



Thermogravimetric analysis of agricultural residue carbonized briquettes for domestic and industrial applications

Vianney Andrew Yiga^{a,*}, Michael Lubwama^{a,b}

^a Department of Mechanical Engineering, Makerere University Kampala, Uganda

^b Africa Centre of Excellence in Materials, Product Development and Nanotechnology, MAPRONANO, Makerere University Kampala, Uganda

* Corresponding Author

Email: vyiga@cedat.mak.ac.ug

ABSTRACT

For a material to be used as energy, understanding its combustion behavior is crucial. Biomass is one such material that is increasingly gaining traction. Biomass may be utilized by direct combustion or transformation into fluid or solid biomass-based fuels. In this work, slow pyrolysis of groundnut shells, bagasse, rice husks and coffee husks was done to produce briquettes with cassava starch binder. Thermogravimetric analysis (TGA) was carried out using an Eltra Thermostep thermogravimetric analyzer. The samples were heated from ambient to 920 deg. Celsius. This analysis provided combustion explanations in terms of the weight loss, burning rates, peak temperatures, char residues and mean reactivity. TGA results showed that binder inclusion reduced the amount of fixed carbon present in the developed briquettes, thus slightly reducing their calorific values. Rice husks briquettes yielded the least weight loss (20.9% and 24.7% for 30g and 50g binder incorporations respectively) compared to others, owing to former's higher ash contents. Increase in binder contents reduced the amount of char residues, caused by reducing ash contents in the developed briquettes. Peak temperatures and char residues generally increased with increasing binder content. This signifies increasing thermal stabilities of the developed briquettes. Highest char residues were obtained by briquettes developed with rice husks at 30g binder while briquettes developed with bagasse briquettes developed at 50g binder had the least char residues. The highest mean reactivities were obtained in briquettes developed from bagasse and coffee husks while briquettes developed from rice husks had lowest mean reactivities. Briquettes developed in this

study showed sufficient combustion properties suitable to provide energy for domestic and industrial applications.

INTRODUCTION

Energy is among the most essential elements of human life. It can be used for heating, lighting, commercial, transport activity, industrial and most importantly for cooking. The current world population is over 7.3 billion, projected to reach 8.5 billion by 2030 and 11.2 billion by 2100. As the world population gradually increases, there is increasing energy demand, a trend which leads to rapid depletion of non-renewable fossil energy resources and consequent negative impacts of increasing fuel prices [1]. Biomass is a renewable material that is derived from plants. It constitutes one of the third largest alternative sources of primary energy in the world aside from coal and oil [2]. Lignocellulosic biomass consists mainly of three polymeric materials namely lignin, cellulose and hemicellulose. These polymeric materials strongly affect the properties of fuel derived from a given biomass material [3]. Lignocellulosic biomass, grown through photosynthesis comes from various sources including wood from trees/forests and agricultural residues. In Uganda, over 19 million tonnes of agricultural residues are generated annually from agricultural processing [4]. Often times, agricultural residues find only little or no productive utilization as energy sources. In fact, majority of these residues are disposed off using open burning [5]. One way to turn these residues into productive energy sources is by densifying into briquettes through carbonization/slow pyrolysis [6,7].

Briquette production from agricultural residues is not new. Recent studies in briquetting of agricultural residues include: Martinez et al., 2019 produced and characterized briquettes from coffee and pine combinations [8]. These briquettes had favourable energetic characteristics to increase the Brazilian energy matrix. Lubwama et al., 2019 developed briquettes from combinations of different agricultural residue biochars [9]. The developed briquettes had high mechanical strengths as well as necessary calorific values for domestic energy supply. Maize cob and bean straws have been blended to produce briquettes of high quality at low temperatures and large particle sizes. The quality was due to mechanical interlocking bonding in the agricultural residues [10]. Onifade et al., 2019 produced briquettes from corn cob residues. Their study was scoped around water resistance properties of the developed briquettes and factors affecting water absorption. Low die pressures, high hold time and low particle density were among the highlighted factors for high water resistance of briquettes [11]. In their study, Safdar et al., 2019 have produced briquettes from wheat straw, rice straw, maize straw and cotton sticks using an indigenously designed mechanical piston press. The produced briquettes had suitable bulk densities, durability, compressive strengths and calorific values [12]. Coffee grounds and sugarcane fibers have been utilized to produce briquettes with good calorific values ranging between 20.32MJ/kg and 21.92MJ/kg [13]. Kpelou et al., 2019 produced briquettes from coconut husks, palm kernel shells and corn cobs and tested their energy efficiency [14]. Their study concluded that binders enhance thermomechanical stability of briquettes.

