes

Plate 7. Water boiling test experiment

Determination of compressive strength of briquette samples: The compressive strength is the force required to crush or break a material. In line with [15] and [16], the compressive strength of the briquette samples was determined using a triaxial cell compressive strength testing machine Model ELE 2914 at the Structural Laboratory of Civic Engineering Department of University of Maiduguri, Borno State. Nigeria. This machine is 1000 KN capacity capable of compressing non-metallic materials. It is powered by electricity but hydraulically operated. The length and width of the briquette samples were measured and recorded. Each sample was centrally placed on the lower plate inside the machine, the plate was winded up electrically until a contact was made with the top plate, the strain dial gauge on the pillar was adjusted to zero, and the reading on the strain dials and the load in the starting position under zero compressive load was recorded. The machine is now powered on. Using stop clock the readings of the load dial were recorded at every 0.2 mm and strain dial reading recorded, while others at an interval of 0.4 mm were taken. Loading and taking readings continue until it was certain that failure has occurred for more consecutive readings of the load dial to showed a decrease or a constant load. The machine will then be stopped and the motor is allowed to stop completely and was put into reverse until the load is taken off. The machine plate was lowered to enable the sample removed. The other way the machine will be switched from the mains and allowed to warm up for about 3 minutes. The samples will then put on the movable bed, and the control lever applied upward to bring contact between the upper fixed bed and the movable lower bed on which the samples will be sitting on the lower base of the machine shown by plate. The dial reading will be taken immediately, the crack will be noticed in the briquette samples, an indication that the feedstock has been compressed. The value of the reading recorded from the machine was the strain dial force reading. The compressive strength of the samples will be calculated using the equation (6). The unit will be given in KN/m^2.

$$\text{Compressive strength} = \sigma = RCR(100-\varepsilon\%)/100A_0 \times 1000\ \text{KN/m2} \tag{6}$$

Where R is load dial reading, C_R is mean calibration load dial reading (2.11 Div.) constant, A_0 is cross-sectional area = $\pi D^2/4$, D is diameter of briquette (mm), ε is the strain of sample =σ, $\varepsilon\% = DL/L_0$, DL is changed in length of sample at compression, and L_0 is initial length of sample before compressed Akpabio, et al. [15].

2.5 Ultimate Analysis

Determination of nitrogen by Kjeldahl method of briquette samples: The method used by Adekunle, et al. [17] was adopted with slight modification. Kjeldahl has been adapted as a standard method of Nitrogen analysis in briquette sample, wastewater, soil, fossil fuels, to name a few.

Plate 8. Compressive strength test experiment

This method of determination using ASTM-D3179 standard is composed of three distinct steps: These are digestion, distillation, and titration. 0.3 – 0.5 g of briquette samples were weighed one at a time into a Kjeldahl digestion flask. 25 ml Sulphuric acid and 1.5 g each of copper sulphate and potassium sulphate (catalysts) were added into the flask. The flask was heated gently until boiling; the mixture was then diluted with 100 ml of distilled water and allowed to cool.

The digested reaction mixture, on cooling, is transferred to a round-bottomed distillation flask, and distilled with a concentrated alkali solution (NaOH). Ammonia produced is absorbed in a known volume of HCl solution of known strength $(NH4)_2SO4 + 2NaOH \rightarrow Na_2SO4 + 2H_2O + 2NH3$.

The purpose of the digestion step is to break the intricate structure and chemical bonds that hold a chemical substance (briquette) down to simple chemicals and ionic structures.

Degradation: Sample + $H_2SO_4 \rightarrow (NH_4)_2SO_4(aq) + CO_2(g) + SO_2(g) + H_2O(g)$

Liberation of ammonia: $(NH_4)_2SO_4(aq) + 2NaOH \rightarrow Na_2SO_4(aq) + 2H_2O(l) + 2NH_3(g)$ at the distillation step the flask was then connected to the Kjeldahl distillation apparatus and sodium hydroxide solution was added to the mixture and then heated to boiling, the ammonium ion (NH_4+) changes to ammonia (NH_3). Capture of ammonia: $B(OH)3 + H_2O + NH_3 \rightarrow NH_4+ + B(OH)_4-$. The final step is when ammonia gas was condensed into the receiving flask containing 2% boric acid. Bromocresol green and methyl red indicators were added dropwise and the alkaline distillate was titrated against 0.1 M hydrochloric acid. Back titration: $B(OH)_3 + H_2O + Na_2CO_3 \rightarrow NaHCO_3(aq) + NaB(OH)_4(aq) + CO_2(g) + H_2O$.

The procedure was repeated for the remaining three samples of briquette and the percentage of nitrogen was calculated as shown in equation:

Let, Mass of the briquette sample = Wg

The volume of the standard acid required for complete neutralization of the evolved ammonia = V mL

Normality of the standard solution of acid = Nfrom the law of equivalence (normality equation),

1000 mL of 1 N acid = 1000 mL of 1 N NH_3 = 17g NH_3 = 14 g nitrogen

Then, V mL of N acid = V mL of NH_3 NV milliequivalent of acid = NV milliequivalent of ammonia

Therefore, Mass of N in the evolved NH_3 = 14 X N X V/1000 (g)

Then, %N in the B.S. samples = 14NV/1000 X 100/W X 14NV/W

Therefore: %N in the B.S. = 1.4 X Normality of HCl X Vol of HCl used for complete neutralization of NH3/Mass of the briquette sample taken.

Determination of carbon, hydrogen and oxygen by Liebig's method of briquette samples: To determine carbon, hydrogen and oxygen contents in briquette samples the method used by Adekunle, et al. [17] was adopted. The values were determined based on (ASTM- D3178) standards.

