

Journal of Energy Research and Reviews

11(2): 36-62, 2022; Article no.JENRR.87375 ISSN: 2581-8368

Performance Evaluation of Raphia Palm (*R. vinifera***) Seeds Briquettes with Cassava Starch as Binder**

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Authors' contributions

This work was carried out in collaboration among all authors. Author BA designed the study, performed the statistical analysis, wrote the protocol and wrote the first draft of the manuscript. Authors JSI and AOE managed the analyses of the study, providing supervisory roles. Author KAK managed the literature searches to the extent of establishing a focus before his life was rudely cut short. All authors (with the exception of author KAK) read and approved the final manuscript.

Article Information

DOI: 10.9734/JENRR/2022/v11i230275

Open Peer Review History:

This journal follows the Advanced Open Peer Review policy. Identity of the Reviewers, Editor(s) and additional Reviewers, peer review comments, different versions of the manuscript, comments of the editors, etc are available here: https://www.sdiarticle5.com/review-history/87375

Original Research Article

Received 09 March 2022 Accepted 17 May 2022 Published 21 May 2022

ABSTRACT

The evaluation of the potentials of Raphia palm seeds briquettes as fuel source was carried out to obtain optimum particle size, binder ratio and compaction pressure with cassava starch as binder for domestic heating applications. The seeds were collected, dried, hammer-milled and sieved into four different particle sizes (1.18, 1.70, 2.36 and 3.35 mm) and were densified. Four compaction pressures (2.5, 5.0, 7.5 and 10.0 MPa) and four binder ratios (15, 20, 25 and 30 %) were used. Proximate analysis of the briquettes was carried out in a bomb calorimeter and their effects on the combustion properties of the briquettes evaluated using the least significant difference (LSD) by employing two-way analysis of variance. The results revealed that the briquette with particle size 3.35 mm, binder ratio 20 % and compaction pressure 2.5 MPa gave the highest energy value of 12,785 kJ/kg, fixed carbon of 10.91 %, volatile matter of 64.30 %, ash content of 11.85 % and moisture content of 12.86 % which was the best quality briquette compared to the other samples. A predictive model for the heat value of the briquettes was developed and found to be adequate (pvalue \leq 0.0001) for use to predict the heat value of the briquettes produced. Optimization of the energy value of the briquettes was also carried to determine the optimum heat value and the probability that the result is achievable was 100 %. The optimization process gave the heat value of

the briquettes to be 12,821.7 kJ/kg at the optimal condition value (24.12 % binder ratio, 1.18 % particle size and 3.00 MPa compaction pressure). The briquettes show good promise for use as fuel for household heating and small scale industrial applications.

Keywords: Binder ratio; briquettes; compaction pressure; energy value; particle size; Raphia palm seed.

1. INTRODUCTION

Energy is one of the necessities for human existence. In Nigeria energy availability is currently a great challenge both in the rural and urban areas with the rapidly increasing cost of cooking gas and kerosene, and environmental problems associated with firewood. Hence, concerted efforts are being made for an urgent transition to more sustainable, affordable and ecofriendly energy systems [1].

Currently, fossil fuel is the major source of energy from which the commonly used fuel products like kerosene and cooking gas are obtained [2]. However, the nonrenewability and the negative impact of fossil fuel on the environment such as the greenhouse gas emission has become a global concern [3]. Nigeria's over dependence on petroleum and its derivatives for domestic and industrial energy applications has led to instability in the prices of oil, gas and other sources of energy [4]. Most Nigerians living in the rural areas have depended solely on fuel wood for their energy needs for the past decades. Continual exploitation of fuel wood used in domestic heating applications would lead to deforestation, which causes soil erosion, floods, landslides etc.

Nigeria and other countries in the sub-Sahara are facing severe problem of forest degradation due to increased fuelwood consumption among other causes [5]. Out of the total energy demand in Nigeria, fuel-wood use account for about 37% [6]. In Nigeria, large quantities of agricultural residues such as the rice husk and sawdust are mostly dumped in waste sites unutilised and their recycling are rarely practiced which has led to environmental problems such as pollution [7]. Therefore, it is important to pay closer attention to renewable energy sources such as biomass from agricultural residues to reduce the problem of environmental pollution and deforestation [8]. However, direct use of biomass as a sustainable energy resource constitutes some disadvantages due to its high moisture, low energy value, heterogeneity and low density [9]. Direct combustion of biomass is not beneficial because

of the negative aspects coming from the intrinsic properties of biomass such as low density, low calorific value in a unit volume, high moisture, and storage, handling and transportation problems. From this point of view, it is important to develop strategies by which biomass is converted to secondary fuels which have better characteristics in comparison to the parent material [10].

Agro waste is the most promising energy resource for developing countries like Nigeria. The decreasing availability of fuel wood has necessitated that efforts be made toward efficient utilization of agricultural wastes. Fortunately, research has shown that a clean and affordable fuel source which is substitute to fuel wood can be produced by blending bio-mass (agricultural waste) [11]. Agricultural residues which sometimes pose challenges to the environment can be put to good use for providing energy for the teeming population of the world. Enormous quantity of agricultural residues and wastes are generated in Nigeria but they are poorly utilized and badly managed, most being left to decompose or burnt in the field resulting in environmental pollution and degradation. However, the use of these residues by directly burning them is associated with low thermal efficiency and wide spread air pollution that has been found to cause cataract among women that are exposed to the smoke and respiratory complications in children they carry on their backs when cooking with these biomass [12].

One of the simple techniques that is currently used to overcome some of the limitations to the direct usage of biomass as energy source is briquetting or densification. Briquetting is the mechanical compaction of dry, loose and tiny particle size materials with or without the addition of an additive to form a regular shaped solid through the application of pressure [13]. Compaction pressure plays a vital role in increasing the energy density and the combustion characteristics of a briquette, given the fact that denser solid fuel tends to burn for a longer time and more durable for handling and transportation [14].

So many research has been carried out in this area. Aliyu et al. [15] studied the effect of compaction pressure and biomass type (rice husk and sawdust) on some physical and combustion properties of briquettes. They used a simple manually operated briquetting machine suitable for rural communities without access to electricity supply with a 3- ton hydraulic jack and a pressure gauge to facilitate pressure variation. The results showed that the physical properties improved with increasing compaction pressure. All the produced briquettes at different compaction pressures from the different biomass exhibited over 90% shatter index while the briquette produced from RH at compaction pressure of 525.5 kN/m^2 had the highest combustion rate. The RH/SD briquette moulded at compaction pressure of 630.6 kN/m² had the least combustion rate. The ignition time of the briquettes increased with increasing compaction pressure from 1.28 to 1.58. However, the study found that the RH biomass briquette exhibited a superior solid fuel quality property compared to the other briquette samples.

Oluwaseyi et al*.* [16] characterized briquettes from forest wastes by employing an optimization approach. They used undesirable forest materials, such as jatropha seed shells and *Eucalyptus camaldulensis* wood shavings for production of briquettes with *Acacia senegal* as the binder in mixing proportions of 0:100, 25:75, 50:50, 75:25 and 100:0 and the binder varied from 50, 60, 70, 80 to 90 g. The briquettes had mean values of 0.66 kg \cdot m⁻³, 11.51, 91.12 and 99.7 % for the density, moisture content, water resistance and shatter index, respectively. They observed that the materials are potential organic wastes which could be used as a feedstock for the production of briquettes. Briquettes have also been produced from various agricultural residues and other waste materials [17-22]. However, there is no known report in literature on the evaluation of briquettes potentials of Raphia palm seeds as fuel source in Nigeria or elsewhere. Therefore, the aim of this study was to evaluate the briquettes potentials of Raphia palm seeds as fuel source.

Raphia palm is a monocotyledonous plant belonging to the family Palmaceae. It has a trunk covered with attractive unusual coils, usually reproduces through seeds and grows up to 10 m tall and 60 cm in trunk diameter. From scientific reports and investigations, it has been shown that the origin of Raphia palms is traceable to

West Africa, particularly along swampy and semi swampy area of tropical and equatorial rain forest or derived savannas [23].

Endemic to Africa, its distribution covered many countries of the tropical area like Cameroon, Burkina Fasso, Nigeria, Madagascar, Gambia, Ghana, Guinea, Ivory Coast, and Kenya. About 30 species are known among cited are *Raphia farinifera*, *Raphia sudanica*, *Raphia vinifera*, *Raphia regalis* and *Raphia hookeri* which is commonly distributed in West Africa. Raphia palm produces fruits that are oblong-ellipsoid in a scaly cone comprised of rhombus triangular reddish-brown scales. The fruits contain an important part called pulp or mesocarp which is considered inedible in some parts of the country. In addition, it is used as a bitter flavouring or occasionally as food, particularly when fresh. Due to its stomachic and laxative properties, it is used as medicine [24]. Every part of Raphia palm tree is useful economically, both in the food industry sector and the art sector. In the food industry sector, the mesocarp of the ripe raphia fruit pulp which is rich in many nutrients such as lipid (40-52%), protein (6.1%), carbohydrate (61.4%), vitamins such as niacin (0.27 mg), vitamin A (0.15 mg) and minerals (3%), as reported by Esiegbuya et al*.* [25] cannot only be used as food supplement but can also be a main source of lipid since it yields edible oil, which can be use and exploit as a cheap and local product which lead to a decrease of resource wasting and environmental pollution [26]. Although, studies have been done on *Raphia vinifera's* fruit, very few have interest on it seeds in terms of briquettes production as an alternative energy source. The plant and seed of *Raphia hookeri* are shown in Fig. 1.

