

Valorization of *Pangium edule* shell waste into high-performance charcoal briquettes using cassava waste starch and pine resin as renewable bio-based binders

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ABSTRACT

The valorization of underutilized forestry residues into renewable solid biofuels offers an opportunity to reduce waste while supporting low-carbon energy transitions. This study investigated *Pangium edule* shell waste as a feedstock for charcoal briquette production using cassava waste starch and pine resin as a dual renewable binder system. A completely randomized factorial design was employed to examine the effects of cassava waste starch (8, 12, and 16 wt%) and pine resin (0 and 5 wt%) on proximate composition, physical properties, ignition behavior, and higher heating value (HHV). Moisture, volatile matter, ash, and fixed carbon contents ranged from 3.45–7.15%, 20.67–29.15%, 2.01–2.71%, and 61.00–73.86%, respectively, while density, compressive strength, ignition time, and HHV ranged from 0.60–0.66 g cm⁻³, 3.45–7.02 kg cm⁻², 41–128 s, and 6.620–7.080 kcal kg⁻¹, respectively. Increasing starch concentration significantly increased moisture, volatile matter, density, and compressive strength, but reduced fixed carbon. Pine resin markedly shortened ignition time, while its effect on ash content was comparatively limited. Among the tested formulations, 12 wt% cassava waste starch + 5 wt% pine resin (A2B2) provided the most favorable performance profile, with the highest density (0.66 g cm⁻³), compressive strength (7.02 kg cm⁻²), and HHV (7.080 kcal kg⁻¹), together with the shortest ignition time (41 s), while maintaining 5.35% moisture, 2.18% ash, and 72.97% fixed carbon. Although volatile matter exceeded the corresponding SNI 01-6235-2000 limit, all formulations complied with the specified moisture and ash limits. The findings demonstrate that integrating *P. edule* shell waste with cassava waste starch and pine resin can produce a structurally robust and energy-rich solid biofuel. The study provides experimental evidence for a dual-binder strategy that balances mechanical integrity, ignition performance, and energy content, while highlighting the potential of underutilized forestry and agro-industrial residues for circular bioenergy applications.

Keywords: biomass waste valorization, bio-based binders, charcoal briquettes, *Pangium edule* shell, pine resin, renewable solid biofuel.

INTRODUCTION

The increasing global demand for energy and the urgent need to mitigate climate change have accelerated the transition from fossil-based energy systems toward renewable and sustainable alternatives. Although fossil fuels remain the dominant source of global energy, their extensive utilization contributes substantially to greenhouse gas

emissions and environmental degradation. Consequently, lignocellulosic biomass has emerged as an attractive renewable energy resource because of its widespread availability, carbon-neutral characteristics, and ability to simultaneously enhance energy security and reduce biomass waste. Recent assessments have further emphasized that sustainable biomass utilization plays an increasingly important role in supporting low-carbon

energy transitions and reducing dependence on fossil fuels, particularly in developing countries where locally available biomass resources represent an important component of future renewable energy systems (Alsufyani, 2025; Abah et al., 2025). Among the available biomass conversion technologies, charcoal briquetting has gained considerable attention because densification improves bulk density, storage stability, transportation efficiency, and combustion performance, thereby increasing the suitability of biomass as a renewable solid fuel (Aransiola et al., 2019; Tumuluru et al., 2011).

The utilization of biomass has evolved beyond conventional waste disposal toward biomass valorization within the framework of the circular bioeconomy. Rather than being discarded through open burning or landfilling, forestry residues can be transformed into renewable energy carriers and other value-added products, extending biomass life cycles while minimizing environmental impacts. Such integrated utilization contributes to resource efficiency and supports sustainable production systems by replacing fossil-derived materials with renewable biomass resources (Lewandowski, 2015). In addition to improving resource efficiency, biomass valorization contributes to environmental sustainability by reducing waste generation and greenhouse gas emissions while supporting circular bioeconomy strategies based on renewable biological resources (Abah et al., 2025).

Indonesia possesses abundant lignocellulosic biomass generated from forestry and agroforestry systems. However, many indigenous forest residues remain underutilized despite their considerable bioenergy potential. One such resource is the shell of *Pangium edule* Reinw., a tropical tree species widely distributed throughout Southeast Asia. During the processing of *P. edule* seeds for traditional food products, substantial quantities of shell residues are generated and are generally discarded without further utilization. Following carbonization, these shells produce charcoal with relatively high carbon content and a rigid lignocellulosic structure, indicating their suitability as a feedstock for renewable solid biofuel production. Nevertheless, compared with conventional feedstocks such as coconut shells, bamboo, sawdust, rice husks, and oil palm residues, the utilization of *P. edule* shell waste for charcoal briquette production has received little scientific attention.

Binder selection is a critical factor influencing the physicochemical and mechanical

quality of charcoal briquettes. Starch-based binders are widely used because they are biodegradable, inexpensive, and environmentally friendly. However, commercial starches may increase production costs and compete with food resources. Recovering starch from cassava processing residues offers a sustainable alternative by converting agro-industrial waste into a renewable bio-based adhesive (Obi et al., 2022). This approach not only minimizes waste generation but also promotes circular resource utilization through the replacement of virgin materials with recovered biomass-derived products (Aransiola et al., 2019).

In addition to starch-based binders, renewable natural additives can further improve briquette quality. Pine (*Pinus merkusii*) resin is a renewable non-timber forest product rich in resin acids and terpenoid compounds that exhibit adhesive and combustible properties. Compared with petroleum-derived additives, pine resin is biodegradable, renewable, and environmentally compatible with sustainable biofuel production. Previous studies have demonstrated that pine resin can improve ignition characteristics and increase the calorific value of charcoal briquettes while maintaining their renewable nature (Pari et al., 2023).

Numerous studies have investigated charcoal briquettes produced from coconut shells, bamboo, rice husks, sawdust, sugarcane bagasse, and oil palm biomass. These studies consistently demonstrate that binder composition significantly affects moisture content, volatile matter, fixed carbon, density, compressive strength, and combustion performance (Tumuluru et al., 2011; Sanchez-Roque et al., 2025). However, most previous investigations have focused on conventional feedstocks and single binder systems. The integration of underutilized forestry residues with waste-derived starch binders and renewable forest products has rarely been explored, particularly as a strategy to simultaneously enhance physicochemical, mechanical, and combustion properties while supporting circular biomass utilization (Eling et al., 2024). Recent studies have highlighted the growing need to develop biofuel materials from locally available biomass wastes combined with renewable additives to improve both environmental sustainability and fuel performance; however, systematic evaluations of multi-binder systems remain limited (Abah et al., 2025; Sanchez-Roque et al., 2025).