These elements are estimated by its combustion in an atmosphere of pure oxygen to form carbon (IV) oxide (CO_2) and water (H_2O), from KOH and anhydrous $CaCl_2$.

A known mass of 0.3 g each of the briquettes samples was placed in a porcelain boat and was strongly heated with cupric oxide at 400°C to 450°C. Pure dry oxygen is slowly passed through the tube. C& H present was oxidised to CO_2 & H_2O respectively. $C + O_2(g) \rightarrow CO_2(g)$ and $4H + O_2(g) \rightarrow 2H_2O$. Combustion is complete in 8 to 10 hours and results in the formation of CO_2 and H_2O from Lime water or KOH which is used to absorb obtained CO2 while Anhydrous. $CaCl_2$ or Anhydrous. $CuSO_4$ is used to absorb H_2O. The increase in the wt. of lime water or KOH is the wt. of CO_2 while the increase in the wt. of Anhydrous. $CaCl_2$ or Anhydrous. $CuSO_4$ is the weight of H_2O. ($CxHy + O \rightarrow xCO + y/2[H_2O]$). The elements were absorbed in the absorption tubes after 8 to 10 hours. The absorption tubes were then removed and weighed after cooling them to laboratory temperature to determine the amount of CO_2 and H_2O formed in the reaction. The masses and the percentages of C, H and O in the briquette samples were calculated as follows.

Let the mass of briquette samples = Wg, Increase in the mass of $CaCl_2$ tube = W_1g, Increase in the mass of KOH tube = W_2g.

So, Mass of H_2O formed =W1 g, Mass of CO_2 formed = W2 g

Then, Mass of H in the B.S. = 2/18 X W_1 g [H = 2, H_2O = 18]

Mass of C in the B.S. =12/44 X W_2 g [C = 12, O = 18, CO_2 = 44]

Therefore: %C = 12/44 X W2 /W1 X100. %H = 2/18 X W1/W2 X 100. %O = 100 – (Total %C +%H)

Determination of inorganic elements using energy dispersive x-ray fluorescence spectrometry (ED-XRFS) of briquette samples: The ED-XRFS is a non-destructive method of quantitative and qualitative elemental analysis of solid and liquid sample materials. In this process, the high energy content of an X-ray beam causes a sample to generate X-rays characteristics of the atoms in the sample when inner K, L or M electrons are removed from target atoms and outer electrons fill the vacancies.

Elements present in the sample are identified from the energies of this characteristic radiation, and concentrations are evaluated from intensity measurements. 20.00 g each of the briquette samples were finely grounded to pass through 200-250 mesh sieve. Each sample was dried in an oven at 105ºC for at least 1hr and cooled. Thereafter, the sample was intimately mixed with a binder in the ratio of 5.0 g sample(s) to 1.0 g cellulose flakes binder and pelletized at a pressure of 10-15 tons/inch in a pelletizing machine, at this stage, the pelletized sample(s) were stored in a desiccator for analysis. The machine, ED-XRFS, Plate 9 was switched on and allow to warm up for 2 hrs. Finally, appropriate programs for the various elements of interest are employed to analyze the sample material(s) for their presence or absence. The result of the analysis was reported in percentage (%) for minor and major concentrations of elements.

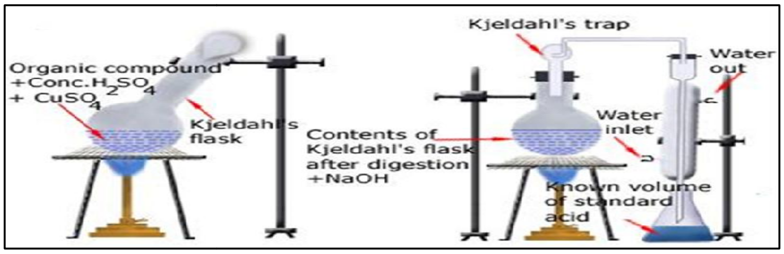

Fig. 1. Apparatus for the estimation of nitrogen by Kjeldahl's method (www.chemistry world, 2015)

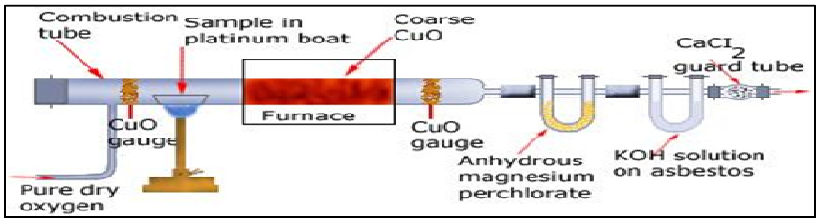

Fig. 2. Apparatus for estimating carbon and hydrogen (www.chemistry world, 2014)

Plate 9. Energy dispersive X-Ray fluorescence spectrometry (ED-XRFS) Minipal4

3. RESULTS AND DISCUSSION

3.1 Results

3.1.1 Physical properties of briquette samples

Table 1 presents the result of the physical properties of a four randomly selected formed briquettes. The results revealed that the formed briquette has a comparatively large volume of 133.5 cm^3 and a mass of 140.0 to 160 g which means they would be able to maintain combustion for a relatively long period given their volumes and masses. The density of the briquette was found to be 1.05 to 1.20 gcm^{-3} which is the same as the result obtained for the density of sawdust charcoal briquette reported by Akowuah, et al. [18]. This result falls within the range recommended by Jibrin, et al. [19] for sawdust briquette produced by the screw extrusion process. These parameters indicate that the briquettes will not crumble during transportation and storage because the values obtained for the density are quite moderate. Also, the physical condition of the briquettes revealed that their external surfaces were rough and the structure of the cross-section was compact and homogenous having a brownish, light brown, and mud black colour. These are good indices for efficient burning of the briquettes. The results are shown in Fig. 1.