Therefore, it is imperative that concerted efforts are made to address the domestic energy problems in Nigeria with the use of biomass briquettes that will drastically reduce the use of fuel wood. The production of briquette using Raphia palm seeds could mitigate pollution problems associated with the use of the raw biomass waste and this will also reduce the over dependence on fuel wood and petroleum derivatives for domestic heating applications.

The aim of the research is to evaluate the performance of Raphia palm seeds briquettes as a fuel source. It would help to document the optimum scientific procedure for densifying raphia seed residue generated in Nigeria. The results of this study would extend the knowledge in producing briquettes from tropical non timbre wood. It would help provide technology for producing briquettes in rural areas where energy scarcity is experienced. The study would provide better understanding of variables that influences briquetting of raphia palm seeds. It would help to reduce pressure on the forest by minimizing the usage of firewood and charcoal, as domestic and small scale industrial fuel would be substituted with briquettes. A shift from the use of petroleum products like kerosene, coal and liquefied petroleum product to the use of biomass briquettes as industrial and domestic fuel would help reduce the greenhouse effect since biomass is carbon neutral. It would help to diversify the sources of energy in Nigeria and therefore help to improve the energy security.

Fig. 1. Raphia palm plant and seeds [32]

1.1 Theoretical Background

A good quality briquette should produce sufficient, be smokeless to promote indoor air quality, and convenient for users. Raw material preparation affects the quality of briquettes with different briquetting machines requiring varying optimum raw materials conditions. Important raw material characteristics affecting briquette quality and selection of proper process conditions include moisture content, particle size, shape, and particle size distribution [27]. Combination of different biomass materials could improve the quality characteristics of briquettes. The parameters for measuring the briquette quality include calorific value, density, comprehensive strength, ash content, volatile matter content, ignition time, and burn time among others [28]. The calorific value of a briquette is the measure of its energy content, and a high value is desirable [29, 30]. Briquette density and compressive strength are influenced by material composition.

The most appropriate moisture content in biomass raw material for briquetting varies,

depending on the material and the process factors. Moisture present in the biomass material
facilitates starch qelatinization. protein facilitates starch gelatinization, denaturation and fibre solubilization processes during densification of biomass [31]. Steam-treated biomass is superior, as the additional heat modifies physiochemical properties to such an extent that binding between the particles is significantly enhanced, improving densification quality. Generally, moisture content in the range 6 to 16% is appropriate. Moisture beyond 16% even for smaller part of the raw material reduces briquette quality and eventually makes the process impossible. At high moistures (>20% w.b.), coherent biomass briquettes/pellets may not be produced because the cell structure remains largely intact due to the incompressibility of the biomass particles [20].

The particle size of a material is paramount in briquette making [33]. In general, density and durability of briquettes are inversely proportional to the particle size [20,34]. Medium or fineground materials are desirable in pelleting because they have greater surface area for moisture addition during steam conditioning, resulting in increased starch gelatinization and better binding. Finely ground materials will make very dense briquettes requiring high pressure and temperature to agglomerate without a binder. A certain percentage of fine to medium particles are required to improve pelleting efficiency and reduce costs. Generally, it is agreed that biomass material of 6 - 8 mm size with 10 - 20% powdery component (< 4 mesh) gives the best results [35]. The presence of different size particles improves the packing dynamics and also contributes to high static strength [36].

Studies on densified fuels derived from blends of two biomass materials indicate that the durability and mechanical strength of briquettes can be improved [37]. Physical and mechanical properties of mixed bio-coal briquettes have indicated that mixing ratio plays a significant role [38]. Briquetting material should be granular and uniform so that it can flow easily in bunkers and storage silos. It should also be easy for the material to flow. Cohesiveness is also an important characteristic of the biomass material. Lubricants and binders can impart these characteristics for compaction [35].

The process factors which affect briquettes' quality include temperature, pressure, preheating of raw material, cooling lines and nonhomogeneous distribution of particles. By varying the temperature of biomass, the briquette density, crushing strength and moisture stability are significantly influenced, with higher temperatures increasing pellet durability. However, temperature should not be increased beyond the decomposition temperature of biomass which is around 300° C [35, 39].

Compacting pressure plays an important role in the quality of briquettes/pellets. Low pressure systems such as manual presses (0.2 - 5.0 MPa) are only able to eliminate the voids between particles but incapable of raising the temperature or collapsing the cells within the particles. Increasing the compressing force results in increased density and binding force between the particles [40]. Briquettes produced at lower pressures (30 - 60 MPa) fall to pieces easily, but those at higher pressures (150 - 250 MPa) are consistent and compact. It has been reported pellets density is proportional to the natural logarithm of the applied pressure and that increase in pressure significantly increases density. Hence, briquetting pressure should be selected at an optimum value [41].

Preheating biomass before densification is widely used, as it results in the formation of more stable and dense pellets or briquettes. Tumuluru et al*.* [31] indicated that preheating biomass could significantly increase the throughput of the pelletizing machine and reduce the energy requirement per kg of products. Furthermore, they indicated that preheating biomass to temperatures between 100 and 130 \degree C improves its binding characteristics. Also, it has been reported that preheating to $200 - 225$ °C reduces wear and energy consumption in briquetting process because it softens the raw material before compaction, thereby reducing work and compaction pressure by a factor of two and increases screw life from 17 to 44 hours.

When a mechanical press is used for producing briquettes, the quality depends highly on the cooling and transport lines mounted on the machine. A briquette being pushed out of a press is very hot because of the friction in the nozzle. A hot briquette does not need substantial strokes or twists. The longer time the briquette can remain under pressure in the cooling line the longer and harder it will be. Cooling lines of 35 to 50 m in length are very common [8].

Two important things to consider during densification are the ability of the particles to

form pellets/briquettes with considerable mechanical strength and the ability of the process to increase density. The first is the type of bonding or interlocking mechanism could result in a better densified biomass. It has been suggested that the strength of pellets/briquettes formed depends only on the type of interaction and the material characteristics. The type of interaction include die diameter, die temperature, compacting pressure, usage of binders, and preheating of the biomass mix. The physical properties of biomass raw material include moisture content, density of the individual particles, bulk density, particle size, void volume and thermal properties. Furthermore, the chemical characteristics of the raw material of importance include the proximate and ultimate analysis, and the higher heating value. The physical properties of the biomass are very important in any description of the binding mechanisms of biomass densification [34]. It has been reported that the elastic and plastic deformation of the particles at higher pressures also contributes to compaction of biomass during densification. It has been also suggested that the possible mechanism of bonding during possible mechanism of bonding during densification of biomass could be due to the formation of solid bridges. The pressure applied reduces the melting point of the particles and causes them to move towards one another, thereby increasing the contact area and changing the melting point to a new equilibrium level [42]. Densification of biomass under high pressure brings about mechanical interlocking and increased adhesion between the particles, forming intermolecular bonds in the contact area [43].

Briquetting can be done with or without binder. Doing without the binder is more convenient but it requires sophisticated and costly presses and drying equipment which makes such process unsuitable in developing countries like Nigeria [44]. Binders are added to raw materials that cannot densify alone to form strong briquettes, resulting in enhanced bonding and more stable properties. Physical properties such as density, compressive strength and impact resistance index also show significant improvement due to binders [45, 46]. Despite the great variety of binders, starch binders enhance high quality briquettes. Cassava is a good binder because it has high starch content and is readily available. However, excessive use of cassava for briquette production has a negative impact on food security and therefore, its value should be optimized to minimize wastage [33,47,48].

Two main qualities of briquettes that need to be considered are that it shall remain solid until it has served its function and perform well as a fuel. The first aspect which implies that the product should be intact when handled or stored, is mainly a function of the quality of the densification process for a given raw material, and the second aspect is mainly related to the properties of the material, and the shape and density of the individual briquette [16].

The final moisture content of briquette or pellet made from biomass depends greatly on process conditions. Higher moisture content in the final product occurs when the initial moisture content is greater than 15%. ACCORDING TO BioGen/UK Code of Good Practice, ÖNORM M7135, SS 18 71 20, DIN 51731 AND CTI - R 04/5 the (British, Austrian, Swedish, German and Italian standards respectively) the moisture content of briquettes should be less or equal to 10, 18, 12, 12 and 15% respectively. Pellets with moisture content lower than 5% can result in revenue loss as they tend to break up during storage and transportation while those with high moisture content are subject to bacterial and fungal decomposition resulting in significant dry matter [49-51]. According to Ozyuguran and Yaman [52], the moisture content of the briquette can be determined by weighing a portion of a sample and oven drying it at 105 ℃ for three hours. The change in weight can then be used to determine the moisture content using equation 1.

$$
\% MC = \frac{w_1 - w_2}{w_1} \times 100\tag{1}
$$

where MC = Moisture content, w_1 = initial weight and w_2 = dry weight.

Pellets or briquettes with higher density are preferred as fuel because of their high energy content per unit volume and slow burning property. The density of briquettes is greatly influenced by the material's moisture content, particle size, process pressure and temperature, and on the original biomass density [34,41,53]. The German DIN 51731 defines briquettes density to be within the interval values 1000 - 1400 $kg/m³$. The weight is determined in the laboratory using a digital balance and the volume by a simple calculation based on direct measurement of the dimensions of the briquettes [54]. The density of a material is define as the mass per unit volume of that material as shown in equation 2.

$$
\rho = \frac{m}{v} \tag{2}
$$

where $m =$ weight of charcoal briquettes in kg, $v =$ Briquettes volume in m³ and $p =$ density of charcoal briquette in kgm⁻³.