Despite numerous studies on biomass briquettes and natural binders, the combined use of *Pangium edule* shell biomass with cassava waste starch and pine resin has not been systematically investigated. Previous studies have primarily investigated either individual natural binders or alternative biomass feedstocks, whereas very few studies have evaluated the synergistic effects of combining two renewable binders with an underutilized forest biomass in a single briquetting system (Wu et al., 2025; Sanchez-Roque et al., 2025). Moreover, current knowledge remains insufficient regarding how the interaction between starch- and resin-based binders governs the physicochemical, mechanical, and fuel properties of biomass briquettes. Consequently, the relationships between binder composition and briquette quality, as well as the mechanisms governing these changes, have not yet been clearly established.

Therefore, this study was designed to fill this knowledge gap by investigating how different proportions of cassava waste starch and pine resin affect the physical, mechanical, and fuel characteristics of briquettes produced from *Pangium edule* shell biomass. We hypothesized that combining starch and resin would produce complementary binding mechanisms capable of improving briquette integrity and combustion performance beyond that achievable with a single natural binder. The scientific objective of this work was not only to evaluate briquette quality but also to determine the relationship between binder composition and key fuel properties and to identify the binder combination providing the most balanced performance.

MATERIALS AND METHODS

Raw materials

Shell waste of *Pangium edule* Reinw. was collected from traditional food-processing industries in South Sulawesi, Indonesia. The shells were cleaned with tap water to remove adhering impurities, air-dried, and carbonized using a drum kiln under limited oxygen conditions. The resulting charcoal was crushed and sieved through a 60-mesh screen to obtain a uniform particle size. Cassava waste starch recovered from tapioca-processing residues was used as the primary bio-based binder. Pine

(*Pinus merkusii*) resin obtained from local resin producers served as a natural additive. Distilled water was used during binder preparation to ensure homogeneous mixing.

Briquette preparation

Cassava waste starch was first gelatinized by heating with distilled water until a homogeneous adhesive paste was obtained. The starch paste was then thoroughly mixed with *P. edule* shell charcoal powder, followed by the addition of pine resin according to the respective experimental treatment. The mixture was manually homogenized until a uniform consistency was achieved. Prior to mixing, the charcoal was ground and passed through a 60-mesh sieve to obtain a relatively uniform particle size.

The target briquette density was set at 0.70 g cm^{-3} . A cylindrical steel mold with an internal diameter of 5.5 cm and a height of 3.5 cm was used, corresponding to a mold volume of approximately 83.11 cm^3 . Based on the target density, 58.18 g of the prepared briquette mixture was required for each mold ($0.70 \text{ g cm}^{-3} \times 83.11 \text{ cm}^3$). This target mass was maintained consistently across all experimental treatments to standardize the initial material loading and facilitate comparison of briquette properties.

The experimental preparation sequence is illustrated in Figure 1. The raw materials, including *P. edule* shell and pine resin, are shown in Figure 1a, while the resulting *P. edule* shell charcoal is presented in Figure 1b. Cassava waste starch was prepared as the binder as shown in Figure 1c, after which the charcoal and binders were thoroughly mixed (Figure 1d). The resulting mixture was then compacted using the manually operated hydraulic press shown in Figure 1e. An estimated pressing pressure of approximately 20.6 MPa (equivalent to approximately 210 kg cm^{-2} , based on a nominal 5-ton-force pressing load distributed over the 5.5-cm-diameter mold area) was applied and maintained for 60 s before pressure release. The same estimated pressing pressure, holding time, pressing procedure, and mold configuration were consistently applied to all experimental treatments to minimize variation in compaction conditions. The molded briquettes were subsequently oven-dried at $105 \pm 2^\circ\text{C}$ until constant weight, as illustrated in Figure 1f, cooled in a desiccator, and then subjected to physicochemical characterization.



Figure 1. Experimental setup and briquette preparation process, (a) raw materials, including *Pangium edule* shell and pine resin, (b) *Pangium edule* shell charcoal, (c) preparation of cassava waste starch binder, (d) mixing of charcoal and binders, (e) briquette molding and pressing, and (f) drying process

Experimental design

The experiment was conducted using a completely randomized design (CRD) with a two-factor factorial arrangement to evaluate the effects of cassava waste starch and pine resin as bio-based binders on the properties of *P. edule* shell charcoal briquettes. The first factor was the concentration of cassava waste starch, consisting of three levels: 8 wt% (A1), 12 wt% (A2), and 16 wt% (A3). The second factor was pine resin addition at two levels: 0 wt% (B1) and 5 wt% (B2). The combination of these factors generated six treatment combinations (A1B1, A1B2, A2B1, A2B2, A3B1, and A3B2). Each treatment was replicated three times, resulting in a total of eighteen experimental units. The measured response variables included proximate composition (moisture content, volatile matter, ash content, and fixed carbon), physical properties (density and compressive strength), and combustion performance (ignition time and higher heating value). This experimental design enabled the evaluation of the effects of

both individual binders and their combined interaction on the overall quality of the briquettes.

Briquette characterization

Proximate analysis

Moisture content

Moisture content was determined according to the Indonesian National Standard (SNI 01-6235-2000). Approximately 2 g of briquette sample was dried in an oven at 105 ± 2 °C until constant mass. Moisture content was calculated as the percentage of weight loss relative to the initial sample weight.

Ash content

Ash content was determined following ASTM D3174. Oven-dried briquette samples were combusted in a muffle furnace at 750 °C until complete oxidation of combustible materials. The remaining inorganic residue was weighed and expressed as a percentage of the oven-dry sample.

Volatile matter

Volatile matter was determined in accordance with ASTM D3175. Approximately 1 g of oven-dried sample was heated in a covered crucible at 950 ± 20 °C for 7 min. The weight loss, excluding moisture, was reported as volatile matter content.

Fixed carbon

Fixed carbon content was calculated indirectly from the results of proximate analysis using the following equation:

$$\text{Fixed Carbon (\%)} = 100 - (\text{Moisture} + \text{Ash} + \text{Volatile Matter}) \quad (1)$$

Physical properties

Density

Briquette density was determined by measuring the oven-dry mass of each briquette using an analytical balance (Ohaus, Pioneer PX224, readability 0.0001 g) and calculating its volume from the measured diameter and height using a digital caliper (Mitutoyo, CD-6"CSX, resolution 0.01 mm). The volume was calculated using the cylindrical geometry equation:

$$V = \pi(d/2)^2 h \quad (2)$$

where: d is the briquette diameter and h is its height.

Density was calculated as the ratio of oven-dry mass to briquette volume and expressed in g cm^{-3} . Three briquettes were measured independently for each treatment ($n = 3$).

Compressive strength

Compressive strength was measured using a Universal Testing Machine (UTM; Instron, Model 3369, maximum load capacity 50 kN). Prior to testing, the briquettes were conditioned at room temperature (25 ± 2 °C) for 24 h. Each briquette was positioned vertically and subjected to axial compressive loading at a constant cross-head speed of 5 mm min^{-1} until structural failure occurred. The maximum compressive load sustained immediately before failure was recorded as the failure load. Compressive strength was calculated by dividing the maximum failure load by the initial cross-sectional area of the briquette,

$$CS = F/A \quad (3)$$

where: F is the maximum failure load and A is the cross-sectional area.