3.1.2 Determination of percentage moisture content of briquette samples

The moisture content of the briquettes samples is 10.5% for the rice husk: Cassava starch formulation while it was 10.8% for the sawdust: Cassava starch, and 10.6, and10.5% for cotton stalk charcoal blended with rice husk and sawdust: Cassava starch formulation respectively (Table 2), the results were presented on Fig. 3. Physico-chemical properties of cassava starch paste that was used as a binder in the formulation of briquettes samples, was less than 10%. This result falls within the findings of Adekunle, et al. [17].

At 18% moisture content and below, a material does not contain free water but the water that is chemically bonded with a material bound water. This means that so long as a material contains a moisture content of less than 18%, most of its physicochemical properties would not be influenced by moisture content [8]. However, it was important to analyze the various properties of briquettes at almost similar moisture content levels. Dass, et al. [2] recommends charcoal briquettes for household use to be analyzed for these properties at moisture content not exceeding 18%.

3.1.3 Determination of percentage ash contents of briquette samples

Table 3 presents the minimum and maximum ash content for the briquettes produced using 6:2,6:2, 6:6:4, 6:6:4 formulations for rice husk, sawdust, cotton stalk charcoal blended with ric husk, sawdust and cassava starch paste, formulations, and the results were 32.8, 33.8, 0.3, and 30.3% respectively as indicated on Table 3 which conforms with Ikele, et al. [13] finding.

It is advantageous when briquette has a lower percentage of ash content since it saves on handling and disposal costs after the sample has been used for various economic purposes which is an advantage in terms of handling after use of

Table 1. Determination of physical properties of the briquette sample

S/N	B.S	M(g)	L(mm)	D(mm)	V(cm^3)	texture	cqlour	Density(gcm^{-3})
1	Rice Husk	145	170	50	133.5	rough	brown	1.10
2	Sawdust	140	170	50	133.5	rough	Light brown	1.05
3	CRH	145	170	50	133.5	rough	mud black	1.09
4	CSD	160	170	50	133.5	rough	mud black	1.20

CRH: Cotton Stalk Charcoal Blended with Rice Husk, CSD: Cotton Stalk Charcoal Blended with Sawdust

Table 2. Determination of percentage (%) moisture content of briquette samples

S/N	Briquette Sample	Wi (g)	Wf (g)	MC (%)
1	Rice Husk	18.7	16.7	10.6
2	Sawdust	18.5	16.5	10.8
3	CRH	18.9	16.9	10.6
4	CSD	19.0	17.0	10.5

CRH: Cotton Stalk Charcoal Blended with Rice Husk, CSD: Cotton Stalk Charcoal Blended with Sawdust

Table 3. Determination of percentage (%) ash content of briquette samples

S/N	Briquette samples	Initial weight of dry sample (g)	Weight of ash at cooling (g)	AC (%)
1	Rice Husk	6.4	2.1	32.8
2	Sawdust	6.5	2.2	33.8
3	CRH	6.6	2.0	30.3
4	CSD	6.6	2.0	30.3

CRH: Cotton Stalk Charcoal Blended with Rice Husk, CSD: Cotton Stalk Charcoal Blended with Sawdust

the briquette. High ash content decreases the burning rate and reduces the heating value of the fuel. The tolerance level of ash content for fuel is below 4%. Described by Imeh, et al. [8]

3.1.4 Determination of percentage volatile matters of briquette samples

The sample with the least volatile matter is expected to have the least energy value. Cotton stalk charcoal blended with rice husk was observed to have the lowest volatile matter of 20.5%. This accounts for its low energy values to drive off all volatile matters. Cotton stalk charcoal blended with sawdust has the highest volatile matter of 25.9% (Table 4). This implies that high energy will be required to burn off the volatile matter before the release of its heat energy. These results fall within the limits reported by Agu, et al. [4].

3.1.5 Determination of percentage fixed carbon content of briquette samples

Cotton stalk charcoal blended with rice husk has the highest percentage fix carbon of 52.6% (Table 5). The high values can be traced to its major components ash contents and volatile matters as reported by Jack [20]. High fixed carbon implies high burning efficiency. The lowest carbon content was observed in rice husk briquette, the increase in fixed carbon when compared to the overall constituents is most likely due to the concentration of binder in the briquette preparation. Yahaya and Ibrahim [6] showed that fixed carbon of briquettes made from agro-waste increased with increasing binder concentration. This trend was further observed in the work of Wang, et al. [21]. It is also probable that some moisture might be lost during the overall briquetting process, which will also result in higher fixed carbon which will further enhance the burning efficiency of briquette samples.

3.1.6 Determination of water boiling/burning efficiency of briquette samples

Water boiling time is a function of the burning efficiency value. The results obtained from the water boiling/ burning efficiency for briquette samples are presented in Fig. 3 and Table 6. From the result, Table 7 shows the variation of temperature with time for rice husk, sawdust, cotton stalk charcoal blends briquettes and firewood. It is seen from these figures that the cotton stalk charcoal blended with rice husk briquette attained a temperature of 100°C in 20 minutes while firewood attained 100°C in 1hr. In 25th minutes the cotton stalk charcoal blended with sawdust briquette rose to 100°C followed by sawdust briquette rose to 100°C in 30th minutes, this shows a better combustion characteristic compared to firewood, which burns slowly from 31°C in 0 minutes through 100°C in 60 minutes. This clear difference can be observed from the graph of temperature versus time for both the cotton stalk charcoal briquettes and firewood as shown in Table 7, this result falls within the limit as reported by Akpabio and Illalu [15] findings and in confirmation to Yahaya and Ibrahim [6] findings.