Stability of briquette refers to the changes in its dimensions after removing it from the die, and it arises from the pressure lose by the humidity escape as steam [20,55]. Stability serves as an index of the extent of resistance of briquettes to changes in their initial physical dimensions and shape. It is desirable that they maintain their initial state. Plíštil, et al*.* [56] reported no appreciable expansion in length after 5 hours and negligible radial expansion. Tumuluru et al*.* [27] used statistical analysis of rice husks to establish a multiple correlation equation as depicted in equation 3.

$$
Y = \alpha_0 + \alpha_1 P + \alpha_2 T \tag{3}
$$

where $Y =$ percent volume expansion, T ($^{\circ}$ C) and P (kg/m^2) = die temperature and pressure, respectively. α_0 , α_1 and α_2 are constants. Hotpressing temperature during briquetting significantly facilitates solidification and declines expansion of the briquette.

Mechanical properties of briquettes, known as its durability refers to the ability of the briquette to withstand mechanical handling. It is probably the most important criterion for evaluating the quality of densified biomass and this test is intended to assess the ability of product to withstand the rigors of handling keeping their mass, shape, and integrity. Compressive strength, impact resistance index, tensile strength and hardness are some of the mechanical properties relevant to the durability of briquettes. Materials with higher density are more likely to possess higher ultimate stress [57,58].

The mechanical strength of briquettes depend on the properties of the raw material, its structure, compaction pressure and moisture content. The higher the compacting pressure the higher the compressive strength [15,56]. Briquette quality can also be evaluated in terms of its hardness. Harder briquettes are of better quality and hardness of briquettes is related to its elastic and plastic properties. The harder the briquette the higher will be its breaking strength. It is possible to check briquettes' hardness by inserting it into a glass of water. A quality briquette should fall to the bottom in a moment because it has a higher specific density than water. Next, when the briquette falls into pieces sooner than in 5 minutes, its quality is very low, before 15 minutes, medium and up to 20 minutes good quality [20,59].

Impact resistance index of briquette is its ability to withstand shock load. Briquettes with impact resistance index value equal to 100 or more are considered as good briquettes. Additionally, according to the Italian standard for briquettes/pellets (CTI-R04/5), durability greater or equal to 97.7% is adequate. Generally, researchers have classified the impact resistance index into high (> 0.8) , medium $(0.7 - 0.8)$ and low (< 0.7) [60,61].

The volatile matter, ash content, fixed carbon and calorific value of biomass are major parameters considered for determining the thermal characteristics of briquettes. These properties also depend on the biomass used [62]. High proportion of volatile matter has been attributed to high proportion of organic matter in a biomass material. Materials with relatively high volatile matter indicate easy ignition, fast burning and proportionate increase in flame length. Some biomass generally contain volatile matter of around 70 - 80% with low char content [63,64]. The percentage volatile matter of briquettes can be determined by keeping a portion in an oven until a constant weight is obtained. The over dried sample is then kept in the muffle furnace at a temperature of 550 ℃ for 10 minutes after which the volatile matter in it is allowed to escape, the crucible allowed to cool in a desiccator and weighed to obtain the mass of volatile parts of the sample [52,65]. The percentage of volatile matter can be computed by using the equation 4.

$$
\% \, VMC = \frac{w_2 - w_3}{w_3} \times 100 \tag{4}
$$

where VMC = volatile matter, w_2 = dry weight and w_3 = weight of sample.

Ash is the non-combustible component of biomass and it influences heat transfer to the surface of a briquette and diffusion of oxygen to the fuel surface during char combustion. High ash content results into dust emissions which lead to air pollution and affects the combustion volume and efficiency. The higher the fuel's ash content, the lower its calorific value [33, 66]. Furthermore, ash content of different types of

biomass is an indicator of slagging behavior of the biomass. Usually slagging takes place with biomass fuels containing more than 4% ash and non-slagging fuels with ash content less than 4% [69]. According to Babajide et al*.* [12], 2 g of oven dried pulverized briquette should be placed in a crucible (w_2) . The crucible should then be placed in the furnace for 4 hours at 550 ℃ to obtain the ash weight (w_4) . The ash content can then be calculated using equation 5.

$$
\% \text{ Ash content} = \frac{w_4}{w_2} \times 100 \tag{5}
$$

where w_4 = ash weight and w_2 = dry weight.

Fixed carbon of the briquette is a percentage of carbon (solid fuel) available for char combustion after volatile matter is distilled off or lost to the atmosphere. Therefore, fixed carbon gives a rough estimate of the heating value of fuel and acts as the main heat generator during burning [64]. According to Babajide et al*.* [12], the percentage fixed carbon can be calculated by subtracting the sum of percentage volatile matter and percentage ash content from 100% as depicted in equation 6.

$$
Fixed carbon (%) = [100 - (VMC + Ash)]\n% \tag{6}
$$

Calorific value is the amount of heat released by the combustion of a mass of fuel. The calorific value of biomass-briquette is enhanced by the type of binder used. The shape of briquettes has also been observed to enhance the calorific value of briquettes. Hollow briquettes are reported to give better combustion properties, the hole(s) helping air circulation for continuous burning [33, 68]. The calorific value can be determined based on ASTM D5865. The specific heat of combustion can be calculated from equation 7 [12].

$$
SHC = 0.35(147.6 \times \%FC) + (144 \times
$$

\n
$$
\%VMC + \%Ash
$$
 (7)

where $SHC = Specific heat of combustion$, $FC =$ Fixed carbon, $VMC = Volatile matter and Ash =$ Ash content.

Ultimate analysis involves the determination of carbon, oxygen, hydrogen, nitrogen, sulphur and ash, and can be determined based on the American Society of Testing and Materials (ASTM) D3178, D3179 and D3177 standards.

The carbon content is determined by using equation 8 [69].

 (8)

where FC = Percentage fixed carbon content (%), VMC = Percentage volatile matter (%) and MC = Percentage moisture content.

The hydrogen content can be determined using equation 9 [70].

$$
HC(\%) = [(0.036FC) + 0.086(VMC - 0.1Ash) - (0.0035MC2)(1 - 0.02MC)]
$$
\n(9)

where $HC = Hydrogen content$, $FC = Fixed$ carbon, VMC = Volatile matter, $MC =$ Moisture content and AS = Ash content.

The nitrogen content is determined using equation 10 [70].

$$
Nitrogen content (\%) = 2.10 - 0.020VMC
$$
\n(10)

where VMC = Volatile matter content.

The oxygen content can be determined using equation [70].

Oxygen content (%) =
$$
100 - (C + H + N +
$$

Ash) (11)

The sulphur content can be determined by igniting 1 g of sample and two portions of calcium and magnesium oxide with the other portion in an anhydrous sodium carbonate. The sulphur is then dissolved in water and precipitated as barium sulphate. The precipitate is filtered, and the ash content of the precipitate determined and weighed. The sulphur content can then be calculated using equation 12 [71].

Subflux content (%) =
$$
\frac{A-B}{C}
$$
 × 13.74 (12)

Where $A =$ mass of barium sulphate from sample (g), $B =$ mass of barium sulphate from blank (g) and $C =$ mass of sample (g).

2. MATERIALS AND METHODS

The biomass material used for this study were Raphia palm seeds using cassava starch as the binder. The seeds were collected from dump sites at Tyowanye in Buruku Local Government Area, Benue State and processed at Joseph Sarwuan Tarka University, Makurdi. They were sun dried for seven days to reduce excess moisture after which it was hammer milled. The seed residues was then sieved into 4 particle sizes of 1.18, 1.70, 2.36 and 3.35 mm based on ASTM E11-Sieve designation.

Cassava starch was prepared following the method used by Owuamanam et al. [72]. The tubers were well peeled, washed with clean water and grated to obtain a smooth slurry. The slurry was further mixed with water to form free flowing slurry which was then filtered using a muslin cloth. The filtration continued until all the starch was extracted and the woody mass discarded. The filtrate was allowed to settle in the plastic bucket before the supermatant, the surface of the starch was mashed with clean water to obtain white-odourless starch. The thick starch paste was scooped into a clean calico bag and pressed to dewater, crumbed within the palm, sun-dried to obtain cassava starch and packaged in an air tight container. The starch prepared was used as binder in this study.

4 binder ratios of 15, 20, 25 and 30% of weight of sample were used in order to determine the effect of binder concentration on physical and chemical characteristics of briquettes produced from Raphia palm seeds [70,73]. Cassava starch was chosen as binder because of availability, ease of preparation and low cost.

The briquettes were produced following the method used by Babajide et al. [12]. The Raphia palm seed residue of particular particle size was put in a container. Cassava starch was prepared with 100 $cm³$ of hot water and appropriate quantity based on the binder ratio was then added. Water was sprinkled until a homogeneous mixture was achieved and then hand fed into a cylindrical mould of 40 mm height and 50 mm diameter. The mixture was then placed under a hydraulic press and varying pressures of 2.5, 5.0, 7.5 and 10.0 MPa were separately applied. A dwell time of 3 minutes was allowed before the samples were extruded from the mould and left to dry at atmospheric temperature. Table 1 shows the briquette samples that were produced with varying particle sizes (PS), binder ratios (BR) and compaction pressures (CP). Fig. 2 shows some of the samples.

