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Original Research Article

Bio-Briquette Production from Hydrothermal Liquefaction Residue of Locust Bean Pulp Using Cassava Starch Binder

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ABSTRACT

The growing demand for sustainable energy and effective waste management has intensified research into biomass valorization. This study explores the production of bio-briquettes from hydrochar obtained via hydrothermal liquefaction (HTL) of locust bean (Parkia biglobosa) pulp, using cassava starch as a biodegradable binder. Process optimization was conducted with the Taguchi method, considering binder concentration (10–30 %), particle size (300–600 µm), and compaction pressure (5–15 MPa), with moisture content as the primary response. Analysis of variance (ANOVA) identified binder concentration as the most significant factor (contribution ratio 91.64 %), followed by compaction pressure (6.11%) and particle size (1.74 %). Optimal conditions were 30 % binder, 300 µm particle size, and 15 MPa pressure, yielding briquettes with improved moisture content (1.09%), density (0.52 g/cm³), compressive strength (1.82 kN), calorific value (18.20 MJ/kg), and reduced ash content (8.71 %). Compared to control samples, the optimized briquettes exhibited higher ignition potential, durability, and combustion efficiency, meeting the requirements for household and small-scale industrial use. The findings demonstrate the potential of converting HTL residues of locust bean pulp into high-quality bio-briquettes, contributing to renewable energy generation, waste valorization, and sustainable rural energy solutions.

KEYWORDS

Biomass, Hydrochar, Locust bean pulp, Bio-briquette, Cassava starch binder, Hydrothermal liquefaction, Renewable energy, Waste-to-energy.

INTRODUCTION

The increasing global energy demand, coupled with pressing environmental concerns and the depletion of fossil fuel reserves, has intensified the search for renewable and sustainable energy alternatives. Among these, biomass has gained significant attention due to its wide availability, carbon neutrality, and potential to reduce greenhouse gas emissions. The valorization of agricultural and food-processing residues into bioenergy not only addresses energy challenges but also contributes to waste management and environmental sustainability [1–3].

Hydrothermal liquefaction (HTL) is an emerging thermochemical conversion technology that processes wet biomass under subcritical or supercritical water conditions (typically 250–

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374 °C and 4–22 MPa), producing a bio-crude oil phase alongside aqueous, gaseous, and solid residues. The solid residue—commonly termed hydrochar or biochar—is often rich in fixed carbon, aromatic structures, and oxygen-containing functional groups, making it a promising precursor for solid biofuel production [4–6]. Despite its potential, hydrochar is frequently underutilized or discarded as waste.

Locust bean (Parkia biglobosa) pulp is an abundant agro-waste byproduct in sub-Saharan Africa, particularly in Nigeria, where it is traditionally processed for culinary and medicinal uses. Post-HTL processing of this biomass generates a solid residue that retains significant calorific value and physicochemical properties suitable for briquetting. Harnessing this underexploited biomass for fuel applications aligns with the global push toward circular bioeconomy and decentralized energy systems.

Briquetting is a densification process whereby fine or powdered biomass is compressed into uniform, compacted blocks—bio-briquettes—which offer improved energy density, better handling, and cleaner combustion compared to raw biomass. The briquetting process often necessitates the inclusion of a binder to ensure cohesion, structural integrity, and durability of the briquettes during storage, transportation, and combustion [4,7,8].

Cassava starch, derived from cassava (Manihot esculenta) roots, is an effective, biodegradable, and widely available binder in many tropical regions. Upon gelatinization, cassava starch forms a viscous adhesive matrix that enhances particle binding and mechanical strength. Its non-toxic nature and clean combustion characteristics make it particularly attractive for rural biomass energy systems [9–11].

The present study explores the utilization of hydrochar obtained from the HTL of locust bean pulp as a feedstock for bio-briquette production, with cassava starch employed as a natural binder. The study aims to assess the mechanical and combustion characteristics of the resulting briquettes and evaluate their suitability as a renewable solid fuel.