3.1.7 Determination of compressive strengths of briquette samples

Table 7 presents the compressive strength of the briquette samples. Cotton stalk charcoal blended with rice husk and sawdust are observed to have the highest compressive strength of 158.1, KN/m2 respectively, these results fall within the limits of Akpabio and Illalu [15] findings. This may be due to the uniform formulation and integration of particles of varying sizes. This is in confirmation with Gambo, et al. [22] findings which stated that a mixture of different particle sizes will give optimum briquette quality due to high inter-particle bonding with nearly no inter-particle spacing. Rice husk and sawdust briquettes have a lower compressive strength of 155.9 KN/m2. The higher compressive strength may be due to the elastic nature influenced by the presence of gelatin collagen fiber in the samples for the briquette. The lowest compressive strength in the other samples may be due to their particle size being larger after calcination. Larger particle size causes cracks and fracture of briquette as well as low fusion points. Compressive strength is one of the most important characteristics of a briquette that determines the stability and durability of the briquette as described by Akpabio and Illalu [15]. This trend was further observed in the work of Wessapan, et al. [16] research.

3.1.8 Ultimate analysis of briquette samples

The results of the ultimate analysis and the determination of the elemental composition of briquettes samples by Liebig, Kjeldahl methods, and ED-XRF spectrometry are depicted in Tables 8, 9 and 10, Figs. 4, 5 and 6 respectively. The ultimate analysis involves the estimation of important chemical elements that makes up the biomass, namely: Carbon, hydrogen, oxygen, nitrogen, and sulphur. The results of the determination of C, H, O, N, and S in briquette samples are shown in Table 8 and shown in Fig. 4.

It can be seen in Fig. 4 that, the carbon content of cotton stalk blended with rice husk and sawdust, are (77.2% each) is higher than that of rice husk and sawdust (76.8%), this is in agreement with Adekunle, et al. [17] findings, that a good briquette sample should have high amount of carbon, the higher is the burning efficiency value the better is the quality of a briquette sample. In Fig. 4, the carbon content decrease from 77.2% to 76.8%. Since carbon is one of the major combustible constituents of briquettes, it then follows that these briquettes have higher combustible carbon constituents than other agricultural briquettes although lower in comparison to coal briquettes [10]. The carbon contents of the briquette samples produced in this research are high enough to be good fuel for domestic use for heat applications reported by Adekunle, et al. [17].

Rice husk, has 4.14% hydrogen content while cotton stalk blended with rice husk has 2.89%.

Table 4. Determination of percentage (%) volatile matters of briquette samples

S/N	Briquette samples	Initial weight (g)	Weighted at cooling (g)	VM (%)
1	Rice Husk	18.6	14.5	22.0
2	Sawdust	18.4	18.8	25.0
3	CRH	18.0	14.3	20.5
4	CSD	18.5	13.7	25.9

CRH: Cotton Stalk Charcoal Blended with Rice Husk, CSD: Cotton Stalk Charcoal Blended with Sawdust

Table 5. Determination of percentage (%) Fixed carbon of briquette samples

S/N	Briquette samples	AC (%)	VM (%)	PFC (&)
1	Rice Husk	32.8	22.0	45.2
2	Sawdust	37.5	15.6	46.9
3	CRH	33.9	13.5	52.6
4	CSD	30.5	19.1	50.6

CRH: Cotton Stalk Charcoal Blended with Rice Husk, CSD: Cotton Stalk Charcoal Blended with Sawdust

Table 6. Water boiling test of briquette samples Vs firewood

S/N	Time (s)	Rice husk	Sawdust	CRH	CSD	Firewood
		Temp (°C)	Temp (°C)	Temp (°C)	Temp(°C)	Temp (°C)
1.	0	31	31	31	31	31
2.	5	50	55	62	60	45
3.	10	58	68	78	70	50
4.	15	66	79	93	80	55
5.	20	74	86	100	90	60
6.	25	82	92	-	100	65
7.	30	90	100	-	-	70
8.	35	100	-	-	-	75
9.	40	-	-	-	-	80
10.	45	-	-	-	-	85
11.	50	-	-	-	-	90
12.	55	-	-	-	-	95
13.	60	-	-	-	-	100

CRH: Cotton Stalk Charcoal Blended with Rice Husk, SD: Cotton Stalk Charcoal Blended with Sawdust