The resulting compressive strength was expressed in kg cm^{-2} . Three independent briquettes were tested for each treatment ($n = 3$), and the same loading configuration and testing procedure were applied consistently across all treatments.

Combustion properties

Ignition time

Ignition time was evaluated by exposing each briquette to a constant flame under ambient laboratory conditions. The ignition time was recorded as the elapsed time required for sustained combustion after initial flame contact and expressed in seconds.

Calorific value/higher heating value (HHV)

The calorific value/higher heating value (HHV) of the briquettes was determined using an oxygen bomb calorimeter in accordance with ASTM D5865. Prior to analysis, briquette samples were oven-dried at 105 ± 2 °C to constant mass to minimize the influence of residual moisture on the calorific value determination. Approximately 1.0 g of the dried and homogenized sample was placed in the combustion crucible and combusted in an oxygen atmosphere at an initial pressure of approximately 30 atm within the sealed bomb vessel. The temperature rise of the surrounding water jacket was recorded automatically, and the gross calorific value (HHV) was calculated based on the calibrated calorimeter response and the appropriate standard correction factors. The calorimeter was calibrated prior to analysis according to the manufacturer's procedure and the requirements of the applicable standard. Three independent measurements were performed for each treatment ($n = 3$), and the results were reported as kcal kg^{-1} .

Measurement uncertainty and potential experimental errors were considered throughout the briquette characterization procedures to ensure the reliability and reproducibility of the experimental results. The principal sources of experimental variability included differences in charcoal particle size, binder distribution, moisture content, briquette dimensions, compaction conditions, and drying consistency. These sources of variability were minimized through standardized sample preparation and testing procedures,

including the use of charcoal particles passed through a 60-mesh sieve, consistent binder formulations and target mixture mass, identical mold dimensions, uniform pressing procedures and holding time, and drying at 105 ± 2 °C until constant weight. The same measurement procedures and instrument settings were applied consistently across all treatments. Replicate briquette specimens were analyzed under identical experimental conditions, and the resulting mean values were used for subsequent statistical comparisons. These measures were implemented to minimize systematic and random sources of variation and to ensure comparability among treatments.

Statistical analysis

The experimental data were analyzed using two-way analysis of variance (ANOVA) under a completely randomized factorial design to evaluate the main effects of cassava waste starch concentration, pine resin addition, and their interaction on the physicochemical, mechanical, and combustion properties of the briquettes. The experimental design consisted of three levels of cassava waste starch concentration and two levels of pine resin addition, with three independent replicates for each treatment ($n = 3$). For each response variable, the ANOVA model was used to determine the effects of starch concentration, pine resin addition, and their interaction. The corresponding F-statistics and p-values were reported, with statistical significance evaluated at $\alpha = 0.05$ (95% confidence level). When significant differences were detected ($p < 0.05$), treatment means were separated using Tukey's honestly significant difference (HSD) post-hoc test. All results are presented as mean $\pm$ standard deviation (SD), and different superscript letters indicate significant differences among treatment means according to Tukey's HSD test. Where appropriate, 95% confidence intervals were also calculated to describe the precision of the estimated treatment means.

Limitations of the study

This study has several limitations that should be considered when interpreting the results. First, only one biomass source (*Pangium edule*) shell and two natural binders (cassava waste starch and pine resin) were investigated. Therefore, the findings cannot be directly generalized to other lignocellulosic feedstocks or binder systems without

additional experimental validation. Second, the experiments were performed under a single set of briquetting conditions. The influence of processing parameters such as compaction pressure, pressing time, drying conditions, and particle size distribution was not systematically investigated, although these factors may significantly affect the physical and combustion properties of biomass briquettes. Third, the mechanisms responsible for the improved briquette performance were inferred from the observed physical and fuel properties rather than confirmed through microstructural or chemical characterization. Advanced analytical techniques such as scanning electron microscopy (SEM), Fourier-transform infrared spectroscopy (FTIR), thermogravimetric analysis (TGA), and X-ray diffraction (XRD) would provide additional evidence regarding binder interactions and structural changes during briquette formation. Finally, the study focused on laboratory-scale production and standard fuel characterization. Long-term storage stability, combustion emissions, ash composition, mechanical durability during transportation, and pilot- or industrial-scale production were not evaluated and should be addressed in future studies. Future research should optimize briquetting parameters using multi-factor experimental designs, investigate additional environmentally friendly binders, validate the proposed mechanisms through instrumental analyses, and assess the environmental and economic feasibility of large-scale production.

RESULTS

Effect of cassava waste starch and pine resin on proximate composition

Proximate composition provides important information on the fuel characteristics of biomass briquettes because moisture, volatile matter, ash, and fixed carbon collectively influence ignition behavior, combustion characteristics, and energy performance. The effects of cassava waste starch concentration and pine resin addition on the proximate composition of *Pangium edule* shell charcoal briquettes are presented in Figure 2. Across the six formulations, moisture content ranged from 3.45 to 7.15%, volatile matter from 20.67 to 29.15%, ash content from 2.01 to 2.71%, and fixed carbon from 61.00 to 73.86%.

Moisture and volatile matter generally increased with increasing binder concentration. The lowest moisture content was recorded in A1B1 (8% starch + 0% resin) at 3.45%, whereas the highest was observed in A3B2 (16% starch + 5% resin) at 7.15%. A similar pattern was observed for volatile matter, which increased from 20.67% in A1B1 to 29.15% in A3B2. At each starch level, the addition of 5% pine resin increased moisture and volatile matter: from 3.45 to 5.10% and from 20.67 to 22.80% at 8% starch; from 5.85 to 5.35% and from 24.90 to 23.50% at 12% starch; and from 6.50 to 7.15% and from 27.60 to 29.15% at 16% starch. The responses were therefore not strictly monotonic for every pair of treatments, indicating that the effects of starch and resin should be interpreted within the factorial treatment structure rather than from binder concentration alone.

Ash content exhibited a comparatively narrow range of 2.01–2.71% across the treatments. The lowest ash content was observed in A1B1 (2.01%), while the highest occurred in A3B2 (2.71%). The relatively small absolute variation suggests that binder formulation had a less pronounced effect on ash accumulation than on moisture, volatile matter, and fixed carbon. The ash values remained consistently low across all formulations, indicating that the charcoal feedstock contributed predominantly to the mineral residue.

Fixed carbon showed a contrasting response, ranging from 61.00 to 73.86%. The highest value occurred in A1B1 (73.86%), followed by A2B2 (72.97%), whereas the lowest value was recorded in A3B2 (61.00%). Increasing starch concentration from 8 to 16% was generally associated with

lower fixed carbon, particularly in the resin-containing treatments, where the value declined from 69.90% in A1B2 to 61.00% in A3B2. However, the response at 12% starch differed between resin levels, with A2B1 showing 66.77% fixed carbon and A2B2 showing 72.97%. This variation indicates that the effect of binder composition on fixed carbon was not adequately described by a simple linear relationship and supports the use of factorial statistical analysis to evaluate the main and interaction effects.