While extensive research has focused on biomass briquetting from residues such as rice husk, maize cobs, sugarcane bagasse, and groundnut shells [12–15], limited studies have investigated the conversion of HTL-derived residues—particularly from locust bean pulp—into bio-briquettes. Moreover, few studies have systematically evaluated the role of cassava starch as a binder in such systems, despite its relevance in local contexts.

This work therefore seeks to address these key research gaps by:

- Investigating the physical, mechanical, and combustion properties of bio-briquettes produced from HTL hydrochar of locust bean pulp;
- Evaluating the effect of cassava starch binder concentration on briquette performance;
- Employing Taguchi experimental design methodology to optimize briquetting parameters for enhanced fuel quality.

The Taguchi design, a robust statistical approach based on orthogonal arrays, enables efficient evaluation of multiple process variables and their interactions with a minimal number of experiments. This methodology improves process robustness and identifies optimal settings for desirable output characteristics, particularly in material and energy systems.

Ultimately, this research contributes to sustainable waste-to-energy strategies, promotes local resource utilization, and offers viable alternatives to traditional fuels in rural and peri-urban communities.

Beyond its environmental benefits, the valorization of locust bean pulp hydrochar into bio-briquettes offers significant economic potential. It presents a low-cost energy alternative for rural and peri-urban households that rely heavily on firewood and charcoal, which are increasingly expensive and unsustainable. Additionally, the use of locally sourced cassava starch as a binder supports agro-based economies and enhances the viability of small-scale briquette enterprises. By transforming agro-waste into marketable energy products, this approach creates income-generating opportunities, reduces energy poverty, and contributes to rural industrialization and circular bioeconomy development in sub-Saharan Africa.

MATERIALS AND METHODS

Material

The solid residue used in this study was obtained from the hydrothermal liquefaction (HTL) of locust bean pulp. The HTL process was carried out at a temperature range of 250–300 °C for 30–60 min under autogenous pressure. After the reaction, the solid residue was separated by filtration, washed with distilled water, and oven-dried at 105 °C to constant weight. The binder from food-grade cassava starch was procured locally. It served as the natural binder for briquette formation due to its good adhesive properties and environmental friendliness. Clean distilled water was used in preparing the cassava starch solution and to aid mixing.

Equipment

- Grinding machine
- Briquetting mold and manual or hydraulic press
- Oven (for drying)
- Weighing balance
- Calorimeter (for heating value test)
- Moisture analyzer or oven for moisture content test
- Sieve shaker (to ensure uniform particle size)

Preparation of raw material

The hydrochar was oven-dried at 105 °C for 24 h to remove residual moisture. It was then ground and sieved to obtain a uniform particle size between 1–2 mm, which enhances compaction and briquette quality. Cassava starch was mixed with hot water in a 10 % weight/volume ratio (10 g starch in 100 mL water) and heated while stirring to form a viscous gel-like paste. This binder was allowed to cool before use.

Briquette Formulation and Production

The dried and sieved hydrochar was mixed with cassava starch binder in varying proportions (90:10, 85:15, 80:20 hydrochar to binder by weight) to evaluate the impact of binder concentration on briquette quality. The mixture was placed in a cylinder briquette mold and compacted with a hydraulic press to form uniform briquettes. Pressure was applied for 2–5 min to ensure structural integrity. The compacted briquettes were air-dried for 48 h and then oven-dried at 105 °C until a moisture content below 10 % was achieved. Proper drying is critical to reduce smoke and improve combustion.

Response analysis

The response for the experimental design, which was the moisture content, was determined for all the briquette samples produced. The moisture content of the samples was determined according to ASTM E872-82 as described by Lee et al. [1]. A known weight (Wi) of the samples was placed inside an oven with a dish of known weight, and allowed to dry inside the oven at a temperature of 105 °C for 5 h until the weight difference became stable. The weight of the dry sample (Wd) was recorded and applied in Eq. (1) to obtain the percentage moisture content (%MC).

$$\%MC = \frac{Wi-Wd}{Wi}\ x\ 100 \tag{1}$$

Characterization of Briquettes

The physical and combustion properties of the Briquette will be analyzed and compared the existing properties gotten from previous research which will serve as control. Physical Properties are described first.