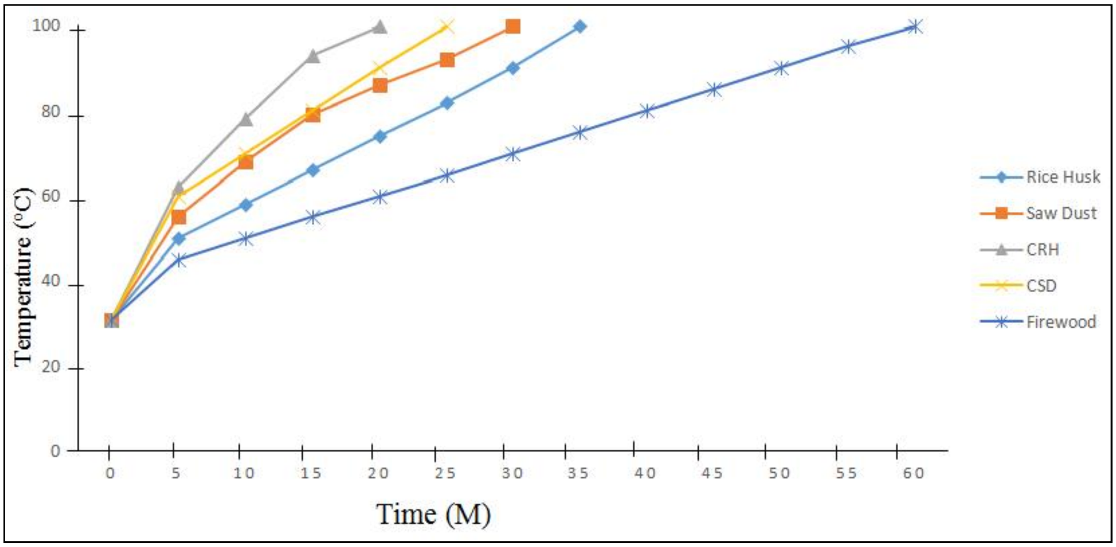

Fig. 3. The water boiling test of briquette samples against firewood

Table 7. Determination of compressive strengths of briquette samples

	Rice Husk	**Sawdust**	**CRH**	**CSD**
S/N	**σ (KN/m^2)**	**σ (KN/m^2)**	**σ (KN/m^2)**	**σ (KN/m^2)**
1.	0	0	0	0
2.	4.2	4.2	7.4	7.4
3.	10.2	10.2	11.7	11.7
4.	12.7	12.7	13.8	13.8
5.	16.9	16.9	25.5	25.5
6.	24.4	24.4	43.5	43.5
7.	40.3	40.3	61.5	61.5
8.	60.5	60.5	80.6	80.6
9.	81.7	81.7	98.7	98.7
10.	92.3	92.3	117.8	117.8
11.	99.7	99.7	128.4	128.4
12.	115.6	115.6	141.1	141.1
13.	118.8	118.8	158.1	158.1
14.	137.9	137.9	158.1	158.1
15.	155.9	155.9	157.0	157.0
16.	150.1	150.1	152.8	152.8
17.	143.2	143.2	151.8	151.8
18.	129.4	129.4	140.0	140.0
19.	120.9	120.9	136.9	136.9
20.	108.2	108.2	133.7	133.7

CRH: Cotton Stalk Charcoal Blended with Rice Husk, CSD: Cotton Stalk Charcoal Blended with Sawdust

In Fig. 4 it is shown that the hydrogen decrease from 4.14 to 2.89%. Hydrogen is mostly associated with volatile matter [23], hence a good briquette sample should have a low amount of hydrogen as in the produced briquettes of this research work, the available low hydrogen in the right proportion will enhance the combustibility of the briquettes samples, this makes the agricultural waste briquettes a good fuel.

In Fig. 4, rice husk has 4.15% nitrogen and 2.8% for cotton stalk blended with sawdust. Fig. 4 shows that the nitrogen content increased relatively in all sample briquettes from 2,8% to

4.15%. The slight increase in nitrogen content may be due to the biomass blend which contains more plant materials that have plant alkaloids, chlorophyll and other porphyrins containing nitrogen in cyclic structures. It can be said that it also draws nitrogen from the air in which it burns during combustion affirmed that nitrogen [17] has no calorific value, but low nitrogen is required in briquettes because it reduces oxidation, hence the low nitrogen in the briquettes samples, as compared to cotton stalk blended with sawdust (2.8%) briquettes will reduce oxidation.

Fig. 4, Table 10 shows that the briquette samples produced in this research work have low oxygen content with rice husk having 19.07% as the least, while cotton stalk blended with rice husk has 19,89%. In Table 8, it is shown that the oxygen content increased relatively in rice husk (19.07%) to cotton stalk blended with rice husk briquette (19.89%). The increase may be due to the blend of plant material that has higher oxygen content. This is significant because the more the oxygen content is it in the solid fuel, the better is its combustibility this results falls within the same range to that of Adekunle, et al. [17] findings. In Table 8, the sulphur content of the briquette samples is very low ranging from 1.03% in sawdust to 1.61% in cotton stal blended with rice husk, these results conform to that of Adekunle, et al. [17] In Table 8, the sulphur decreased relatively with an increase in plant material concentration in all the samples. The addition of calcium hydroxide used as a desulphurizer further reduced the sulphur content in all the briquette samples Sulphur is one of the major undesirable elements in briquettes even though it contributes to the heating value on combustion, it produces acids of sulphur dioxide and sulphur trioxide which corrodes combustion equipment and also cause atmospheric pollution. Since the sulphur in the briquettes is low as compared to woods and charcoal, it will be better as fuel because it will emit less sulphur dioxide to the atmosphere.