The combination of these four parameters demonstrates a clear trade-off in briquette formulation. Higher binder incorporation was generally associated with increased moisture and volatile matter, whereas formulations with lower binder loading tended to retain a larger proportion of fixed carbon. Nevertheless, A2B2 maintained a relatively high fixed carbon content (72.97%) together with moderate moisture (5.35%), volatile matter (23.50%), and ash (2.18%). This combination provides a comparatively balanced proximate composition and helps explain why A2B2 was subsequently identified as the most favorable formulation when the chemical, physical, and combustion properties were considered simultaneously.

The proximate composition of *Pangium edule* shell charcoal briquettes produced using different concentrations of cassava waste starch and pine resin is presented in Table 1. Moisture content ranged from $3.45 \pm 0.09\%$ to $7.15 \pm 0.18\%$, volatile matter from $20.67 \pm 0.31\%$ to $29.15 \pm 0.39\%$, ash content from $2.01 \pm 0.05\%$ to $2.71 \pm 0.12\%$, and fixed carbon from $61.00 \pm 0.46\%$ to $73.86 \pm 0.42\%$. Two-way ANOVA showed

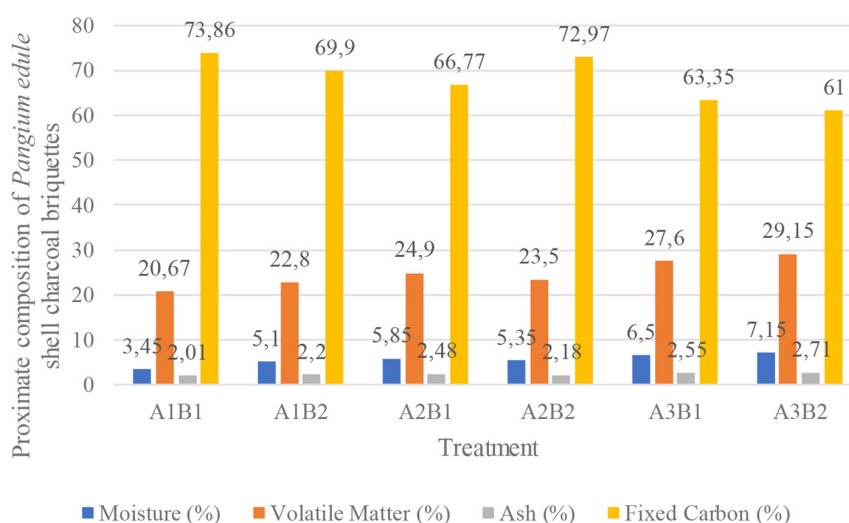


Figure 2. Proximate composition of *Pangium edule* shell charcoal briquettes

that cassava waste starch concentration significantly affected all four proximate properties ($p < 0.001$). Pine resin addition significantly affected moisture content ($F_{1,2} = 52.626$, $p < 0.001$) and volatile matter ($F_{1,2} = 18.304$, $p = 0.001$), but not ash content ($F_{1,2} = 0.155$, $p = 0.701$) or fixed carbon ($F_{1,2} = 0.029$, $p = 0.867$). Importantly, the starch $\times$ pine resin interaction was significant for moisture content ($F_{2,12} = 56.402$, $p < 0.001$), volatile matter ($F_{2,12} = 37.852$, $p < 0.001$), ash content ($F_{2,12} = 14.027$, $p = 0.001$), and fixed carbon ($F_{2,12} = 215.221$, $p < 0.001$). These results indicate that the response of briquette proximate composition to one binder component depended on the level of the other component.

Moisture content increased from $3.45 \pm 0.09\%$ in A1B1 to $7.15 \pm 0.18\%$ in A3B2. Tukey's HSD test showed a clear separation among most treatment means, with A1B1, A2B1, A3B1, and A3B2 belonging to distinct statistical groups, whereas A1B2 and A2B2 were statistically similar (both assigned superscript b). The increase in moisture with higher starch concentration is consistent with the hydrophilic characteristics of cassava starch, whose hydroxyl-rich structure can facilitate water absorption and retention within the briquette matrix. However, the significant starch $\times$ resin interaction indicates that the effect of binder composition on moisture content cannot be interpreted solely as an independent main effect. The interaction was therefore an important component of the observed moisture response. Despite the differences among treatments, all

formulations remained below the 8% moisture limit specified by SNI 01-6235-2000, indicating compliance with this criterion.

Volatile matter increased from $20.67 \pm 0.31\%$ in A1B1 to $29.15 \pm 0.39\%$ in A3B2. Tukey's HSD test identified five distinct statistical groups, with A1B1, A1B2/A2B2, A2B1, A3B1, and A3B2 forming progressively higher groups according to their superscript assignments. The significant effects of starch concentration, pine resin addition, and their interaction indicate that both binder components contributed to variation in the volatile fraction. The increase is attributable to the incorporation of organic constituents from cassava starch and pine resin, which contribute thermally degradable material to the briquette matrix. Higher volatile matter can facilitate ignition by generating combustible gases during the initial heating stage; however, excessive volatile release may also accelerate devolatilization and fuel consumption. All treatments exceeded the 15% reference value specified in SNI 01-6235-2000, indicating that the briquettes did not comply with this particular volatile-matter criterion, despite meeting the moisture and ash requirements.

Ash content varied within a relatively narrow range, from $2.01 \pm 0.05\%$ in A1B1 to $2.71 \pm 0.12\%$ in A3B2. The ANOVA results showed that starch concentration significantly affected ash content ($F_{2,12} = 51.601$, $p < 0.001$), whereas pine resin alone had no significant effect ($F_{1,12} = 0.155$, $p = 0.701$). Nevertheless, the starch $\times$ resin interaction was significant ($F_{2,12} = 14.027$,

Table 1. Effect of cassava waste starch concentration and pine resin addition on the proximate composition of *Pangium edule* shell charcoal briquettes

Treatment	Moisture (%)	Volatile matter (%)	Ash (%)	Fixed carbon (%)
A1B1 (8% starch + 0% resin)	3.45 ± 0.09^a	20.67 ± 0.31^a	2.01 ± 0.05^a	73.86 ± 0.42^e
A1B2 (8% starch + 5% resin)	5.10 ± 0.16^b	22.80 ± 0.27^b	2.20 ± 0.08^a	69.90 ± 0.36^d
A2B1 (12% starch + 0% resin)	5.85 ± 0.21^c	24.90 ± 0.43^c	2.48 ± 0.11^b	66.77 ± 0.51^c
A2B2 (12% starch + 5% resin)	5.35 ± 0.13^b	23.50 ± 0.34^b	2.18 ± 0.07^a	72.97 ± 0.38^e
A3B1 (16% starch + 0% resin)	6.50 ± 0.24^d	27.60 ± 0.48^d	2.55 ± 0.09^b	63.35 ± 0.57^b
A3B2 (16% starch + 5% resin)	7.15 ± 0.18^e	29.15 ± 0.39^e	2.71 ± 0.12^b	61.00 ± 0.46^a