Agricultural residue briquettes produce very low total greenhouse gas emissions compared to fossil fuels because the former are produced from carbon neutral materials. In fact, such materials have very low sulfur, nitrogen and ash which cause SO_x and NO_x [15,16]. For any material to be used as energy for domestic and industrial applications however, an understanding of its combustion behavior is very crucial. Many studies have used thermogravimetric analysis to study the combustion behaviour of briquettes [6,7,9,17-22]. Therefore, in this study, rice husks, coffee husks, groundnut shells and

bagasse were utilized as raw material in the development of carbonized briquettes and their combustion behavior was studied through thermogravimetric analysis.

2.1 EXPERIMENTAL

2.2.1 materials

Rice husks were collected from Tororo district, Eastern Uganda, coffee husks were sourced from Mubende district, Central Uganda, bagasse was obtained from Jinja sugarcane growers, Eastern Uganda, while groundnut shells were obtained from Gulu district in Northern Uganda. Cassava flour was sourced from Kasubi market in Kampala district.

2.1.2 preparation

Raw materials (rice husks, coffee husks, bagasse and groundnut shells) were sundried for 6-8 hours to reduce their moisture content to <13%. The dried raw materials were then fed into a 200L volume capacity carbonizer. It was made from a steel drum of height to diameter ratio 2:1, with 0.02m diameter holes on its surface [6,7]. The raw materials were then burnt in low air conditions by covering surface holes with mud at intervals, a process known as slow pyrolysis or carbonization which created bio-char. Carbonization was carried out at $\leq 450^\circ\text{C}$ and residence times were 4 hours. The obtained bio-char was measured into portions of 1000g which were then mixed with either 30g or 50g of cassava starch binder. Cassava starch binder was prepared by mixing 30g or 50g of cassava flour in water and bringing to boil, forming a homogeneous starchy solution. Cassava starch binder was then mixed with bio-char and compressed at 7MPa into cylindrical briquettes of 0.05m diameter and 0.05m height. Figures depicting the carbonization and briquetting setups have been presented in past works by the authors [7].

2.1.3 characterization

Thermogravimetric Analysis (TGA) was carried out using an Eltra Thermostep non-isothermal Thermogravimetric Analyzer, Haan, Germany. The decomposition analysis of the developed briquettes was performed under nitrogen atmosphere to avoid unwanted oxidation. Each measurement was carried out on about 1.2g of each sample over a temperature range from ambient to about 920°C at a heating rate of $16^\circ\text{C}/\text{min}$ [6,7,23]. TGA was used to determine moisture content (MC), ash content (AC), fixed carbon (FC) and volatiles (VM) in the developed briquettes. TGA also provided combustion explanations in terms of weight loss, burning rates, peak temperatures, char residues and mean reactivity. Burning rates and mean reactivities were calculated using Equations 1 and 2 respectively. Analytical ultimate analysis of Carbon, Hydrogen and Oxygen was obtained by models provided by Shen et al., 2010 using Equations 3-5 [24].

$$B_R = \frac{(W_i - W_0)}{t_i} \quad (\text{g}/\text{min}) \quad (1)$$

Where B_R is Burning rate, W_i is weight (grams) at time i , W_0 is initial weight (grams) and t_i is the time (minutes) it takes for the initial weight to reach W_i .