Table 8. Elemental determination of chemical elements of briquette samples by Liebig's and Kjeldahl methods

S/no	B.S	C(%)	H(%)	O(%)	N(%)	S(%)
1	Rice Husk	76.8	4.14	19.07	4.15	1.3
2	Sawdust	76.8	3.99	19.22	4.01	1.03
3	CRH	77.2	2.89	19.89	2.11	1.61
4	CSD	77.2	3.2	19.6	2.8	1.14

CRH: Cotton Stalk Charcoal Blended with Rice Husk, CSD: Cotton Stalk Charcoal Blended with Sawdust

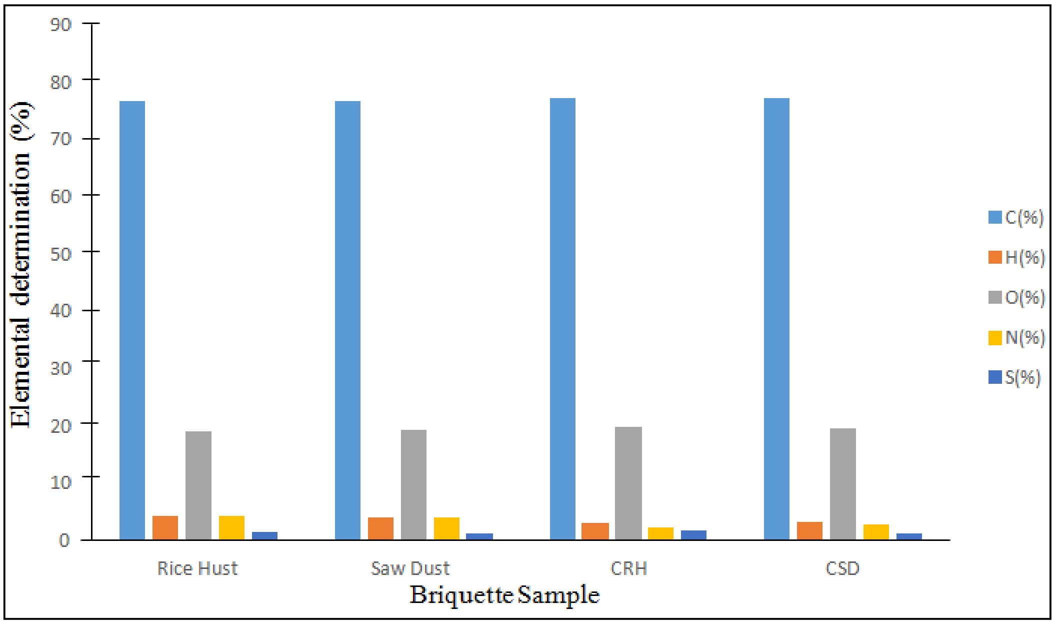

Fig. 4. The elemental determination of briquette samples by Liebig's and Kjeldahl methods

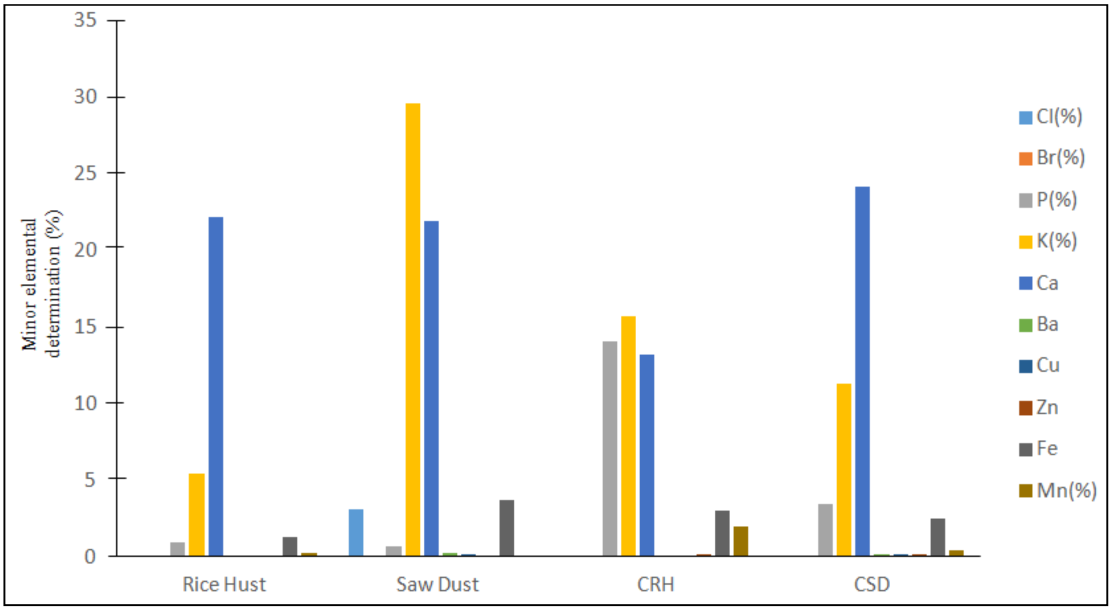

Fig. 5. The minor elemental determination of briquette samples by ED-XRFS methods