Fig. 2. Some of the briquette samples

The compressive strength of the briquettes was determined using a strength testing machine with load cell capacity of 100 kN in accordance with ASTM D 143. The crosshead speed was 0.305 mm/min. Only compact and intact briquettes were used for this test. A sample of briquette to be tested was placed horizontally in the compression test machine and subjected to loading. 4 replicates were made and compressive strength calculated by employing equation 13.

$$
CS(Nmm^{-2})
$$

=
$$
\frac{Max.force \ applied (N)}{Mean \ area \ of \ sample \ face \ (mm^2)}
$$
 (13)

The impact resistance index of the briquettes was determined in accordance to ASTM D440-86 methods of drop shatter developed for coal. The test was conducted two weeks after producing the briquettes. A test sample of briquettes of known weight (w_1) was placed in a plastic polythene bag. The bag was thrown onto concrete floor three times from a height of 2 m. After dropping, the briquettes and fractions were placed on top of a 35 mm square mesh screen and sieved. The durability rating for each type of briquette is expressed as the ratio of weight of material retained on screen (w_2) to weight of briquette before dropping. The handling durability of the briquettes was computed using equation 14 [74].

Import Resistance Index =
$$
\frac{w_2}{w_1}
$$
 (14)

5 briquettes samples were selected and water resistance quality was determined using the German Standard DIN 5173. This was done by immersing a briquette into a container filled with water at room-temperature. The time taken for the briquette to completely disintegrate was determined using a stop watch. Each experiment was replicated four times and the mean time computed.

The density of the briquette samples were determined in accordance with ASTM D 2395 - 07a. Four specimens were prepared from the briquette and then oven-dried, dipped one-byone into paraffin wax and then kept in a desiccator. The volume displacement method which employs the use of a Eureka can and a measuring cylinder were used to determine the volume of the briquette and the density was computed with 4 replicates. The mass was obtained by weighing the briquette on the digital weighing scale. The density after drying (relaxed density) was computed as the ratio of the measured mass to the calculated volume by employing equation 2.

The proximate analysis was done based on ASTM D5865 to determine the percentage of moisture content, volatile matter, fixed carbon, ash content and heating value of the briquettes. It was carried out using a bomb calorimeter. The percentage moisture content was determined by measuring 2 g (w_1) of pulverized briquettes into a crucible. The content was dried in an oven at 103 ^oC for two hours to obtain oven dry weight (w_2) [75]. The dried sample were kept in a desi**c**cator to prevent moisture gain before weighing. Moisture Content was then calculated by employing equation 1. The volatile matter was determined by placing 2 g pulverized briquette sample in a crucible with dry weight (w_2) in the furnace for 10 minutes at 550 °C to obtain charred weight [52]. The percentage volatile matter was then calculated using equation 4. Percentage ash content was determined by keeping the charred weight (w_3) in the furnace for 3 hours at 600 °C to obtain ash weight (w_4). The percentage ash content was determined using equation 5. The percentage fixed carbon was computed by subtracting the sum of percentage volatile matter and percentage ash content from 100 using equation 6, and the heating value was calculated using equation 7.

Sample	PS (mm)	BR (%)	CP (MPa)	Sample	PS (mm)	BR (%)	CP (MPa)
A ₁	1.18	15.0	2.5	\vert 1	1.18	15.0	7.5
A2	1.18	20.0	2.5	12	1.18	20.0	7.5
A ₃	1.18	25.0	2.5	13	1.18	25.0	7.5
A4	1.18	30.0	2.5	4	1.18	30.0	7.5
B1	1.70	15.0	2.5	J ₁	1.70	15.0	7.5
B2	1.70	20.0	2.5	J2	1.70	20.0	7.5
B ₃	1.70	25.0	2.5	J3	1.70	25.0	7.5
B4	1.70	30.0	2.5	J4	1.70	30.0	7.5
C ₁	2.36	15.0	2.5	K ₁	2.36	15.0	7.5
C ₂	2.36	20.0	2.5	K ₂	2.36	20.0	7.5
C ₃	2.36	25.0	2.5	K ₃	2.36	25.0	7.5
C ₄	2.36	30.0	2.5	K4	2.36	30.0	7.5
D ₁	3.35	15.0	2.5	L1	3.35	15.0	7.5
D ₂	3.35	20.0	2.5	L2	3.35	20.0	7.5
D ₃	3.35	25.0	2.5	L ₃	3.35	25.0	7.5
D4	3.35	30.0	2.5	L4	3.35	30.0	7.5
E1	1.18	15.0	5.0	M ₁	1.18	15.0	10.0
E ₂	1.18	20.0	5.0	M ₂	1.18	20.0	10.0
E ₃	1.18	25.0	5.0	M ₃	1.18	25.0	10.0
E ₄	1.18	30.0	5.0	M4	1.18	30.0	10.0
F ₁	1.70	15.0	5.0	N ₁	1.70	15.0	10.0
F ₂	1.70	20.0	5.0	N ₂	1.70	20.0	10.0
F ₃	1.70	25.0	5.0	N ₃	1.70	25.0	10.0
F4	1.70	30.0	5.0	N ₄	1.70	30.0	10.0
G ₁	2.36	15.0	5.0	O ₁	2.36	15.0	10.0
G ₂	2.36	20.0	5.0	O ₂	2.36	20.0	10.0
G ₃	2.36	25.0	5.0	O ₃	2.36	25.0	10.0
G4	2.36	30.0	5.0	O ₄	2.36	30.0	10.0
H1	3.35	15.0	5.0	P ₁	3.35	15.0	10.0
H2	3.35	20.0	5.0	P ₂	3.35	20.0	10.0
H ₃	3.35	25.0	5.0	P ₃	3.35	25.0	10.0
H ₄	3.35	30.0	5.0	P ₄	3.35	30.0	10.0

Table 1. Coding of the Briquette Samples

The ultimate analysis of the briquettes involves the determination of the weight percentage of carbon, hydrogen, nitrogen, sulphur and oxygen. The first four elements are determined directly but the amount of oxygen was estimated by difference. The percentage of carbon (% C) was determined using equation 8, percentage of hydrogen (% H) using equation 9, percentage of nitrogen (% N) using equation 10 and percentage of sulphur (% S) using equation 12. The percentage of oxygen (% O) was then calculated by deducting the sum total of % C, % H, % N and % S from 100 using equation 11.

Response surface methodology (RSM) was used to predict the optimum heating value of the briquette samples using Design Expert 8.0.6 software, employing the approach of Chukwuneke et al. [59]. The input variables considered were particle size, binder ratio and compaction pressure. The output was the

heating value of the briquette samples and it was modeled based on coded and actual factors. The data was analyzed using two-way analysis of variance (ANOVA) to investigate the effect of the experimental factors on the quality characteristics of briquettes produced with the aid of the computer software Statistical Package for the Social Sciences (SPSS version 20).

3. RESULTS AND DISCUSSION

Table 2 shows the effect of particle size (PS) on the combustion properties of briquettes, using least significant difference (LSD). The effect on ash content (AC), moisture content (MC) and volatile matter (VMC) of the briquettes from the LSD are also shown in Fig. 3. The result shows that the AC of 18.20% for PS of 2.36 mm is significantly different ($p \le 0.05$) from the value of 16.20% for PS 3.35 mm. It is also evident from the result that the value of 16.20% for PS 3.35 mm is significantly different ($p \le 0.05$) from 12.75% for PS 1.18 mm, but 12.75% AC for PS 1.18 mm is not significantly different ($p \ge 0.05$) from 12.25% for PS 1.70 mm. It can be concluded that the fine PS of 1.70 mm gave the lowest AC for the briquettes. Finer grain particles are expected to ignite faster and have a speedy combustion. The faster combustion rate will allow them burn to ash especially more than the bigger PS samples in agreement with the report of Chin and Shiraz [76].

Table 2 and Fig. 3 also show the variation in percentage of fixed carbon (FC) of the briquettes with PS. The results show that the FC of 4.24% for PS size 1.18 mm is significantly different $(p \le 0.05)$ from the value of 3.47% for PS 1.70 mm. It is also evident that the value of 3.47% for PS 1.70 mm is not significantly different ($p \ge$ 0.05) from the value of $3.17%$ for PS 2.36 mm. Also, the value of 3.17% for PS 2.36 mm is not significantly different ($p \ge 0.05$) from 2.95% for particle size 3.35 mm. The percentage of FC content in briquettes is a critical factor that influences the calorific value of the fuel. The general trend shown in Fig. 2 is a reduction with increasing PS [17].

Also, the effect of PS on moisture content (MC) are presented in Table 2 and Fig. 3. The result showed that the MC of 15.60% for PS of 1.18 mm is not significantly different ($p \ge 0.05$) from the value of 15.48% for PS 1.70 mm. However, the MC of 14.96% for PS 2.36 mm is not significantly different ($p \ge 0.05$) from the value of 14.55% for PS 3.35 mm. But the value of MC of 15.60% for PS 1.18 mm is significantly different $(p \le 0.05)$ from the value of 14.55% for PS 3.35 mm. The MC ranges from 15.60% to 14.55% and it can be concluded that the moisture content was in satisfactory range for briquetting [77].