Density. The density of each of the produced briquettes was determined after sun drying of the briquettes, as a ratio of measured mass to the volume of the briquettes. The mass of the produced briquettes (mb) was determined using a digital weighing balance, while the dimensions, which include the diameters (db), and height (hb) of the briquettes, were measured using a vernier calliper. The measured diameters and height were subsequently used to calculate the volumes (Vb) of the briquettes using **Eq.** (2), while the corresponding densities (ρb) were calculated using **Eq.** (3) **[7]**.

$$Vb = \pi \frac{db}{4} hb \tag{2}$$

$$Pb = \frac{mb}{Vb} \tag{3}$$

Shatter index. The shattering index of the produced briquettes was measured according to ASTM D440- 86 (2002) method of drop shatter, developed for coal **[8]**. The test was conducted after sun drying of the briquette samples. The weight of each of the samples was measured and placed in a polythene bag. The bag was dropped from a height of 2 m onto a concrete floor four times. After the dropping, the briquettes and their fractions were placed on top of a 35 cm square mesh screen and sieved. The mass of the remaining briquettes was measured, and the shatter index, expressed as the ratio of the weight of material retained on the screen (mbs) to the weight of the briquettes before the dropping (mb), was determined using **Eq.** (4) **[8]**.

$$\text{Shatter index} = \frac{Mbs}{Mb} \times 100 \tag{4}$$

Compressive strength. The axial compressive strength (N/mm^2) of the briquette was measured using a Universal Testing Machine with a digital control and display unit for test control and result display. The dimension of the sample was first measured and input into the system, and the briquette was placed directly under the plunger to be pressed. The machine applied load to the briquette until failure occurred in the briquette. The applied force was recorded and displayed until the maximum force that corresponds to the failure is recorded. The compressive strength is then calculated using **Eq.** (5) **[8]**.

$$\text{Compressive strength} = \frac{maximum\ force\ applied}{surface\ area\ of\ the\ briquette} \tag{5}$$

Durability index. Durability index of the produced briquette is its ability to resist water absorption. This was determined by immersing a measured sample of the briquette into 150 mL of water at room temperature for 30 s. The weight of the briquette before and after soaking was determined, and the briquette's hydrophobicity expressed in percent was calculated using **Eq.** (6) **[7]**.

$$\text{Durability index} = 100 - \left(\frac{Mw - Mb}{Mb} \times 100\right) \tag{6}$$

The combustion properties are described next.

Calorific value. The calorific value of the sample was determined using the bomb calorimeter according to ASTM D240 **[20]**. The mass of water in the calorimeter (W), the mass of water equivalent of calorimeter and stirrer (w), and the initial and final temperature readings of the calorimeter (T 1 and T 2), respectively, as well as the mass of the loaded sample (m_s) were recorded and applied in **Eq.** (7) to determine the calorific value.

$$\text{Calorific Value} = \frac{(W-w)(T1-T2)}{MS} \tag{7}$$

Volatile matter. The volatile matter of the sample was determined using ASTM E 872 as described by Basu [25]. The residual dry sample from moisture content determination was placed in a crucible with cover to avoid the interaction of the sample with air. The sample in the crucible was heated at 900 °C in a furnace for 7 min to drive off the volatiles. The weights of the samples before and after heating (Wd and Wh, respectively) were measured and applied in Eq. (8) to obtain the percentage volatile matter (%VM).

$$\%VM = \frac{Wd-Wh}{Wd} \tag{8}$$

Fixed carbon content. The fixed carbon according was obtained by subtracting the sum of percentage content of moisture, ash, and volatile matter from 100% of the whole sample as expressed in Eq. (9) [26].