Note: Values are presented as mean $\pm$ standard deviation (SD; $n = 3$). Different superscript letters within the same column indicate significant differences among treatment means according to Tukey's HSD post-hoc test at $p < 0.05$. Two-way ANOVA showed that cassava waste starch concentration significantly affected moisture content ($F_{2,12} = 317.014$, $p < 0.001$), volatile matter ($F_{2,12} = 476.031$, $p < 0.001$), ash content ($F_{2,12} = 51.601$, $p < 0.001$), and fixed carbon ($F_{2,12} = 757.531$, $p < 0.001$). Pine resin addition also significantly affected moisture content ($F_{1,12} = 52.626$, $p < 0.001$), volatile matter ($F_{1,12} = 18.304$, $p = 0.001$), whereas no significant was detected for ash content ($F_{1,12} = 0.155$, $p = 0.701$), fixed carbon ($F_{1,12} = 0.029$, $p = 0.867$). The starch $\times$ pine resin interaction was significant for moisture content ($F_{2,12} = 56.402$, $p < 0.001$), volatile matter ($F_{2,12} = 37.852$, $p < 0.001$), ash content ($F_{2,12} = 14.027$, $p = 0.001$), and fixed carbon ($F_{2,12} = 215.221$, $p < 0.001$).

$p = 0.001$), indicating that the response of ash content to starch concentration varied according to resin addition. Tukey's HSD test separated the treatments into two statistical groups: A1B1, A1B2, and A2B2 were statistically similar (group a), whereas A2B1, A3B1, and A3B2 formed group b. Thus, although the numerical difference between the lowest and highest ash contents was relatively small, the treatment structure produced statistically detectable differences. All treatments remained well below the SNI maximum ash limit of 8%, indicating a relatively low mineral residue content.

Fixed carbon showed a contrasting response, declining from $73.86 \pm 0.42\%$ in A1B1 to $61.00 \pm 0.46\%$ in A3B2. Cassava waste starch concentration had a highly significant effect on fixed carbon ($F_{2,12} = 757.531$, $p < 0.001$), whereas pine resin addition alone did not significantly affect this parameter ($F_{1,12} = 0.029$, $p = 0.867$). However, the starch $\times$ resin interaction was highly significant ($F_{2,12} = 215.221$, $p < 0.001$), demonstrating that the effect of starch concentration on fixed carbon depended strongly on the presence or absence of pine resin. Tukey's HSD test identified five statistical groups. A1B1 and A2B2 shared the same superscript (e), indicating no significant difference between their fixed carbon contents, whereas A1B2, A2B1, A3B1, and A3B2 occupied progressively lower statistical groups. This result is particularly relevant because A2B2 maintained a high fixed carbon content of $72.97 \pm 0.38\%$, statistically comparable with the highest value recorded in A1B1 ($73.86 \pm 0.42\%$), despite containing both starch and pine resin.

The distinct responses of the four proximate properties indicate that binder formulation generated a multidimensional effect on briquette fuel characteristics rather than a uniform improvement or deterioration across all parameters. Increasing starch concentration generally increased moisture and volatile matter while reducing fixed carbon, whereas the effect of pine resin was parameter-dependent. The significant interactions for all four proximate properties further demonstrate that the contribution of pine resin cannot be interpreted independently of starch concentration. This interaction was particularly pronounced for fixed carbon, as evidenced by the very high interaction F-value ($F_{2,12} = 215.221$).

Among the treatments, A2B2 (12% cassava waste starch + 5% pine resin) exhibited a comparatively balanced proximate profile, with 5.35

$\pm 0.13\%$ moisture, $23.50 \pm 0.34\%$ volatile matter, $2.18 \pm 0.07\%$ ash, and $72.97 \pm 0.38\%$ fixed carbon. Although A1B1 had the lowest moisture and highest fixed carbon, it lacked pine resin and subsequently exhibited less favorable physical and ignition characteristics in the subsequent analyses. Conversely, the higher-binder treatments A3B1 and A3B2 showed increased moisture and volatile matter accompanied by substantially reduced fixed carbon. Therefore, A2B2 was not selected as the optimum formulation solely on the basis of proximate composition; rather, its suitability was further evaluated by integrating its chemical characteristics with density, compressive strength, ignition time, and higher heating value. This multi-response interpretation provides a more robust basis for identifying the final formulation.

Effect on physical properties

Physical properties are important indicators of briquette quality because they influence structural integrity, resistance to mechanical damage, and the efficiency of handling, transportation, and storage. The physical appearance of the *Pangium edule* shell charcoal briquettes produced under different binder formulations is presented in Figure 3. The briquettes exhibited a relatively uniform cylindrical form across the treatments, although differences in surface appearance and compactness were visually apparent among formulations.

Physical properties are important indicators of briquette quality because they determine structural integrity, resistance to mechanical damage, and the efficiency of handling, transportation, and storage. Density reflects the degree of particle consolidation within the briquette matrix, whereas compressive strength indicates its resistance to deformation and fracture under mechanical loading. The effects of cassava waste starch concentration and pine resin addition on the density and compressive strength of *Pangium edule* shell charcoal briquettes are shown in Figure 4.

As shown in Figure 4, density increased from 0.60 g cm^{-3} in A1B1 to 0.62 g cm^{-3} in A1B2 and 0.64 g cm^{-3} in A2B1, reaching the highest value of 0.66 g cm^{-3} in A2B2. At the highest starch concentration, density declined slightly to 0.65 g cm^{-3} in both A3B1 and A3B2. Thus, the density response was not proportional to binder concentration across the entire experimental range. The maximum density at A2B2 suggests that the combination of 12% cassava waste starch and 5% pine resin provided



Figure 3. Physical appearance of *Pangium edule* shell charcoal briquettes produced under different binder formulations

favorable particle consolidation, whereas further increasing starch concentration to 16% did not produce an additional increase in density.

Compressive strength exhibited a more pronounced response to binder formulation than density. The strength increased from 3.45 kg cm^{-2} in A1B1 to 5.12 kg cm^{-2} in A1B2 and 5.95 kg cm^{-2} in A2B1, reaching a maximum of 7.02 kg cm^{-2} in A2B2. At 16% starch, compressive strength decreased slightly to 6.58 kg cm^{-2} in A3B1, but increased marginally to 6.73 kg cm^{-2} in A3B2 following the addition of 5% pine resin. The response therefore indicates that moderate binder incorporation produced the greatest mechanical resistance, while further increasing starch concentration beyond 12% did not result in a corresponding increase in strength.

The comparison between treatments with and without pine resin further illustrates the contribution of the resin component. At 8% starch, the addition of 5% pine resin increased density from 0.60 to 0.62 g cm^{-3} and compressive strength from 3.45 to 5.12 kg cm^{-2} . At 12% starch, the corresponding increases were from 0.64 to 0.66 g cm^{-3} and from 5.95 to 7.02 kg cm^{-2} , respectively. At 16% starch, however, resin addition did not alter density (0.65 g cm^{-3} in both A3B1 and A3B2) but increased compressive strength from 6.58 to 6.73 kg cm^{-2} . These results indicate that the contribution of pine resin to physical performance was dependent on the starch concentration, with its most pronounced effect occurring at the intermediate starch level.