Fig. 3. Effect of Particle Size on Ash Content, Fixed Carbon and Moisture Content of Briquette

Table 2 and Fig. 4 show the variation in heat value (HV) of the briquettes with change in PS. The result shows that the HV of 11,161 kJ/kg for particle size 1.18 mm is not significantly different $(P \ge .05)$ from the value of 11,115 kJ/kg for PS of 1.70 mm. However, the value of 11,115 kJ/kg for PS of 1.70 mm is significantly different ($P \le 0.05$) from 10,489 kJ/kg for PS 3.35 mm and the value of 10,489 kJ/kg for PS 3.35 mm is not significantly different (P \geq .05) from 10.247 kJ/kg for particle size 2.36 mm. It can be concluded that particle size 1.18 mm gave the best heat value. The calorific value of 11.78 MJ/kg obtained for teak leaves briquette by Madhurjya and Deben [78] compare reasonably with the HV for this study. The high heating values of the briquettes was between 10,247 to 11,161 kJ/kg. This energy value is sufficient to produce heat required for household cooking and small scale industrial heating applications [78].

Fig. 4. Effect of particle size on heat value of briquette

The effect of the PS on volatile matter content (VMC) is shown in Figure 5 and Table 2. The results showed that the VMC of 68.92% for PS 1.70 mm is not significantly different ($P \ge 0.05$) from the value of 67.38% for PS of 1.18 mm. It is also evident from the results that the value of 67.38% for PS 1.70 mm is significantly different (P ≤ .05) from 65.87% for PS of 3.35 mm. The value of 65.87% for PS 3.35 mm is significantly different (P \leq .05) from 63.59% for PS of 2.36 mm. It can be concluded that the particle size 1.70 mm gave the highest VMC of 68.92% for the briquettes. It was observed that briquettes with fine particles had higher volatile matter. This is high and signifies easy ignition of the briquettes and proportionate increase in flame length as suggested by Loo and Koppejan [79]. The 68.92% VMC recorded is comparable with values obtained for other biomass in previous studies [45, 46].

PS	AC (%)	FC (%)	HV (kJ/kg)	MC(%)	VMC(%)
1.18	12.75	4.24	11161	15.60	67.38
1.70	12.25	3.47	11115	15.48	68.92
2.36	18.20	3.17	10247	14.96	63.59
3.35	16.20	2.95	10489	14.55	65.87
LSD ($P \le .05$)	0.46	0.19	136.8	0.71	0.90

Table 2. Effect of Particle Size (PS) on combustion properties of briquettes

Fig. 5. Effect of particle size on volatile matter of briquette

The results in Table 3 shows the effect of binder ratio (BR) on the combustion properties of briquettes, using least significant difference (LSD). Table 3 and Fig. 6 show the variation in AC of the briquettes with change in BR. The result showed that the AC of 15.12% for BR of 20% is not significantly different (P ≥ .05) from the value of 15.01% for BR of 15%. It is also evident from the result that 15.01% for BR of 15% is not significantly different ($P \ge 0.05$) from value of 14.89% for 25% BR. However, 14.89% for 25% BR is not significantly different ($P \ge 0.05$) from the value of 14.41% for 30% BR. Low AC offers high HV for briquettes [80]. 30% BR recorded the least AC of 14.41% and the highest AC was recorded at 20% BR. High AC is said to reduce ignitibility of briquettes [67]. It can be concluded from these results that the BR 30% gave lower ash content and that sample will exhibit better ignition.

The effect of BR on fixed carbon (FC) using least significant difference (LSD) are confirmed by Fig. 6. The results showed that the FC of 3.51% for BR of 15% is not significantly different ($P \ge 0.05$) from the value of 3.49% for BR 25%. It is also evident from the results that the value of 3.49% for BR 25% is not significantly different ($P \ge 0.05$)

from 3.46% for BR of 30%. However, 3.46% for BR of 30% is not significantly different ($P \ge 0.05$) from 3.37% for BR of 20%. The fixed carbon were not significantly different from each other (P ≥ .05) as also shown by the relatively horizontal trend in Fig. 6. The low FC content tends to prolong cooking time by its low heat release and is an advantage for the briquettes [81].

Fig. 6. Effect of Binder Ratio on Ash Content, Fixed Carbon and Moisture Content of the Briquettes

Fig. 6 and Table 3 also show the variation in MC of the briquettes with change in BR. The results show that the value of 15.29% for BR 25% is not significantly different ($P \ge .05$) from the value of 15.22% for BR 30%. It is also evident from the results that the value of 15.22% for BR 30% in turn is not significantly different ($P \ge .05$) from 15.04% for BR 20%. Also, the MC of value 15.04% for BR 20% is not significantly different $(P \ge .05)$ from 15.03% for binder ratio 15%. It can be concluded from the results that MC was in the satisfactory range in agreement with Raju et al. [81] and do not vary significantly with BR as confirmed by Fig. 5.

Table 3. Effect of Binder Ratio (BR) on combustion properties of briquettes

BR	AC (%)	FC (%)	HV (kJ/kg)	MC (%)	VMC(%)
15	15.01	3.51	10754	15.03	66.31
20	15.12	3.37	10662	15.04	65.99
25	14.89	3.49	10759	15.29	66.41
30	14.41	3.46	10837	15.22	67.03
LSD ($P \le .05$)	0.46	ΝS	NS	ΝS	ΝS

Fig. 7 and Table 3 show the variation in HV of the briquettes with change in BR. The result showed that the heat value of 10,837 kJ/kg for BR of 30% is not significantly different (P \geq .05) from the value of 10,759 kJ/kg for BR of 25%. It is also evident from the results that the value of 10,759 kJ/kg for BR of 25% is not significantly different (P \geq .05) from 10,754 kJ/kg for BR of 15%. However, the value of 10,754 kJ/kg for BR of 15% is not significantly different from 10,662 kJ/kg for BR of 25%. The values obtained indicated that the heat value increased with increase in BR. The cassava starch as a binder had been reported to have the ability to increasing the calorific value of briquettes [82]. The HV of the briquettes was not significantly different from each other. It can be concluded from these results that the BR of 30% gave the best HV of 10,837 kJ/kg which is better compared to the HV of briquettes produced by Ikelle and Joseph [29] who produced briquettes using different binders.

Fig. 7. Effect of binder ratio on heat value of briquette

Fig. 8 and Table 3 show the variation in VMC of the briquettes with change in BR. The result showed that the VMC of 67.03% for BR of 30% is not significantly different ($P \ge .05$) from the value of 66.41% for BR of 25%. It is also evident from the results that the value of 66.41% for BR of 25% is not significantly different (P \geq .05) from 66.31% for BR of 15%. Furthermore, no significant different (P \ge .05) exists for VMC of 66.31% for 15% and 65.99% 20% BR. Results of the effect of BR performed on the briquettes revealed that the BR of 30% had the highest VMC of 67.03%. This is in line with the findings of Oladeji et al*.* [70]. There was no significant difference ($P \ge 0.05$) in the VMC of the briquettes. The least recorded was 65.99% at BR 15%, yet there was no significant difference (P \geq .05) in

the VMC of the briquettes from 15 - 30% BR. In other words, the VMC do not vary significantly with BR as it is dependent largely on the biomass properties [65].

Fig. 8. Effect of binder ratio on volatile matter of briquette

The results in Table 4 shows the effect of compaction pressure (CP) on the combustion properties of the briquettes, using least significant difference (LSD). Table 4 and Fig. 9 show the variation in AC of the briquettes with change in CP. The results show that the AC of 17.68% for CP of 10 MPa is significantly different $(P \le .05)$ from the value of 14.98% for CP of 5.0 MPa. However, the value of 14.98% for CP of 5.0 MPa is significantly different ($P \le .05$) from value of 13.86% for CP of 7.5 MPa. Also, the value of 13.86% for CP of 7.5 MPa is significantly different (P \leq .05) from 12.87% for 2.5 MPa. From the result, it can be seen that the CP of 10 MPa gave the highest ash content of 17.86% while 2.5 MPa gave the lowest ash content of 12.87%. Fig. 9 confirms that AC generally increases with CP, with a slight decrease between 5 and 7.5 MPa and a sharp increase between 7.5 and 10 MPa. This is consistent with briquettes behavior [40,41].

Fig. 9 and Table 4 show the variation in FC value of the briquettes with change in CP. The results show that the FC of 4.08% for CP of 7.5 MPa is not significantly different ($p \ge 0.05$) from the value of 3.90% for 5.0 MPa. However, the FC of 3.90% for CP of 5.0 MPa is significantly different $(p \le 0.05)$ from the value of 3.20% for 10 MPa. It is also evident from the results that the value of 3.20% for CP of 10 MPa is significantly different $(p \le 0.05)$ from the value of 2.65% for 2.5 MPa. It can be seen from the results that the CP had little effect on the FC since it depends on the biomass used [12,44].

CP (MPa)	AC (%)	FC (%)	HV (kJ/kg)	MC(%)	VMC (%)	
2.5	12.87	2.65	10846	15.49	68.98	
5.0	14.98	3.90	10878	14.41	66.25	
7.5	13.86	4.08	11037	15.17	66.92	
10	17.68	3.20	10251	15.51	63.60	
LSD ($P \le .05$)	0.45	0.19	136.80	0.71	0.90	

Table 4. Effect of Compaction Pressure (CP) on combustion properties of briquettes

Fig. 9. Effect of Compaction Pressure on Combustion Properties of Briquettes

Also, Fig. 9 and Table 4 show the variation in MC value of the briquettes with change in CP. The results show that the MC of 15.51% for CP of 10 MPa is not significantly different ($p \ge 0.05$) from the MC of 15.49% for 2.5 MPa. Again, the value of 15.49% for CP of 2.5 MPa is not significantly different ($p \ge 0.05$) from the value of 15.17% for 7.5 MPa. It is also evident from the results that value of 15.17% for CP of 7.5 MPa is significantly different ($p \le 0.05$) from 14.41% for 5.0 MPa. The percentage MC for the briquettes were within the expected range [49, 50].