$$\%FC=100\% - (\%AC - \%VM - \%MC) \tag{9}$$

Ash content. The ash content was determined according to ASTM E1755- II, as described by Lee et al. [25]. A known weight of the dry sample (Wd) was put inside a crucible and placed inside the furnace, and operated at the temperature of 550 °C for about 8 h until the sample completely turned into ash. The crucible was transferred to a desiccator to cool and weighed to record the weight of the ash (Wa). The obtained data were applied in Eq. (10) to determine the percentage ash content (%AC).

$$\%AC = \frac{Wa}{Wd} x\ 100 \tag{10}$$

RESULTS AND DISCUSSION

Optimization Results

Table 1 presents the experimental design factors with their low and high values for the produced Briquette. The optimum conditions from the optimization are: 8 MPa pressing pressure, particle size 300 µm, and 20 % binding dosage— as seen in Figures 1 and 2 (S/N ratio plot and mean effect plot).

Table 1. Experimental design factors with their high and low values for briquette

Factor	Name	Unit	Low	High
A	Pressing pressure	bar	4	16
B	Particle size	µm	300	600
C	Binding dosage	%	20	50

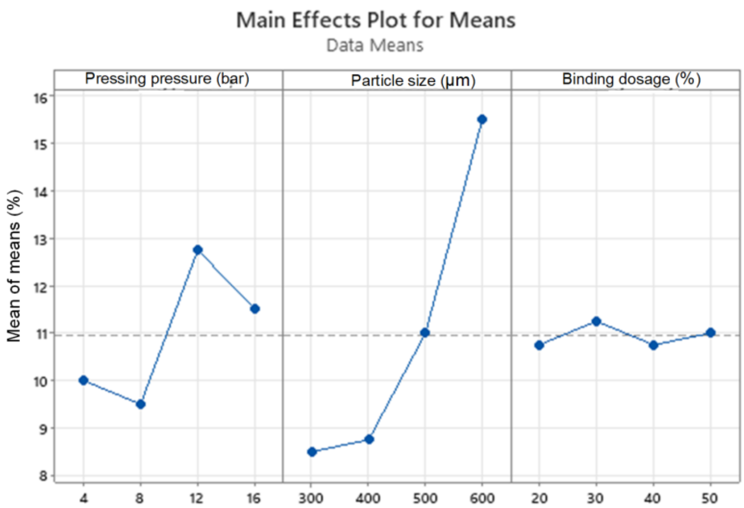

Figure 1. Mean effect plot for mean of moisture content

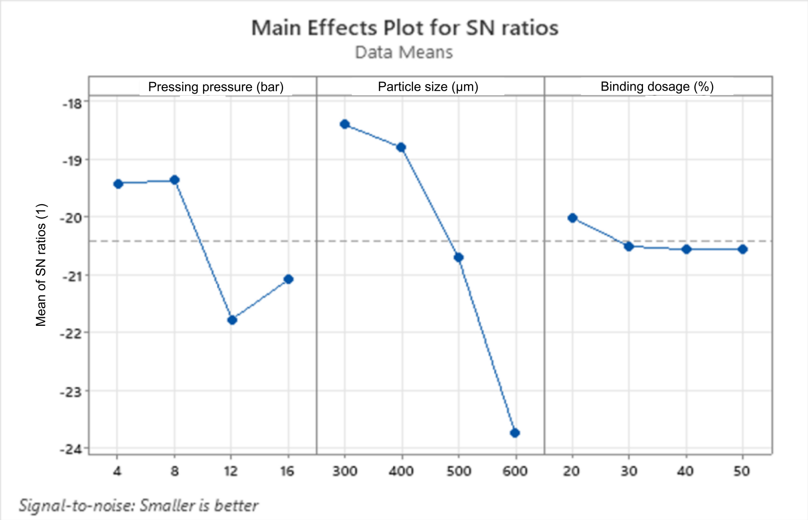

Figure 2. Mean effect plot for signal to noise ratio of moisture content

Analysis of Variance (ANOVA)