The observed pattern is consistent with the role of cassava starch and pine resin as complementary binding agents. Gelatinized cassava starch can promote particle consolidation by

filling interparticle voids and forming a continuous adhesive matrix, while pine resin can strengthen interparticle adhesion through its adhesive and thermoplastic characteristics. At the intermediate formulation, these effects appear to have produced a favorable balance between particle packing and bonding strength. However, the slight decline in density and compressive strength at 16% starch indicates that increasing binder concentration beyond the intermediate level did not provide proportional improvements in physical performance. This suggests that the effectiveness of the binder system depends on formulation rather than simply on maximizing binder content. Among the evaluated formulations, A2B2 (12% cassava waste starch + 5% pine resin) exhibited the highest density (0.66 g cm^{-3}) and compressive strength (7.02 kg cm^{-2}). The combination of these two properties indicates that A2B2 achieved the most favorable physical performance under the experimental conditions. Importantly, this formulation was subsequently evaluated together with proximate composition, ignition time, and higher heating value to determine its suitability based on a multi-response assessment rather than physical properties alone.

The physical properties of *Pangium edule* shell charcoal briquettes produced using different concentrations of cassava waste starch and pine resin are presented in Table 2. Density ranged from 0.60 ± 0.008 to $0.66 \pm 0.012 \text{ g cm}^{-3}$, whereas compressive strength ranged from 3.45 ± 0.17 to $7.02 \pm 0.28 \text{ kg cm}^{-2}$. Two-way ANOVA showed that cassava waste starch concentration significantly affected density ($F_{2,12} = 28.277$, $p < 0.001$) and compressive strength ($F_{2,12} = 179.551$, $p < 0.001$). Pine resin addition also significantly affected density ($F_{1,12} = 7.069$, $p = 0.021$) and

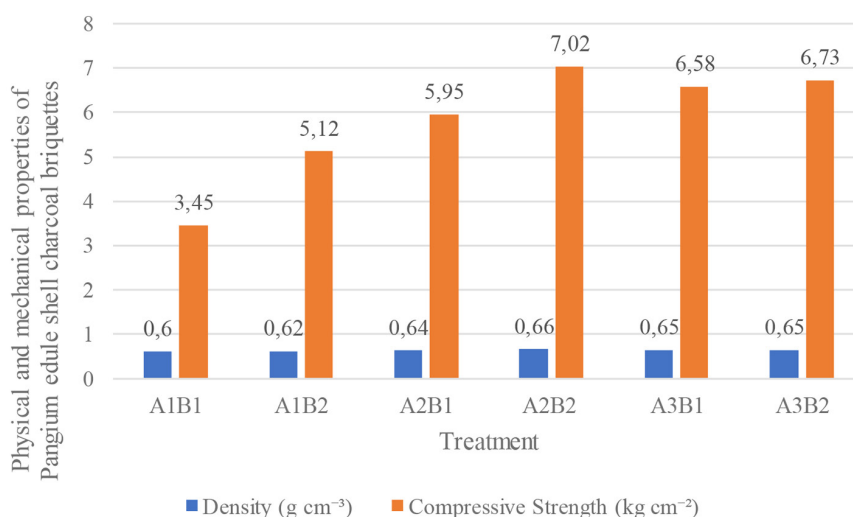


Figure 4. Physical and mechanical properties of *Pangium edule* shell charcoal briquettes

compressive strength ($F_{1,2} = 71.508$, $p < 0.001$). The starch $\times$ pine resin interaction was not significant for density ($F_{2,12} = 1.767$, $p = 0.212$), but was significant for compressive strength ($F_{2,12} = 15.055$, $p = 0.001$). Tukey's HSD post-hoc test further identified significant differences among treatment means, as indicated by the superscript letters in Table 2.

Density increased from 0.60 ± 0.008 g cm⁻³ in A1B1 to 0.62 ± 0.011 g cm⁻³ in A1B2, 0.64 ± 0.009 g cm⁻³ in A2B1, and reached 0.66 ± 0.012 g cm⁻³ in A2B2. Tukey's HSD test indicated overlapping statistical groups across these treatments, with A1B1 assigned group a, A1B2 group ab, A2B1 group bc, and A2B2 group c. Thus, A1B1 differed significantly from A2B1 and A2B2, whereas A1B2 was not significantly different from either A1B1 or A2B1, and A2B1 was not significantly different from A1B2 or A2B2. At the highest starch concentration, density decreased slightly to 0.65 ± 0.010 g cm⁻³ in A3B1 and 0.65 ± 0.013 g cm⁻³ in A3B2; both treatments shared group c with A2B2, indicating that their densities were not significantly different from A2B2. Accordingly, A2B2, A3B1, and A3B2 formed the highest statistical group for density, despite A2B2 having the numerically highest mean. The significant main effects of starch concentration and resin addition, together with the absence of a significant $A \times B$ interaction, indicate that density was primarily associated with the independent effects of the two binder components rather than their statistically demonstrable interaction.

The increase in density at the intermediate formulation is consistent with improved particle

consolidation during compaction. Gelatinized cassava starch may have filled interparticle voids and increased cohesion within the charcoal matrix, while pine resin may have contributed additional adhesion between adjacent particles. However, these mechanisms should be regarded as plausible interpretations rather than directly demonstrated mechanisms because no microstructural or spectroscopic analyses were conducted in the present study. The slight decline from 0.66 g cm⁻³ in A2B2 to 0.65 g cm⁻³ in A3B1 and A3B2 further suggests that increasing starch concentration beyond 12% did not produce additional densification under the experimental conditions.

Compressive strength exhibited a stronger response to binder formulation than density. The lowest compressive strength was recorded in A1B1 (3.45 ± 0.17 kg cm⁻²), followed by A1B2 (5.12 ± 0.22 kg cm⁻²), A2B1 (5.95 ± 0.19 kg cm⁻²), A3B1 (6.58 ± 0.25 kg cm⁻²), A3B2 (6.73 ± 0.31 kg cm⁻²), and A2B2 (7.02 ± 0.28 kg cm⁻²). Tukey's HSD test assigned A1B1, A1B2, A2B1, and A2B2 to distinct statistical groups (a, b, c, and d, respectively). A3B1 was assigned group cd, indicating that its compressive strength was not significantly different from either A2B1 or A2B2. Similarly, A3B2 was assigned group d and was therefore not significantly different from A2B2. Thus, although A2B2 had the highest numerical compressive strength, the Tukey results indicate that its value was statistically comparable with A3B1 and A3B2.