Fig. 10 and Table 4 show the variation in HV of the briquettes with change in CP. The results show that the HV of 11,037 kJ/kg for CP of 7.5 MPa is significantly different ($p \le 0.05$) from 10,878 kJ/kg for 5.0 MPa. However, the HV of 10,878 kJ/kg for CP of 5.0 MPa is not significantly different ($p \ge 0.05$) from the value of 10,846 kJ/kg for 2.5 MPa. The result also shows no significant difference ($p \ge 0.05$) between heat value of 10,846 kJ/kg and 10,251 kJ/kg for CP of 2.5 MPa and 10 MPa. From the result, the HV of the briquette increases with increase in CP and decreases as the CP was further increased. The compaction pressure of 7.5 MPa gave the highest HV of the briquettes. This indicates that for a particular PS and BR, the required CP for favorable briquette performance is an optimum value [66, 67].

Fig. 11 and Table 4 show the variation in VMC values of the briquettes with change in CP. The result also showed that the VMC of 68.98% for

CP of 2.5 MPa is significantly different ($p \le 0.05$) from 66.92% for 7.5 MPa. However, the value of 66.92% for CP of 7.5 MPa is not significantly different ($p \ge 0.05$) from 66.25% for 5.0 MPa. It is evident that the value of 66.25% for CP of 5.0 MPa is significantly different ($p \le 0.05$) from 63.60% for CP 10 MPa. The result shows that the VMC decreases with increase in CP which is in agreement with the observations of Waweru and Chirchir [38]. Though VMC primarily depends on the biomass used, it is obvious that the CP required for any given biomass under certain conditions must be optimized to prevent deterioration of VMC which reduces combustion efficiency [62, 65].

Fig. 10. Effect of Compaction on Heat Value of Briquette

Fig. 11. Effect of Compaction Pressure on Volatile Matter of Briquette

Table 5 shows the variation in combustion properties of the briquettes with interaction of PS and BR. The analysis LSD shows that the interaction between PS of 1.18 mm and BR of 30% gave the least AC of 11.08% which is significantly different ($p \le 0.05$) from the AC of

11.46% for interaction between PS 1.70 mm and BR of 30%. Beyond these, the interaction effect between PS and BR had no definite pattern on the AC of the briquettes. However, it was observed that fine particle size 1.18 and 1.70 mm exhibited lower AC compared to 2.36 and 3.35 mm respectively. The highest AC of 18.85% was obtained for interaction between 2.36 mm PS and 20% BR. It can be deduced that the interaction effect between the 1.18 mm PS and 30% BR gave the least low AC which indicates good thermal property of the briquettes [12, 82].

From the results presented in Table 5, no definite pattern was established for the interaction effect of PS and BR on FC of the briquettes using LSD, but it could be seen that interaction of PS 1.18 mm and BR 30% with FC of 5.03% is significantly different ($p \le 0.050$) from the FC of 4.13% for interaction between PS of 1.70 mm and BR of 25% although, higher starch BR was expected to increase the FC of briquettes [18,33]. The interaction effect between PS of 1.18 mm and BR 30% however, gave the best interaction effect on the FC of the briquettes.

The results of interaction effect of PS and BR on HV using least square difference (LSD) are also presented in Table 5. The result shows that the interaction between PS 1.18 mm and BR 30% gave the highest HV of 11,482 kJ/kg, which is significantly different ($p \le 0.05$) from 11,298 kJ/kg for interaction between PS of 1.70 mm and BR of 25%. Though the interaction effect

between PS and BR gave no definite pattern on the HV of the briquettes, it could be generally concluded that 1.18 mm PS with 30% BR had higher HV while the 2.36 mm PS with BR of 20% gave the lower HV of 10,194 kJ/kg. Therefore, briquette produced from PS 1.18 mm blended with BR of 30% can be concluded to be a good candidate for high energy giving fuel.

The results of interaction effect of PS and BR on MC using LSD are also presented in Table 5. The MC ranged between 15.83% and 14.30%. The interaction between PS 1.18 mm and BR of 25% gave the highest MC of 15.83%, which is significantly different ($p \le 0.05$) from the MC of 14.30% for PS 2.36 mm and BR of 15%. It can be concluded from this interaction that the MC of the briquettes are within the acceptable range for good combustion [19,33].

The results of the interaction effect between PS and BR on VMC using LSD are also presented in Table 5. The results gave no definite pattern in the interaction effect. However, it was shown from the results that the highest VMC of 70.38% was recorded for interaction between PS of 1.70 mm and the highest BR 30% and the lowest of 63.16% was recorded for the interaction between 2.36 mm PS and the highest BR of 30%. High VMC is an indicator of easy ignition of briquettes. Therefore, it can be stated that interaction between PS of 1.70 mm and BR of 30% gave the highest VMC for the briquettes.

PS	BR	AC (%)	FC (%)	HV (kJ/kg)	MC (%)	VMC(%)
1.18	15	13.15	3.81	11012	15.57	67.39
	20	12.96	4.09	11108	15.56	67.39
	25	13.09	4.04	11041	15.83	66.97
	30	11.08	5.03	11482	15.42	67.76
1.70	15	13.17	3.30	10939	15.43	68.10
	20	12.46	3.56	11098	15.40	68.58
	25	11.91	4.13	11298	15.54	68.61
	30	11.46	2.88	11124	15.53	70.38
2.36	15	17.51	2.81	10248	14.81	64.48
	20	18.85	3.11	10194	14.81	63.30
	25	18.26	3.23	10240	15.13	63.41
	30	18.17	3.53	10308	15.08	63.16
3.35	15	16.20	4.13	10818	14.30	65.28
	20	16.23	2.72	10247	14.40	64.69
	25	16.17	2.57	10458	14.64	66.67
	30	16.21	2.40	10434	14.85	66.84
LSD ($P \le 0.05$)		0.91	0.38	273.60	NS	NS

Table 5. Effect of particle size and binder ratio on combustion properties of briquettes

The results in Table 6 show the interaction of PS and CP on combustion properties of briquettes, using least significant difference (LSD). The results of the interaction effect between PS and CP on AC using LSD are presented in Table 6. The results show the least AC of 8.17% for the interaction between fine PS 1.18 mm and CP of 2.5 MPa. The highest AC of 22.75% was obtained for the interaction between coarse PS of 2.36 mm and CP of 5.0 MPa. It can be concluded from these results that interaction between 1.18 mm PS and 2.5 MPa CP gave the least AC which is an indicator of good thermal property of briquette [33]. The AC of the briquettes were significantly different ($p \le 0.05$) from each other.

Further analysis results of interaction effect PS and CP on the FC content using LSD are also presented in Table 6. From the results, no definite pattern was established in the interaction effect on FC content of the briquettes, but it could be seen that the interaction of 1.18 mm PS and 7.5 MPa CP with FC of 4.87% is not significantly different ($p \ge 0.05$) from FC of 4.64% for interaction between coarse PS of 3.35 mm and CP of 5.0 MPa. Subsequently, the interaction effect between PS and CP gave no particular pattern. The least FC content of 1.14% was obtained for the interaction between 3.35 mm PS and the CP of 2.5 MPa. The interaction effect between 1.18 mm PS and CP of 7.5 MPa however, gave the highest interaction effect on the FC of the briquettes produced in this study. As earlier mentioned, FC depends on the biomass employed but a combination fine PS and moderate CP enhances the FC thereby improving briquette efficiency [20,21].

The results of the analysis of interaction effect of PS and CP on the HV of the briquettes using LSD are also presented in Table 6. The result shows that the interaction between 1.18 mm PS 2.5 MPa CP gave the highest HV (11,738 kJ/kg), which is significantly different ($p \leq 0.05$) from the value of 11,453 kJ/kg for interaction between 1.70 mm PS and 5.0 MPa CP. Thereafter, the interaction effect between PS and CP gave no definite pattern on high HV of the briquettes but it could be generally concluded that the fine PS with less CP have higher HV and coarse PS with higher CP gave lower heat value [22,33]. The least heat value of 9,596 kJ/kg was obtained from the interaction between the coarse PS of 3.35 mm and CP of 2.5 MPa.

The results of the analysis of the interaction effect between PS and CP on MC LSD are also presented in Table 6. The results show that the interaction between 3.35 mm PS and CP of 5.0 MPa gave the least MC of 13.48%, which is significantly different ($p \le 0.05$) from the moisture content of 15.88% for the interaction between 1.70 mm PS and CP of 7.5 MPa. The interaction effect between PS and CP on the MC of the briquettes exhibited no definite pattern. However, it was observed that the coarse PS of 3.35 mm gave lowest MC compared to the fine and medium PS of 1.18 and 1.70 mm respectively. It can be concluded that the MC was in the satisfactory range [51,83].