Analysis of Variance (ANOVA) is conducted to examine the factors that significantly affect the responses (Moisture content, Table 2). The percentage contribution P reports the significance level. The Fisher test (F-Test) is used to determine statistically the parameters that have a significant effect on the quality characteristics [29,30]. The lower the percentage value (P value), the more significant is the factor. Table 3 shows the ANOVA results for moisture content response. The most significant factor for the Briquette produced is the particle size, as depicted in **Tables 4** and **5**. The result obtained from the ANOVA is in agreement with the analysis of the signal-to-noise ratios obtained in **Tables 4** and **5**.

Table 2. Result of the sixteen runs with their signal to noise ratio and mean value

Pressing Pressure (bar)	Particle size (µm)	Binding dosage (%)	Moisture content (%)	SNRA (1)	MEAN (%)
4	300	20	6	-15.5630	6
4	400	30	8	-18.0618	8
4	500	40	10	-20.0000	10
4	600	50	16	-24.0824	16
8	300	30	8	-18.0618	8
8	400	20	8	-18.0618	8
8	500	50	9	-19.0849	9
8	600	40	13	-22.2789	13
12	300	40	10	-20.0000	10
12	400	50	9	-19.0849	9
12	500	20	14	-22.9226	14
12	600	30	18	-25.1055	18
16	300	50	10	-20.0000	10
16	400	40	10	-20.0000	10
16	500	30	11	-20.8279	11
16	600	20	15	-23.5218	15

Table 3. Analysis of Variance for moisture content response

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Pressing pressure	3	26.187	8.7292	3.77	0.078
Particle size	3	126.188	42.0625	18.19	0.002
Binding dosage	3	0.688	0.2292	0.10	0.958
Error	6	13.875	2.3125		
Total	15	166.938			

Table 4. Response Table for Signal to Noise Ratios, SNRA (1)

	Pressing Pressure	Particle size	Binding dosage
1	-19.43	-18.41	-20.02
2	-19.37	-18.80	-20.51
3	-21.78	-20.71	-20.57
4	-21.09	-23.75	-20.56
Delta	2.41	5.34	0.55
Rank	2	1	3

Table 5. Response Table for Means

Level	Pressing Pressure (bar)	Particle size (μm)	Binding dosage (%)
1	10.000	8.500	10.750
2	9.500	8.750	11.250
3	12.750	11.000	10.750
4	11.500	15.500	11.000
Delta	3.250	7.000	0.500
Rank	2	1	3

Experimental results

The produced briquette samples are shown in Figure 3, while the result of the experimental design.

Figure 3. Picture of the produced briquettes samples

The obtained response from the produced briquettes are shown in Table 2. The experimental result, as shown in Table 2, shows that the briquette with the lowest moisture content of 6 % was produced at the parameter combination of 300 mm particle size, 4 MPa pressing pressure and 20 % binder dosage, while the highest moisture content is 18 % and was obtained from the production parameter of 600 mm particle size, 12 MPa pressing pressure and 30 % binder dosage. The optimum design parameters were obtained as 12 MPa pressing pressure and 30 % binder dosage and were now used to produce the briquette that was characterized.

Characterization report

Density. The result of the density of the briquette from locust bean using the optimum design setting is shown in Table 6 and illustrated in Figure 4. The result demonstrates that the density of the briquette is 950 kg/m^3 as against that of the control which is 900 kg/m^3. This goes to show that the briquette produced with locust bean is an improvement because briquette is best at a higher density.