$$M_R = \frac{DTG_{\min}}{T_{\text{peak}}} \quad (\%/ \text{min}/^\circ\text{C}) \quad (2)$$

Where M_R is mean reactivity, DTG_{\min} is the maximum decomposition rate of change ($\%/ \text{min}$) in weight and T_{peak} is peak temperature ($^\circ\text{C}$).

$$C = 0.635FC + 0.460VM - 0.095AC \quad (\%) \quad (3)$$

$$H = 0.059FC + 0.060VM + 0.010AC \quad (\%) \quad (4)$$

$$O = 0.340FC + 0.469VM - 0.023AC \quad (\%) \quad (5)$$

Where *C* is Carbon (%), *H* is hydrogen (%) and *O* is Oxygen (%).

3.1 RESULTS AND DISCUSSION

3.1.1 Physical properties

The physical properties of the developed briquettes are shown in Table 1. The developed briquettes have fixed carbon contents ranging from 37.4% - 49.5% in rice husks briquettes with 30g binder and groundnut shell briquettes with 50g binder. Fixed carbon is positively linked to the calorific value of briquettes [6,7]. The developed briquettes contained only minimal moisture contents (3.5% - 9.3%). Moisture is less desired for briquettes because it contributes to reduction of calorific values of developed briquettes. The developed briquettes also had low values for volatile matter owing to the carbonization process that ensures that volatiles escape from the raw materials as temperature increases [25]. Due to the high amount of fixed carbons obtained in the developed briquettes, weight loss in the briquettes at the highest combustion temperatures are expected to be low and thus enhanced thermal stabilities [9]. Enhanced thermal stabilities are required to enhance both calorific values and boiling times, characteristics which are required for energy in both domestic and industrial applications.

Table 1: Physical properties of the developed briquettes

Raw material	Binder (g)	Fixed Carbon (%)	Moisture content (%)	Ash content (%)	Volatile matter (%)
Rice husks	30	38.3±1.4	3.5±2.3	39.6±0.1	18.7±1.0
Rice husks	50	37.4±0.3	6.2±0.0	36.6±0.2	19.8±0.5
Coffee husks	30	46.4±0.6	8.9±0.4	15.0±0.5	29.6±0.2
Coffee husks	50	43.1±1.3	9.3±0.2	16.3±0.9	31.3±0.6
Bagasse	30	49.4±0.6	6.1±0.0	11.9±1.4	32.5±0.7
Bagasse	50	48.7±1.0	6.6±0.0	10.3±0.3	34.5±0.9
Groundnut shells	30	49.4±0.6	8.0±0.2	13.8±3.6	28.8±3.2
Groundnut shells	50	49.5±2.9	7.0±0.0	21.5±1.8	21.9±1.1

3.1.2 Weight loss

Weight loss changes with time for the developed briquettes are shown in Figure 1. Rice husks briquettes yielded the least weight loss (20.9% and 24.7% for 30g and 50g binder incorporations respectively) compared to other briquettes, owing to their higher ash contents. Lubwama et al., 2019 attribute this to the inherent high silica contents in rice husks which when combusted form various forms of silicon dioxide which is a natural

flame retardant [9]. Bagasse and coffee husks briquettes had the highest weight loss due to their low ash compositions and higher volatile matter contents (see Table 1). Higher volatile matter compositions enhance ignitability which enhances burning rates and thus intensifying weight losses with increase in time [6,9]. In the first 80 minutes of the experiment, coffee husks reached highest weight reductions due to their higher moisture contents (see Table 1). It should be noted that increase in binder content had no striking effect of the thermograms.