Further analysis of the results of the interaction effect between PS and CP on the VMC of the briquettes using LSD are also presented in Table 6. The results show that the highest VMC of 73.13% was recorded for the interaction between 1.70 mm PS and 2.5 MPa CP. The lowest VMC of 59.95% was recorded for the interaction between PS of 1.18mm and the CP 10 MPa. High VMC in briquettes is an indicator of easy ignition. Therefore, the study revealed that interaction between 1.70 mm PS and 2.5 MPa CP gave the highest VMC for the briquettes [15].

Table 7 shows the variation in combustion properties of the briquettes with interaction of BR and CP using LSD. For the interaction effect of BR and CP on the AC, some significant difference at $p \le 0.05$ exist. The interaction with FC is also slightly significantly different. However, the interactions with HV, MC and VMC were all not significant, indicating that there was no definite pattern for the interactions with BR and CP. The interaction effect shows that the lowest AC was produced by 30% BR and 2.5 MPa CP indicating the sample as a good candidate for desirable combustion properties. Conversely, the sample with BR 20% and CP 10 MPa had the highest AC value. For FC, the results indicate that the interaction with 15% BR and 2.5 MPa CP had the least value of 2.43% while the sample with 5.0 MPa CP and the same BR had the highest value. This again shows that the FC is enhanced by using an optimum value of CP [13].

The results of the analysis of the interaction effect of PS, CP and BR on the combustion properties of the briquettes using LSD are presented in Table 8. For the interaction effect of PS, CP and BR on the AC, the interaction of PS

Table 6. Effect of particle size and compaction pressure on combustion properties of briquettes

Table 7. Effect of Binder Ratio and Compaction Pressure on Combustion Properties of Briquettes

of 1.70 mm, 10 MPa CP and 15% BR gave the least AC value of 5.31%, which is significantly different ($p \le 0.05$) from the value of 6.47% for interaction between 1.18 mm PS, CP of 10 MPa and 15% BR. The interaction effect between PS, BR and CP gave no definite pattern on the AC value of the briquettes but it could be generally concluded that fine PS with less binder had the least AC value and the coarse PS with more starch binder gave the higher AC value [33]. The highest AC value of 23.13% was obtained for the interaction between coarse PS of 2.36 mm, BR

20% and CP of 2.5 MPa. The AC value of the briquettes were significantly different from each other. Lower AC is an indication of good quality briquette. Higher AC in a fuel usually leads to higher dusty emissions, air pollution and affects the combustion volume and efficiency [33]. Overall, only about 18.75% of the samples had $AC \ge 20\%$, indicating that a good proportion had acceptably low values of AC [22].

The results of the analysis of the interaction effect of PS, CP and BR on the combustion properties of the briquettes using LSD are presented in Table 8. For the interaction effect of PS, CP and BR on the AC, the interaction of PS of 1.70 mm, 10 MPa CP and 15% BR gave the least AC value of 5.31%, which is significantly different ($p \le 0.05$) from the value of 6.47% for interaction between 1.18 mm PS, CP of 10 MPa and 15% BR. The interaction effect between PS, BR and CP gave no definite pattern on the AC value of the briquettes but it could be generally concluded that fine PS with less binder had the least AC value and the coarse PS with more starch binder gave the higher AC value [33]. The highest AC value of 23.13% was obtained for the interaction between coarse PS of 2.36 mm, BR 20% and CP of 2.5 MPa. The AC value of the briquettes were significantly different from each other. Lower AC is an indication of good quality briquette. Higher AC in a fuel usually leads to higher dusty emissions, air pollution and affects the combustion volume and efficiency [33]. Overall, only about 18.75% of the samples had $AC \ge 20\%$, indicating that a good proportion had acceptably low values of AC [22].

The results in Table 8 shows the interaction effect between PS, BR and CP on the FC of the briquettes. From the results, no definite pattern was established in the interaction effect on FC content of the briquettes, but it could be seen that the interaction of coarse PS of 3.35 mm, BR 20% and CP of 2.5 MPa with FC of 10.19% is significantly different ($p \le 0.05$) from that of 5.31% for interaction between fine PS of 1.70 mm, BR 15% and CP of 10 MPa and subsequent interactions. Although, higher BR was expected to increase the FC content (solid fuel) of briquettes [33]. The low FC content tends to prolong cooking time by its low heat release [81] However, the FC as reported in this study can be compared with the FC of 5.75 to 8.28% obtained by Adetogun et al. [82]. The sample with PS of 3.35 mm, BR of 20% and CP of 25 MPa gave the best performance in terms of FC. Only about 32.81% of the samples had FC \geq 4% which suggests that the biomass may likely require blending with another material to improve the FC content [71].

The results in Table 8 shows the interaction effect between PS, BR and CP on the HV of the briquettes. The interaction between the PS of 3.35 mm, BR of 20% and CP of 2.5 MPa gave the highest HV with value 12,758 kJ/kg, which is significantly different ($p < 0.05$) from the value of 12,006 kJ/kg for the interaction between PS of

1.18 mm, BR of 15% and CP 10 MPa. The interaction effect between PS, BR and CP gave no definite pattern on HV of the briquettes but it could be generally concluded that coarse PS with less CP have higher HV [83]. It can be seen from the Table that PS of 2.36 mm, BR 20% and CP 2.5 MPa gave the least HV of 9,453 kJ/kg. Samples G1-4 and P1-4 all had $HV < 10,000$ kJ/kg corresponding to relatively higher percentage AC (\approx 20% or more). However, about 79.69% of the samples had $HV > 10,000$ kJ/kg, with samples H1 and A4 having HV > 12,000 kJ/kg. This indicates good potentials for the briquettes for heating applications [10].

Further analysis of the interaction effect between PS, BR and CP on the MC of the briquettes using LSD are also presented in Table 8. The results shows that the lowest MC of 12.86% for PS 3.35 mm, BR 20% and CP 2.5 MPa was increased insignificantly to 16.65% for PS 1.70 mm, BR 15% and CP 7.5 MPa. On the whole, the MC values fall within the acceptable range with only about 10.94% having MC \geq 16%. This is an indication of good quality briquettes [58].

The results for the effect of PS, BR and CP on VMC of the briquettes are also presented in Table 8. The results gave no definite pattern in the interaction effect. However, it was revealed from the results that the highest VMC of 76.38% was recorded for the interaction between fine PS of 1.70 mm, BR of 15% and CP of 10 MPa. The lowest VMC of 59.07% was recorded for the interaction between PS of 2.36 mm, BR of 20% and CP of 10 MPa. This shows that the briquettes were significantly different from each other ($p \le 0.05$). Only 6.25% of the samples had VMC < 60% which further attests to the quality of the briquettes for the desired combustion applications [15].

Table 9 shows the outstanding samples for the ultimate analysis of the briquettes. The results of the ultimate analysis show that sample B4 (PS 1.70 mm, BR 15% and CP 10 MPa) had the highest carbon content (CC) of 48.67% and was significantly higher than the CC of 36.34% for sample G3 (PS 2.36 mm, BR 20% and CP 7.5 MPa). The result of high CC implies that the briquettes will burn efficiently. The CC were all within the acceptable range of 30 to 60% as reported by Chaney [86]. About 67.19% of the samples had $CC > 40\%$, indicating the tendency of a good proportion of the briquettes to burn efficiently [84].

(%) (MPa) $(\%)$ 1.18 2.5 15 15.53 71.21 9.69 3.57 A1 11479.88 8.11 A ₂ 5.0 15.53 72.68 3.68 11729.33 A ₃ 8.42 4.06 7.5 15.70 71.82 11735.95 10 A4 15.75 73.74 6.47 4.04 12005.56 2.5 B ₁ 1.70 15.61 72.03 2.12 10.24 11100.15 B2 5.0 15.25 72.66 9.17 2.92 11465.53 B ₃ 2.89 7.5 16.65 71.46 8.97 11282.43 B4 10 15.70 76.38 5.31 2.61 11894.78 C ₁ 2.36 2.5 15.40 70.83 11.19 2.58 11085.28 C ₂ 3.20 5.0 15.32 65.54 15.94 10536.38 C ₃ 3.20 7.5 15.32 65.50 16.08 10530.62 2.99 C ₄ 10 15.45 64.45 17.11 10307.32 D ₁ 2.5 3.35 15.10 63.08 20.36 1.46 9584.76	PS Sample
D ₂ 5.0 15.11 64.18 19.28 1.04 9598.97	
D ₃ 15.11 64.20 19.20 1.04 7.5 9601.85	
D ₄ 10 15.30 64.22 20.30 1.02 9597.86	
E ₁ 2.5 1.18 20 15.67 68.37 12.80 3.16 10930.16	
E ₂ 5.0 3.37 15.83 67.83 12.97 10924.50	
E ₃ 7.5 15.51 66.54 13.83 4.12 10996.23	
E4 10 4.84 14.32 70.32 10.47 11804.90	
F ₁ 2.5 1.70 14.44 68.66 13.03 3.85 11208.81	
F ₂ 14.60 4.23 5.0 70.10 11.07 11546.63	
F ₃ 7.5 4.86 14.08 70.15 10.91 11770.12	
F4 10 12.31 3.70 14.44 69.55 11285.48	
G ₁ 2.36 2.5 23.13 2.26 14.33 60.26 9453.34	
G ₂ 2.72 5.0 14.34 60.91 22.62 9704.86	
G ₃ 2.72 7.5 14.73 59.93 22.62 9563.74	
3.91 G4 10 14.38 59.07 22.64 9848.45	
2.5 H ₁ 3.35 12.86 64.30 11.85 10.19 12757.61	
H ₂ 5.0 13.06 64.16 12.60 3.08 10296.46	
H ₃ 7.5 69.29 13.88 3.05 13.78 11024.88	
H ₄ 10 14.21 70.56 12.98 2.25 10933.10	
1.18 4.26 \vert 1 2.5 25 15.40 10.26 69.74 11505.09	
12 5.0 15.17 69.20 10.54 5.09 11712.29	
13 4.20 7.5 16.06 69.71 10.04 11480.17	
14 10 67.39 5.93 16.02 10.66 11740.03	
2.5 J ₁ 1.70 15.76 66.21 14.29 3.74 10818.25	
65.21 15.41 3.67 J2 15.71 10650.22 5.0	
J3 16.01 67.96 3.25 7.5 12.78 10902.02	
J4 10 16.05 2.82 69.62 12.51 10993.44	
K ₁ 2.36 2.5 4.20 14.50 61.02 20.24 10228.81	
K ₂ 5.0 4.25 14.53 61.18 20.04 10269.02	
K3 7.5 19.43 15.10 60.98 4.49 10322.62	
K4 10 15.08 61.93 18.29 4.70 10531.51	