Table 6. Result of the density of the produced briquette and the control

Sample	Density (kg/m^3)
Produced briquette	950
Control	900

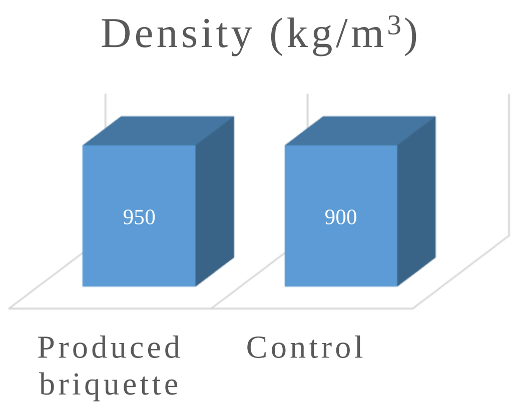

Figure 4. Density plot of the produced briquettes and the control

Shatter index. The result of the shatter index of the briquette from locust bean using the optimum design setting is shown in Table 7 and illustrated in Figure 5. The result showed that the density of the briquette is 95 % as against that of the control which is 90 %. This indicates that the briquette produced with locust bean is an improvement because briquette is best at a higher shatter index.

Table 7. Result of the Shatter index of the produced briquette and the control

Sample	Shatter index (%)
Produced briquette	95
Control	90

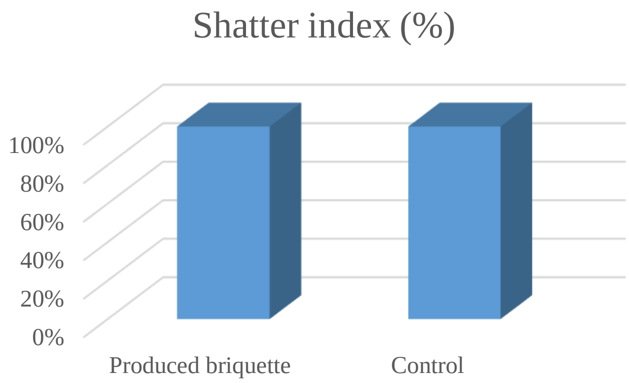

Figure 5. Shatter index plot of the produced briquettes and the control

Compressive strength. The result of the compressive strength of the briquette from locust bean using the optimum design setting is shown in Table 8 and illustrated in Figure 6. The result showed that the density of the briquette is 1.75 N/mm^2 as against that of the control which is 1.70 N/mm^2 this demonstrates that the briquette produced with locust bean is an improvement because briquette is best at a higher compressive strength.

Table 8. Result of the Compressive strength of the produced briquette and the control

Sample	**Compressive strength (N/mm^2)**
Produced briquette	1.75
Control	1.70

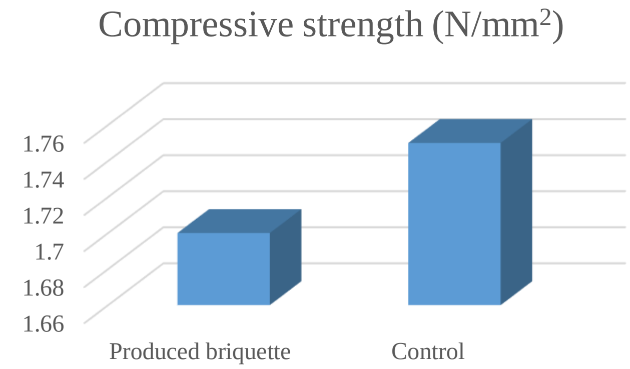

Figure 6. Comparative strength of the produced briquettes and the control

Durability index. The result of the durably index of the briquette from locust bean using the optimum design setting is shown in Table 9 and illustrated in Figure 7. The result showed that the density of the briquette is 85 % as against that of the control which is 80 %. This indicates that the briquette produced with locust bean is an improvement because briquette is best at a higher durability index.