The significant main effects of starch concentration and pine resin addition, together with the significant $A \times B$ interaction, indicate that

Table 2. Effect of cassava waste starch concentration and pine resin addition on the physical properties of *Pangium edule* shell charcoal briquettes

Treatment	Density (g cm ⁻³)	Compressive strength (kg cm ⁻²)
A1B1 (8% starch + 0% resin)	0.60 ± 0.008 ^a	3.45 ± 0.17 ^a
A1B2 (8% starch + 5% resin)	0.62 ± 0.011 ^{ab}	5.12 ± 0.22 ^b
A2B1 (12% starch + 0% resin)	0.64 ± 0.009 ^{bc}	5.95 ± 0.19 ^c
A2B2 (12% starch + 5% resin)	0.66 ± 0.012 ^c	7.02 ± 0.28 ^d
A3B1 (16% starch + 0% resin)	0.65 ± 0.010 ^c	6.58 ± 0.25 ^{cd}
A3B2 (16% starch + 5% resin)	0.65 ± 0.013 ^c	6.73 ± 0.31 ^d

Note: Values are presented as mean ± standard deviation (SD; n = 3). Different superscript letters within the same column indicate significant differences among treatment means based on Tukey's HSD post-hoc test at $p < 0.05$. Two-way ANOVA was performed to evaluate the effects of cassava waste starch concentration (A), pine resin addition (B), and their interaction (A × B). For density, starch concentration significantly affected the response ($F_{2,12} = 28.277$, $p < 0.001$), as did pine resin addition ($F_{1,12} = 7.069$, $p = 0.021$), whereas the A × B interaction was not significant ($F_{2,12} = 1.767$, $p = 0.212$). For compressive strength, significant effects were observed for starch concentration ($F_{2,12} = 179.551$, $p < 0.001$), pine resin addition ($F_{1,12} = 71.508$, $p < 0.001$), and the A × B interaction ($F_{2,12} = 15.055$, $p = 0.001$).

compressive strength was determined by both the individual binder effects and their combined formulation. The increase from 3.45 ± 0.17 kg cm⁻² in A1B1 to 7.02 ± 0.28 kg cm⁻² in A2B2 indicates a substantial improvement in mechanical resistance at the intermediate binder combination. This response is consistent with enhanced interparticle cohesion associated with binder incorporation. Nevertheless, because the present study did not directly characterize the briquette microstructure, the proposed contributions of starch gelatinization and pine-resin adhesion should not be interpreted as experimentally verified mechanisms.

Increasing starch concentration from 12% to 16% did not result in a further increase in compressive strength. In the absence of resin, strength increased from 5.95 ± 0.19 kg cm⁻² in A2B1 to 6.58 ± 0.25 kg cm⁻² in A3B1, whereas in the presence of 5% resin, the corresponding increase from A2B2 to A3B2 was not statistically significant, with values of 7.02 ± 0.28 and 6.73 ± 0.31 kg cm⁻², respectively. This pattern, together with the significant A × B interaction, suggests that the contribution of starch concentration to mechanical strength depended on resin addition. The results therefore do not support a simple assumption that increasing binder concentration continuously improves briquette strength.

Among the tested formulations, A2B2 (12% cassava waste starch + 5% pine resin) produced the highest numerical density (0.66 ± 0.012 g cm⁻³) and compressive strength (7.02 ± 0.28 kg cm⁻²). Its density was statistically comparable with A3B1 and A3B2, while its compressive strength was

statistically comparable with A3B1 and A3B2. Therefore, A2B2 should be described as the formulation with the most favorable numerical physical performance rather than as being statistically superior to every other treatment for both parameters. Its selection as the preferred formulation was subsequently based on integration with the proximate composition, ignition time, and higher heating value, rather than on density or compressive strength alone.

From an engineering perspective, the relatively high density and compressive strength of A2B2 indicate favorable structural characteristics for handling and transportation, where resistance to mechanical damage is important. However, these results should be interpreted within the laboratory-scale conditions of the present study. Long-term storage stability, abrasion resistance during transportation, and performance under pilot- or industrial-scale processing were not evaluated and therefore require further validation. In addition, the proposed roles of starch gelatinization and pine-resin adhesion remain mechanistic interpretations based on the observed physical responses, rather than direct evidence from SEM, FTIR, TGA, or XRD analyses.

Effect on combustion performance

Combustion performance is a key criterion for evaluating biomass briquettes because it determines ignition behavior, heat release, and overall energy conversion efficiency during utilization. In the present study, combustion

performance was assessed using ignition time and higher heating value (HHV), which together represent the ease of ignition and the energy content of the briquettes. The effects of cassava waste starch concentration and pine resin addition on ignition time and HHV are presented in Figures 5 and 6.

As shown in Figure 5, ignition time varied substantially among the binder formulations, ranging from 41 to 128 s. Briquettes without pine resin generally required longer ignition times than those containing 5% pine resin at the corresponding starch concentrations. The longest ignition time was recorded for A1B1 (128 s), followed by A2B1 (95 s) and A3B1 (82 s). In contrast, the addition of 5% pine resin reduced ignition time to 68 s in A1B2, 41 s in A2B2, and 48 s in A3B2. The shortest ignition time was therefore observed in A2B2 (41 s), followed by A3B2 (48 s) and A1B2 (68 s). Overall, the results indicate that pine resin addition consistently reduced ignition time across all three starch concentrations, although the magnitude of the reduction differed among formulations. The reduction was particularly pronounced at the 12% starch level, where ignition time decreased from 95 s in A2B1 to 41 s in A2B2, corresponding to a reduction of approximately 56.8%.

Figure 6 shows that the higher heating value (HHV) ranged from 6.620 to 7.080 kcal kg⁻¹ across the six treatments. The highest HHV was obtained in treatment A2B2 (7.080 kcal kg⁻¹), followed by A1B1 (6.980 kcal kg⁻¹) and A1B2 (6.940 kcal kg⁻¹). Treatment A2B1 recorded an HHV of 6.910 kcal kg⁻¹, whereas increasing the cassava waste starch concentration to 16%

resulted in lower HHV values of 6.730 kcal kg⁻¹ in A3B1 and 6.620 kcal kg⁻¹ in A3B2, with the latter representing the lowest value among the treatments. Among the evaluated formulations, the combination of 12% cassava waste starch and 5% pine resin (A2B2) exhibited the most favorable combustion performance by combining the highest HHV (7.080 kcal kg⁻¹) with the shortest ignition time (41 s).

The combustion performance of *Pangium edule* shell charcoal briquettes produced with different concentrations of cassava waste starch and pine resin is presented in Table 3. Ignition time ranged from 41 ± 2.40 to 128 ± 4.20 s, whereas higher heating value (HHV) ranged from 6.620 ± 43 to 7.080 ± 35 kcal kg⁻¹. Two-way ANOVA was used to evaluate the effects of cassava waste starch concentration, pine resin addition, and their interaction on these combustion properties, while Tukey's HSD test was applied to identify differences among treatment means. The shortest ignition time and highest HHV were both recorded for A2B2, containing 12% cassava waste starch and 5% pine resin.