Table 8. Interaction effect of particle size, binder ratio and compaction pressure on combustion properties of briquettes

Table 8. Continued

Sample	PS	CP	BR	MC(%)	VMC	AC (%)	FC (%)	HV (kJ/kg)
		(MPa)	(%)		$(\%)$			
M1	1.18	2.5	30	15.69	60.23	19.83	4.25	10132.22
M ₂		5.0		15.73	59.85	20.20	4.22	10067.20
M ₃		7.5		16.04	60.13	20.06	3.77	9953.03
M4		10		15.58	59.58	19.60	5.24	10378.50
N ₁	1.70	2.5		15.91	65.48	15.11	3.50	10630.73
N ₂		5.0		16.05	66.37	14.17	3.41	10727.99
N ₃		7.5		15.44	64.87	14.96	5.52	11236.39
N4		10		15.93	65.98	15.70	2.39	10321.65
O ₁	2.36	2.5		15.01	65.80	16.82	2.18	10223.63
O ₂		5.0		15.04	65.91	16.79	2.26	10266.94
O ₃		7.5		15.37	67.23	14.89	2.51	10542.85
O4		10		15.40	67.21	14.62	2.52	10543.40
P ₁	3.35	2.5		15.11	62.20	20.52	2.58	9842.56
P ₂		5.0		15.20	61.12	20.84	2.60	9693.91
P ₃		7.5		15.33	62.24	19.86	2.57	9844.89
P4		10		15.26	62.77	20.32	1.67	9605.35
LSD				NS	3.61	1.83	0.76	647.30
(PS.05)								

Table 9. Ultimate analysis of the outstanding samples

The percentage hydrogen content (HC) of 6.025% for sample B4 (PS 1.70 mm, BR 15% and CP 10 MPa) was significantly different from 4.522% of HC of sample G3 (PS 2.36 mm, BR 20% and CP 7.5 MPa). The HC decreased from 6.025 to 4.522%. According to Ryemshak and Aliyu [85], low HC results in low quantity of VMC. However, the 6.025% HC of sample B4 compares well with 6.29% recorded by Mohammed et al*.,* 2020 for 20:80% of orange peels and corn cobs, and was within the acceptable range of 5 to 6% as reported by Chaney [86]. About 68.75% of the samples had $HC > 5%$.

The briquette samples produced in this study had low oxygen content (OC) with sample G3 (PS 2.36 mm, BR 20% and CP 7.5 MPa) having - 7.89% as least, while sample A4 (PS 1.18 mm, BR 15% and CP 10 MPa) had 9.28%. The OC increased relatively to 10.39% in sample B4 (PS 1.70 mm, BR 15% and CP 10 MPa). This is similar to 10.64% OC 20:80% (orange peels and corn cobs) reported in the findings of Mohammed et al. [62]. Samples D1-4 (mean $OC = -4.63$), G1-4 (mean $OC = -8.31$), K1-4 (mean $OC = -$ 4.698), M1-4 (mean $OC = -4.163$) and P1-4 (mean $OC = -5.16$) had relatively lower values indicative of limited porosity and hence impaired combustion performance. This can be corrected by optimizing Brand blending with a more porous biomass [11,68].

Percentage of Nitrogen content (NC) of sample G3 (PS 2.36 mm, BR 20% and CP 7.5 MPa) of 0.9014% was the highest and was significantly different from 0.6252% NC for sample A4 (PS 1.18 mm, BR 15% and CP 10 MPa) and 0.5724% for sample B4 (PS 1.70 mm, BR 15% and CP 10 MPa). Nitrogen and sulphur are the elements in materials which causes pollution during combustion. These elements react with the surrounding air to produce the harmful NO_x and SO_x [84]. Chaney [86] reported that percentage NC in biomass briquettes should not exceed 1%. All the samples had $NC < 1.0$ but samples D1-4 (mean NC = 0.8206), G1-4 (mean $NC = 0.8992$, K1-4 (mean $NC = 0.8746$), M1-4 (mean NC = 0.8936) and P1-4 (mean NC = 0.8584) had relatively higher values indicative of poorer samples [3].

The sulphur content (SC) increased in all the briquettes varied from 29.03 to 43.51%. Sample B4 had the lowest SC while sample G3 had the highest value. However, biomass fuels with lower SC are preferred Meng [87]. In terms of SC values therefore, sample B4 had the more preferred value. Only about 31.25% of the briquette samples had SC < 40% indicating the need to blend with other biomass or necessity for the use of other binders to enhance lower values of SC [11]. The samples with the relatively higher HC and lower OC all had SC > 40% indicating the shortcoming earlier mentioned.

The ANOVA for the heat value of raphia palm seed briquettes is presented in Table 10. The Table shows a significant model P-value ≤ 0.0001. This indicates the significance and the adequacy of the model. The adequacy is also tested by comparing the model validation parameters [59]. The high R^2 value obtained shows consistency and that the process parameters explain 95 % of the variance of the heat value. Therefore, the model presented in Table 11 can be used to predict the heat value of the briquette.

The experimental data were analysed to check the correlation between the values of the

experimental and the predicted HV and the result of the plot is shown in Fig. 12. The R^2 value of 0.9542 shows that the data points are distributed reasonably near the straight line. This indicates a good relationship between the actual and the predicted heat or energy value of the briquette. The result suggests also that the selected model is adequate for predicting the heat value of the briquette [59].

The results of optimization of the heat or energy value of the briquettes carried out using Design Expert 8.0.6 in Response Surface Methodology (RSM) is presented in Fig. 13. The design gave rise to 35 runs with particle size, binder ratio and compaction pressure as the independent variables and the heat or energy value of the briquettes as a response. The result referring to the highest desirability of 1.00 (100% probability that the optimisation result is achievable) and the optimum heat value of the briquettes was selected as shown in Fig. 13. The optimization process gave a 12821.7 kJ/kg heat or energy value at optimum 24.12% binder ratio, 1.18% particle size and 3.00 MPa compaction pressure; and this is in line with the work carried out by Chukwuneke et al*.* [59].

Table 10. Model Summary and ANOVA of Raphia palm seed Briquettes

Table 11. Developed heat value model for raphia palm seeds briquettes

Fig. 12. Plot of predicted versus actual heat value of Raphia palm seed briquettes

Fig. 13. Optimization result of heat value of raphia palm seed briquettes

4. CONCLUSION

This study has confirmed that Raphia palm seeds which is available in abundant quantity and disposed indiscriminately can be used for the production of better quality briquettes. This will provide an alternative source of energy, minimizing deforestation rate and help to solved agricultural waste management issues as well as converting waste to wealth. The combustion properties of the briquettes are within the acceptable range with the briquette of particle size 1.70 mm, binder ratio 15% and compaction pressure 10 MPa giving better positive attributes of good quality briquette, because it has higher value of volatile matter of 76.38% and least percentage of ash content of 5.31%. The

developed model equation can be used in predicting the heat or energy value of briquettes produced from Raphia palm seed. The optimization process gave the heat or energy value of the briquettes produced from Raphia palm seeds to be 12821.7 kJ/kg at the optimal condition values (24.12% binder ratio, 1.18% particle size and 3.00 MPa compaction pressure).

The use of briquette should be given wide publicity in Nigeria as alternative methods to combating climate change and promotion of renewable clean energies. The rate of deforestation in Nigeria is alarming. Compacting biomass waste into briquettes to completely replace loose biomass waste, will curtail deforestation, saving the natural wood reserves. Sensitization and educative campaigns need to be created among the populaces.

DISCLAIMER

The products used for this research are commonly and predominantly use products in our area of research and country. There is absolutely no conflict of interest between the authors and producers of the products because we do not intend to use these products as an avenue for any litigation but for the advancement of knowledge. Also, the research was not funded by the producing company rather it was funded by personal efforts of the authors.

ACKNOWLEDGEMENTS

The authors acknowledge the entire staff of the Metallurgy and Materials laboratory at Joseph Sarwuan Tarka University, Makurdi for assistance with sample preparations, tests and measurements. This research was sponsored by the authors and has provided the opportunity for a Master's student to prepare for his external examination.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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> *Peer-review history: The peer review history for this paper can be accessed here: https://www.sdiarticle5.com/review-history/87375*