Table 9. Result of the Durability index of the produced briquette and the control

Sample	**Durability index (%)**
Produced briquette	85
Control	80

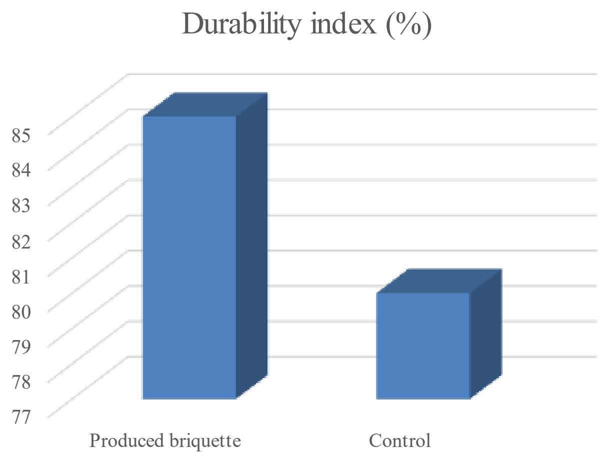

Figure 7. Durability index plot of the produced briquettes and the control

Calorific value. The result of the calorific value of the briquette from locust bean using the optimum design setting is shown in Table 10 and illustrated in Figure 8. The result showed that the density of the briquette is 17 MJ/kg as against that of the control which is 16 MJ/kg. This demonstrates that the briquette produced with locust bean is an improvement because briquette is best at a higher calorific value.

Table 10. Result of the Calorific value of the produced briquette and the control

Sample	Calorific value (MJ/Kg/)
Produced briquette	17
Control	16

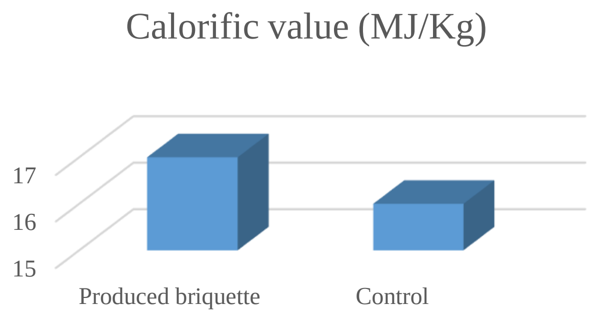

Figure 8. Calorific value plot of the produced briquettes and the control

Volatile matter. The result of the volatile matter of the briquette from locust bean using the optimum design setting is shown in Table 11 and illustrated in Figure 9. The result indicates that the density of the briquette is 75 % as against that of the control which is 70 %. This goes to show that the briquette produced with locust bean is an improvement because briquette is best at a higher volatile matter.

Table 11. Result of the Volatile matter of the produced briquette and the control

Sample	Volatile matter (%)
Produced briquette	75
Control	70

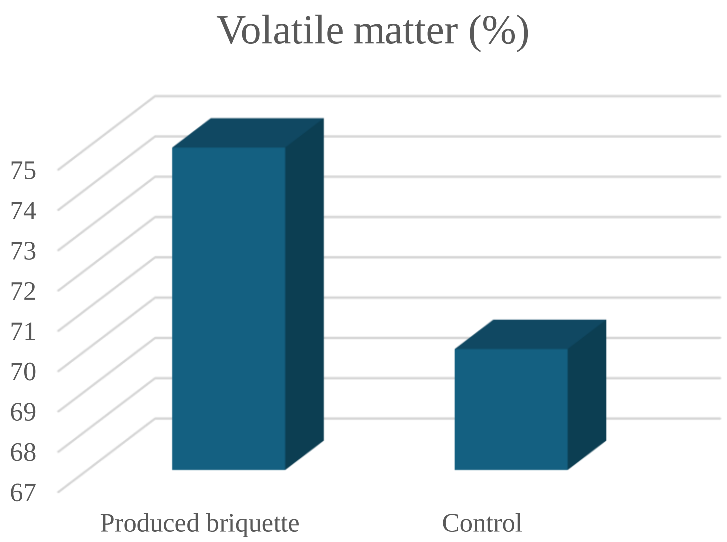

Figure 9. Volatile matter plot of the produced briquettes and the control

Fixed carbon content. The result of the fixed carbon content of the briquette from locust bean using the optimum design setting is shown in Table 12 and illustrated in Figure 10. The result indicates that the density of the briquette is 15 % as against that of the control which is 20 %. This goes to show that the briquette produced with locust bean is an improvement because briquette is best at a higher fixed carbon content.