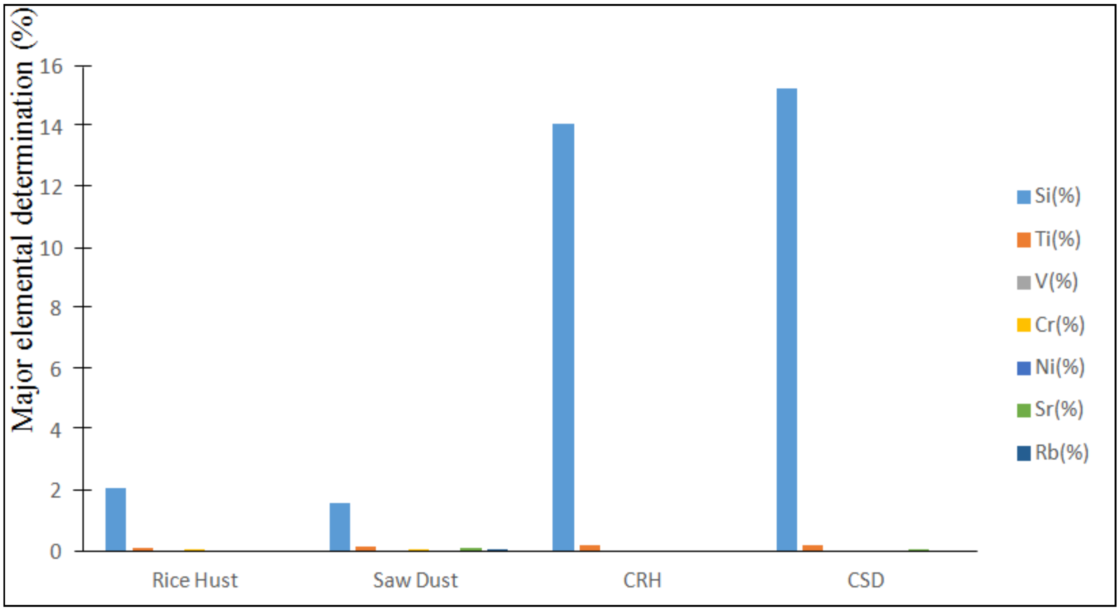

Fig. 6. The Major elemental determination of briquette samples by ED-XRFS methods

Table 9. Minor elemental determination of briquette samples by ED-XRF

S/no	B.S	Cl	Br	P	K	Ca	Ba	Cu	Zn	Fe	Mn (%)
1	Rice Husk	ND	ND	0.9	5.46	22.2	0.06	0.07	0.09	1.32	0.22
2	Sawdust	ND	ND	0.65	29.6	21.9	0.24	0.18	0.05	3.71	0.1
3	CRH	ND	ND	14.1	15.7	13.2	0.01	0.03	0.12	3.02	1.94
4	CSD	ND	ND	3.4	11.3	24.2	0.2	0.2	0.13	2.45	0.43

CRH: Cotton Stalk Charcoal Blended with Rice Husk, CSD: Cotton Stalk Charcoal Blended with Sawdust, ND = Non Detectable

Table 10. Major elemental determinations of briquette samples by ED-XRF

S/no	B.S	Si(%)	Ti(%)	V(%)	Cr(%)	Ni(%)	Sr(%)	Rb(%)
1	Rice Husk	2.1	0.11	ND	0.08	0.02	0.01	0.04
2	Sawdust	1.6	0.19	ND	0.08	0.03	0.14	0.1
3	CRH	14.1	0.22	ND	0.02	0.01	ND	ND
4	CSD	15.3	0.2	ND	0.05	0.04	0.1	ND

CRH: Cotton Stalk Charcoal Blended with Rice Husk, CSD: Cotton Stalk Charcoal Blended with Sawdust, ND = Non Detectable

Tables 9, and 10, show the minor, and major chemical composition analyzed by energy dispersed x-ray fluorescence spectrometer (ED-XRF). The composition of the briquettes is one of the major variables that contribute to the quality of briquettes compacts. The briquettes have low-molecular-weight substances such as organic matter, inorganic matter, and macromolecular substances include cellulose, hemicellulose, and lignin [24] Under-standing the major compositional changes that take place during briquetting can be useful in understanding their compaction behaviour [17].

Because of moisture and volatile matters loss due to temperature rise during briquetting, the chemical compositions of briquettes change slightly. The significant change in elemental contents was observed irrespective of the briquette. Among the elements, calcium had a Maximum of 24.2% in cotton stalk blended with sawdust and 13.2% in cotton stalk charcoal blended with rice husk, this is probably due to the presence of $Ca(OH)_2$ used as a pollution fixing agent. Recently, [24], reported similar observation and it was in agreement with the findings of Adekunle, et al. [17]. The change in ash content is inconsistent across the briquette samples, and similar results were reported for different briquette samples [1]. When compared to other feedstocks, cotton stalk charcoal blended with sawdust had high volatile matters that reflected on high smoke production, as well as a brown liquid, oozed out during briquetting, thereby more changes in the chemical composition including ash content. According to Dass, et al. [2] ash/mineral content of the feed stocks would show their relative abrasiveness to equipment when there is high friction/shear during densification; the higher the ash content, the higher the abrasion. Ash content of agricultural wastes and cotton stalk briquettes increased significantly, whereas sawdust and rice husks briquettes had a significant decrease. Dass, et al. [2], have also reported similar ash/mineral contents of agricultural wastes. Ash content of the agricultural wastes briquettes increased by 30.3 to 33.8%. Inorganic material, non-structural sugars, and nitrogenous material are water-soluble, whereas the results of the ultimate analysis carried out by ED-XRF on briquette samples are shown to have comparatively low level of Cl, Br, Ba, Cu, Zn and Mn, while P, K, and Fe are slightly in appreciable composition, this an indication that the ash contents of the produced briquette samples can serve as a fertilizer for crop production as reported by Theerarattananoon, et al. [24]. This is in agreement with the findings of this research work, which has similar values.

The toxic elements are shown in Table 4, depicted in Fig. 6. The results are observed to be very low which is an indication that no toxic emission will be evolved during combustion of the produced briquette samples, this is an indication that these briquette samples can serve as a solid fuel for domestic heating.

4. CONCLUSION

The produced briquettes in this research work provide better alternatives to wood, charcoal, fossil fuels, and for the production of eco-friendly solid fuel that can be used for domestic heat applications.