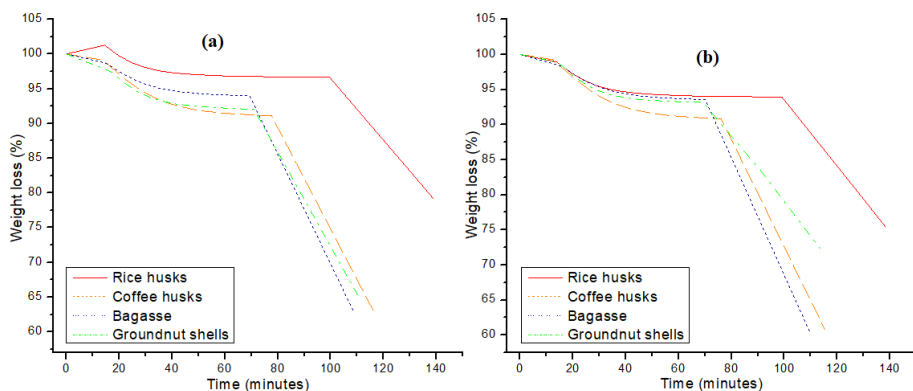


Figure 1: Weight loss for the developed briquettes with (a) 30g binder (b) 50g binder

3.1.3 Burning rate

Burning rates of the developed briquettes are shown in Figure 2. At the first 30 minutes of the experiment, moisture is being ejected from the developed briquettes [26]. Subsequently, coffee husks briquettes and groundnut shells briquettes reached higher rates of burning, followed by bagasse briquettes and rice husks briquettes. These rates went even higher when the ratio of binder content was increased. This is due to the fact that the binder material (cassava starch) used in this study is combustible in nature, compared to other inorganic binders like clay that would otherwise reduce the burning rates [6]. Between 60 and 80 minutes, the burning rates of coffee husks, bagasse and groundnut shell briquettes reduced before steeply increasing. For rice husks briquettes, this trend is noticed around the 100th minute of the experiment. At the tail end of the experiment, bagasse briquettes rise to have the highest burning rates reached (0.00398g/min and 0.0042g/min for 30g (see Figure 2a) and 50g (see Figure 2b) binder incorporation respectively), followed closely by coffee husks and groundnut shells. These high burning rates are due to lower ash and higher volatile matter contents, which have been noted to intensify ignitability with increase in time [6,9,23]. Rice husks briquettes however remain low at 0.001691g/min and 0.00203g/min for 30g and 50g binder incorporation respectively in the tail end of the experiment. The low rates for rice husks briquettes are due to the high moisture contents of rice husks which inhibit flow of heat across a given briquette [9]. In fact, combustion of rice husks during bio-char production and further combustion in the TGA experiment leads to formation of SiO₂ during oxidation of rice husks which creates a natural thermal resistance property it imparts on the developed rice husks briquettes [23]. This natural thermal resistance property retards combustion, thus reducing the burning rates of these briquettes during combustion [9].

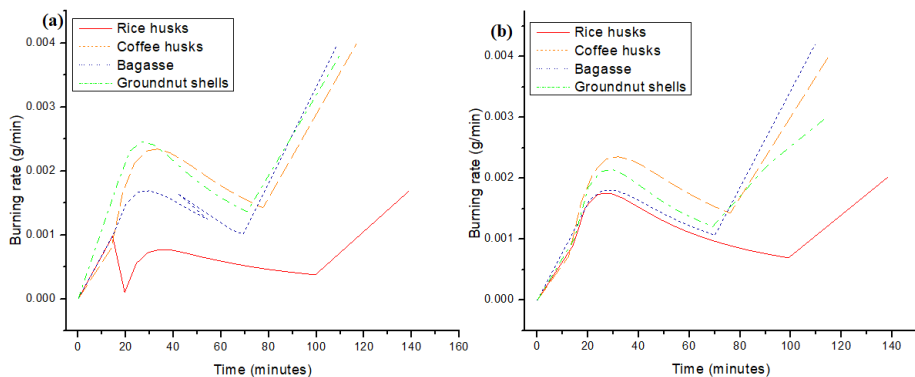


Figure 2: Burning acceleration for the developed briquettes with (a) 30g binder (b) 50g binder