Table 12. Result of the fixed carbon content of the produced briquette and the control

Sample	Fixed carbon content (%)
Produced briquette	15
Control	20

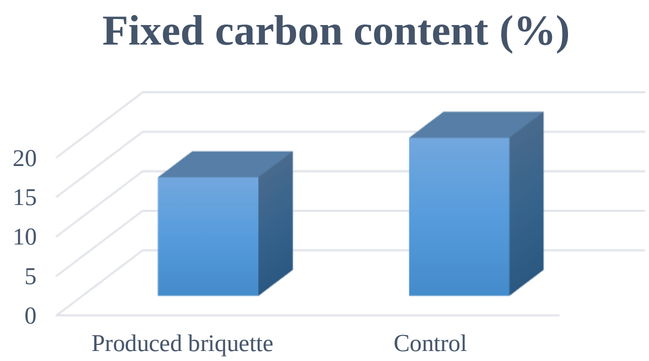

Figure 10. Fixed carbon content plot of the produced briquettes and the control

Ash content. The result of the ash content of the briquette from locust bean using the optimum design setting is shown in Table 13 and illustrated in Figure 11. The result indicates that the density of the briquette is 8 % as against that of the control which is 5 %. This goes to show that the briquette produced with locust bean is almost the same with that of the control and very close to the standard ash content of briquette which is 5 %.

Table 13. Result of the Ash content of the produced briquette and the control

Sample	Ash content (%)
Produced briquette	8
Control	5

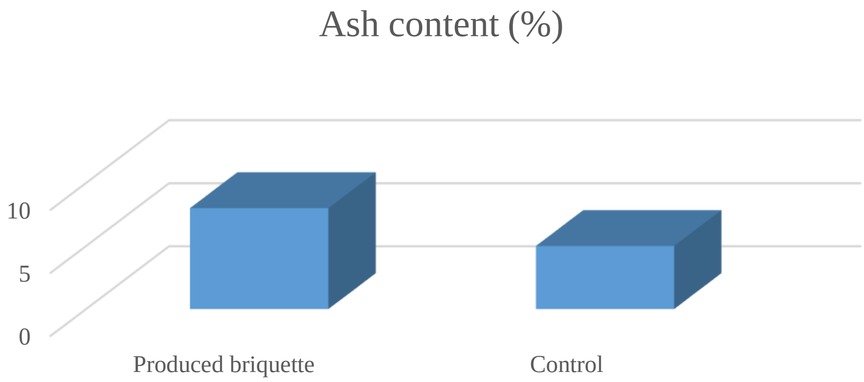

Figure 11. Ash content plot of the produced briquettes and the control

CONCLUSION

This study demonstrated the effective utilization of hydrothermal liquefaction residue from locust bean pulp for the production of bio-briquettes using cassava starch as a binder. Taguchi optimization revealed that binder concentration had the greatest influence on briquette quality, with 30 % starch, 300 µm particle size, and 15 MPa pressure producing briquettes of low moisture content, high density, good mechanical strength, and desirable combustion properties. Compared with control samples, the optimized briquettes exhibited superior calorific value, reduced ash content, and enhanced ignition and durability, confirming their suitability for domestic and small-scale industrial energy applications. The findings highlight the potential of transforming agro-residues into renewable solid fuel, promoting waste valorization, and supporting sustainable rural energy supply. Future studies should explore scale-up production, long-term storage stability, and the integration of alternative low-cost binders to broaden practical application.

Data availability statement

Data supporting the findings of this study are available from the corresponding author upon reasonable request.

Credit authorship contribution statement

Chike Martins Atah: Writing - original draft, Validation, Resources, Methodology, Investigation, Formal analysis, Conceptualization. Writing - review & editing.

Declaration of competing interest

The author declares no competing financial or non-financial interests.

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