Agricultural waste is produced in large quantities and is disposed of indiscriminately most especially in the rural areas of developing countries, thereby causing a health hazard. The increased use of these wastes in briquette form, also help in solving disposal problem apart from providing alternative, good, cheap affordable, environmentally friendly, and cleaner sources of solid fuels to substitute wood, charcoal, and fossil fuels for cooking, warming, and household heating.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

REFERENCES

1. Nkafamiya II, Thliza BA, Dass PM. Production of cotton stalk charcoal/ biomass briquettes for use in low income households. Journal of Scientific nd Engineering Research. 2019;6(8):74-82.
2. Dass PM, Nkafamiya II, Thliza BA, Joseph JI. Proximate analysis of biomass and cotton stalk charcoal briquettes produced from Biu, Nganzai and Zabarmari in Borno State Using Locally Fabricated Briquetting Machine. International Journal of Progressive Science and Technologies. 2019;16(2):83-91.
3. Bala Adamu Thliza, Nkafamiya II, Dass PM, Edmond Moses. Determination of combustion efficiency and compressive stress of cotton stalk charcoal and agricultural wastes. International Journals of Sciences and High Technologies. 2019; 1(17):21-30.
4. Chesta Tiwari. Producing fuel briquettes from sugarcane waste EWB-UK. National Research and Education Journal. 2011; 220(550):39-45.
5. Vongsaysana. Comparison of the physical chemical properties of briquette charcoal and wood Charcoal. Environmental and Natural Resources Journal*.* 2009;7(3):12-24.
6. Yahaya DB, Ibrahim TG. Development of rice husk briquettes for use as fuel. Research Journal in Engineering and Applied Science. 2012;1(2):130–133.
7. Sikkem K, Steiner M, Junginger M, Hiegl W, Hansen M, Faaij A. The European wood pellet markets: Current status and prospects for 2020s. Biofuels, Bioproducts and Biorefining*,* 2013;5:250–278.
8. Imeh E, Onukak Ibrahim A, Mohammed-Dabo, Alewo O. Ameh, Stanley IR Okoduwa, Opeoluwa O. Fasanya. Production and characterization of biomass briquettes from tannery solid waste. 2017;2(17):33-90. Available:www.mdpi.com/journal/recycling
9. Tamilvanan A. Preparation of biomass using various agro-residues and waste papers. Journal of Biofuels*.* 2013;2(4):47-55.
10. Abdu Zubairu, Sadiq Abba Gana. Production and characterization of briquette charcoal by carbonization of agro-waste. Energy and Power. 2014; 4(2):41-47.
11. ASTM Standard D5373 –02. Standard test method instrumental determination of carbon, hydrogen and nitrogen in laboratory samples of coal and coke. ASTM International: West Conshohocken, PA; 2013.
12. Andrew Ndudi Efomah, Agidi Gbabo. The Physical, proximate and ultimate analysis of rice husk briquettes produced from a vibratory block mould briquetting machine. International Journal of Innovative Science, Engineering & Technology. 2015; 2:814– 822.
13. Ikele Issie Ikele, Ogah Sule Philip Ivoms. Determination of heating ability of coal and corn cob briquettes. IOSR Journal of Applied Chemistry. 2014;7(2):72-82.
14. Agu, Mathew Onyema, Hotton Joseph Anthony, Ukwubile Cletus Anes, Yakubu Shedrack. Proximate analysis of Smokeless briquettes using agro-waste (rice chaff, maize cob and sawdust). Journal of Materials Sciences and Applications. 2017;3(3):41–46.
15. Akpabio IO, Illalu A. Conversion of sugarcane waste into solid fuels. Nigerian Journal of Research. 2009;5(1-2):83– 87.
16. Wessapan T, Somsuk N, Borirak T. Design and development of a compact screw-press biomass briquetting machine for productivity improvement and cost reduction. In Proceedings of the First TSME International Conference on Mechanical Engineering, Ubon Ratchathani, Thailand. 2010;20–22.
17. Adekunle JO, Ibrahim JS, Kucha EI. Proximate and ultimate analyses of biocoal briquettes of Nigerian's Ogboyaga and Okaba sub-bituminous coal. British Journal of Applied Science and Technology. 2015; 7(1):114–123.
18. Akowuah JO, Francis K, Stephen JM. Physico-chemical characteristics and market potential of sawdust charcoal briquette. International Journal of Energy and Environmental Engineering. 2012; 3:20.
19. Jibrin Y, Ibrahim YV, Sadiq U, Agbo SA, Iliyasu U. Core power prediction for Nigeria Research Reactor-1 (NIRR-1) using measurements of dose-rate and

temperature. Progress Nuclear. Energy. 2016;91:256–264.

20. Jack Huang. How to make Briquette from daily waste. Renewable Energy World com. 2014;5:1-4.
21. Wang Q, Han K, Gao J, Li H, Lu C. The pyrolysis of biomass briquettes: Effect of pyrolysis temperature and phosphorus additives on the quality and combustion of bio-char briquettes, Fuel. 2017;199:488–496.
22. Gambo M, Salnjaba, Bala Adamu Thliza, Hassan Mshelia U. Determination of compressive strength and combustibility potential of coal and biomass briquette. International Journal of Research. 2017; 4(8):226-233.
23. Ryemshak S, Aliyu J. Proximate analysis, rheological properties and technological applications of some Nigeria coals. International Journal of Industrial Chemistry (IJIC). A Springer Open Journal. 2014;4:7.
24. Theerarattananoon K, Xu Wilson FJ. Effects of the pelleting conditions on chemical composition and sugar yield of corn stover, big bluestem, wheat straw, and sorghum stalk pellets. Bioprocess and Biosystems Engineering. 2012;35(4):615–623.

Peer-review history:
The peer review history for this paper can be accessed here:
http://www.sdiarticle4.com/review-history/53969