3.1.4 Peak temperatures

Figure 3 shows temperatures at maximum weight loss (peak temperatures) for the developed briquettes. The peak temperatures of all the developed briquettes were similar at about 920°C except for groundnut shells briquettes with 50g of cassava starch binder which was 513°C. For the latter, maximum weight loss occurred earlier because of increasing ash contents with increasing binder, coupled with high volatile matter contents which enhance ignitability of the developed briquettes [6,7,9]. Peak temperatures are high because of the fact that char residues are high, accruing from the carbonization process of the raw materials which formed bio-char for briquette manufacture. Bio-char material is hard to combust compared to un-carbonized agricultural residues and thus the former leads to raises in temperatures at maximum weight loss of the developed briquettes.

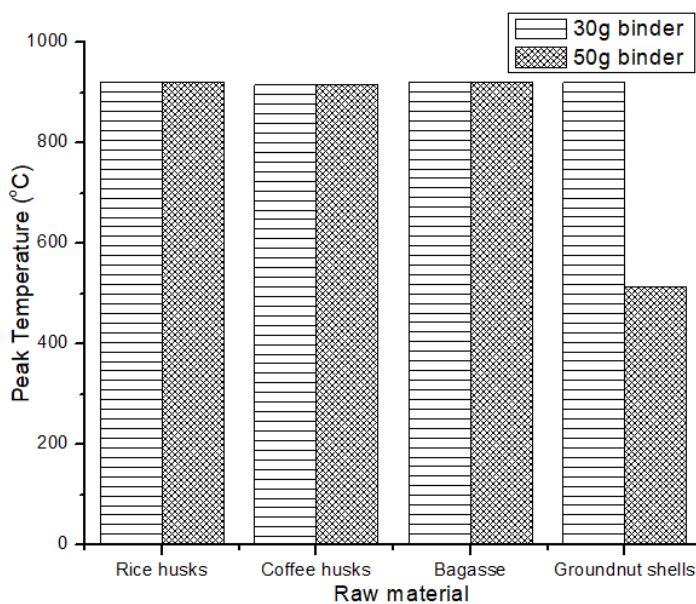


Figure 3: Peak temperatures for briquettes developed from different raw materials

3.1.5 Mean reactivity to char residues ratio

Char residues are material left out after the completion of the combustion process during thermogravimetric analysis. Reactivity analysis of the developed briquettes was obtained by the Ghetti method. Mean reactivity versus char residues plots gives an understanding of the time at which the peak temperature is reached during combustion of the developed briquettes (see Figure 4). Mean reactivities of the developed briquettes ranged between $1.54 \times 10^{-6} \%/\text{min}/^{\circ}\text{C}$ and $3.82 \times 10^{-6} \%/\text{min}/^{\circ}\text{C}$ for rice husks briquettes with 30g of binder and bagasse briquettes with 50g of binder respectively.

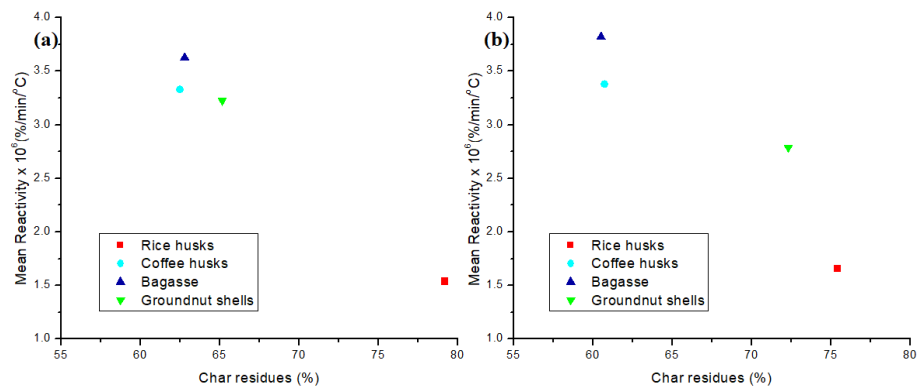


Figure 4: Mean reactivity vs char residues for the developed briquettes with (a) 30g binder (b) 50g binder

In Figure 4a, it is seen that the ratios are low as shown by the dense appearance of data points on the left-hand side of the figure, compared to the ratios in Figure 4b. It can be deduced that increase in binder amount causes an increasing trend in the ratio of mean reactivity to char residues. This increasing ratio is observed by the right-hand shift of points as binder contents are increased. As binder content increases, the amount of residues in the developed briquettes also increases, causing a reduction in the calorific values of the developed briquettes [6,7]. An important factor to note is that rice husks briquettes have high char residues and yet very low mean reactivities. This is expected since low mean reactivity signifies reduced combustion and thus increased production of ash contents in the developed rice husks briquettes which contribute greatly to the amount of char residues obtained (see Table 1). Additionally, the reduction can be attributed to high char conversion time which provides for production of multiple insulative layers in the subsequent bio-char combustion in rice husks briquettes [27].

3.1.6 Ultimate analysis

The carbon, hydrogen and oxygen contents of the developed briquettes are shown in Table 2. It is clear that the developed briquettes contained high carbon contents, mainly those made from coffee husks (41%), bagasse (45%) and groundnut shells (41%). The carbon content has a positive relation with calorific value of a given sample [28]. This explains why rice husks briquettes had low burning rates compared to other briquettes. For energy purposes, the H/C ratios should be as low as possible, reducing the emissions of gases and vapors [29]. This means that the briquettes developed in this study are burn sufficiently without producing many emissions to the atmosphere.

Table 2: Ultimate analysis of the developed briquettes

Raw material	Binder (g)	Carbon (%)	Hydrogen (%)	Oxygen (%)
Rice husks	30	29.16	3.78	20.88
Rice husks	50	29.38	3.78	21.16
Coffee husks	30	41.66	4.67	29.31
Coffee husks	50	40.22	4.58	28.96
Bagasse	30	45.19	4.98	31.76
Bagasse	50	45.82	5.05	32.50
Groundnut shells	30	43.31	4.78	29.99
Groundnut shells	50	39.46	4.45	26.61

CONCLUSIONS

Thermogravimetric analysis was used to study the combustion behaviour of the developed briquettes. This work confirmed that the developed briquettes can be suitably utilized as energy for both domestic and industrial applications. Knowing as moisture is less desired for briquettes because it contributes to reduction of calorific values of developed briquettes, the briquettes developed in this study contained only minimal moisture contents (3.48% - 9.29%).

Fixed carbon contents ranging above 37% are sufficient for provision of calorific values required to cause sufficient burning times as required for both domestic cooking applications and other industrial applications. These fixed carbons also contribute to increased char residues in the developed briquettes, signalling enhanced thermal stabilities of the developed briquettes. Briquettes developed from rice husks had the least weight loss (20.9% and 24.7% for 30g and 50g binder incorporations respectively) compared to other briquettes. This was attributed to their inherent silica in rice husks which when combusted during the carbonization process forms various forms of silicon dioxide which form multiple insulative layers against combustion.

Mean reactivities of the developed briquettes ranged between 1.54×10^{-6} %/min/ $^{\circ}$ C and 3.82×10^{-6} %/min/ $^{\circ}$ C for rice husks briquettes with 30g of binder and bagasse briquettes with 50g of binder respectively. Cassava starch binder was proposed to affect the mean reactivities owing to its low peak temperature during combustion. Rice husks briquettes have the least mean reactivities signalling production of multiple insulative layers during combustion and thus providing more char residues.

High peak temperatures obtained during combustion of the developed briquettes signify that these briquettes can be burnt for longer times and thus finding applications in industry especially in boilers.

Higher carbon contents compared to hydrogen and oxygen in the developed briquettes imply that the briquettes developed in this study can yield enough calorific values during combustion.

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