Ignition time decreased markedly with pine resin addition. At 8% starch, the addition of 5% pine resin reduced ignition time from 128 ± 4.20 s in A1B1 to 68 ± 3.10 s in A1B2. A similar response occurred at 12% starch, where ignition time decreased from 95 ± 3.80 s in A2B1 to 41 ± 2.40 s in A2B2, and at 16% starch, where it decreased from 82 ± 3.50 s in A3B1 to 48 ± 2.70 s in A3B2. The shortest ignition time was therefore obtained in A2B2. According to Tukey's HSD test, A2B2 belonged to a distinct statistical group (a), followed by A3B2 (b), A1B2 (c),

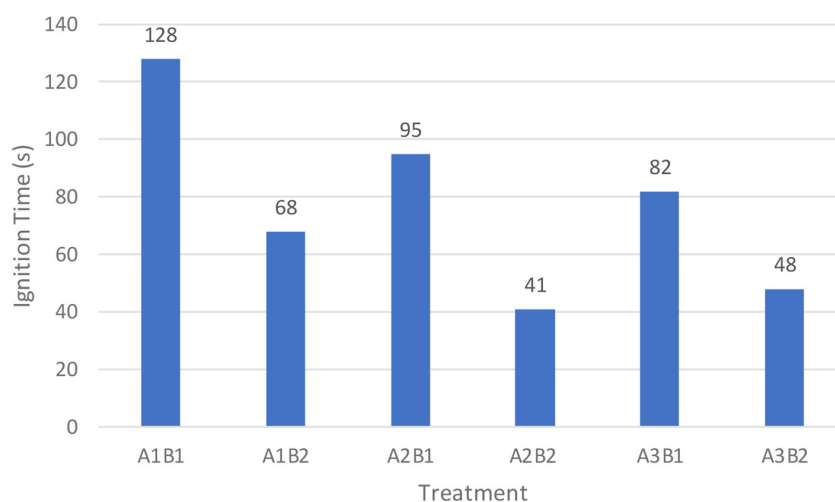


Figure 5. Ignition time of *Pangium edule* shell charcoal briquettes

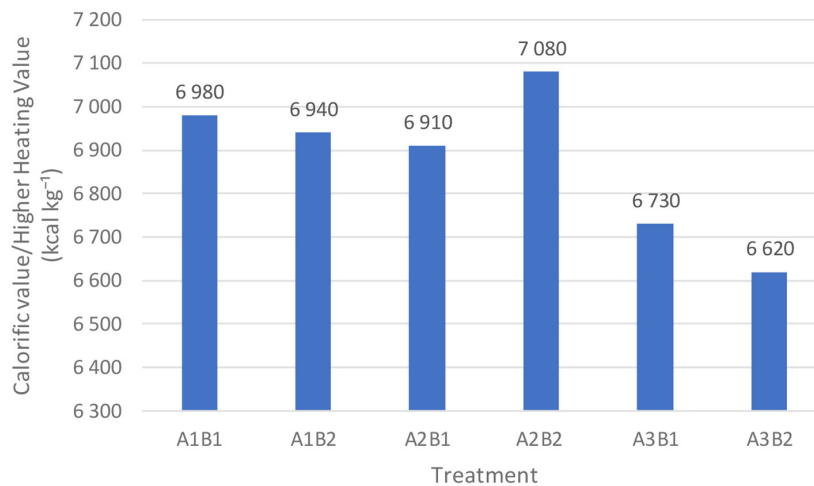


Figure 6. Calorific value/higher heating value of *Pangium edule* shell charcoal briquettes

Table 3. Effect of cassava waste starch concentration and pine resin addition on the combustion performance of *Pangium edule* shell charcoal briquettes

Treatment	Ignition time (s)	Calorific value/Higher heating value (kcal kg ⁻¹)
A1B1 (8% starch + 0% resin)	128 ± 4.20 ^e	6.980 ± 42 ^c
A1B2 (8% starch + 5% resin)	68 ± 3.10 ^c	6.940 ± 38 ^c
A2B1 (12% starch + 0% resin)	95 ± 3.80 ^d	6.910 ± 45 ^b
A2B2 (12% starch + 5% resin)	41 ± 2.40 ^a	7.080 ± 35 ^d
A3B1 (16% starch + 0% resin)	82 ± 3.50 ^d	6.730 ± 40 ^b
A3B2 (16% starch + 5% resin)	48 ± 2.70 ^b	6.620 ± 43 ^a

Note: Values are presented as mean ± standard deviation (SD; n = 3). Different superscript letters within the same column indicate significant differences among treatment means based on Tukey's HSD post-hoc test at $p < 0.05$. Two-way ANOVA was performed to evaluate the effects of cassava waste starch concentration (A), pine resin addition (B), and their interaction ($A \times B$). For ignition time, starch concentration significantly affected the response ($F_{2,12} = 178.952$, $p < 0.001$), as did pine resin addition ($F_{1,12} = 980.923$, $p < 0.001$), while the $A \times B$ interaction was also significant ($F_{2,12} = 24.899$, $p < 0.001$). For HHV, starch concentration significantly affected the response ($F_{2,12} = 111.951$, $p < 0.001$), and the $A \times B$ interaction ($F_{2,12} = 19.289$, $p < 0.001$), whereas pine resin addition alone had no significant effect ($F_{1,12} = 0.121$, $p = 0.734$).

and A2B1 and A3B1 (d), while A1B1 formed the highest ignition-time group (e). Thus, A2B1 and A3B1 were statistically comparable, whereas all other treatment groups differed significantly from one another. These results indicate that the intermediate starch concentration combined with pine resin provided particularly favorable ignition characteristics.

The rapid ignition associated with pine resin is consistent with the presence of volatile organic constituents that can be released during initial heating and contribute to flame establishment. However, because the present study did not directly measure volatile release or combustion kinetics, this explanation should be regarded as a plausible interpretation of the observed response rather than direct experimental confirmation of the underlying

mechanism. The significant main effects of starch concentration and pine resin addition, together with the significant $A \times B$ interaction, further demonstrate that ignition behavior was influenced not only by the individual binder components but also by their combined formulation.

HHV varied from 6.620 ± 43 to 7.080 ± 35 kcal kg⁻¹, with A2B2 exhibiting the highest value and A3B2 the lowest. The relatively high HHV of A2B2 is consistent with its relatively high fixed carbon content (72.97%) and moderate total binder incorporation. Although A1B1 had slightly higher fixed carbon (73.86%), its HHV was lower than that of A2B2. This finding indicates that HHV was not determined solely by fixed carbon content but was also influenced by the overall

composition of the briquette and the characteristics of the incorporated binder materials.

Tukey's HSD results showed that A3B2 had the lowest HHV and formed a distinct statistical group (a). A2B1 and A3B1 belonged to the same statistical group (b), indicating no significant difference between their HHV values, while A1B1 and A1B2 shared group c and were likewise statistically comparable. A2B2 was assigned group d, indicating that its HHV was significantly higher than that of all other treatments. Thus, the highest HHV observed for A2B2 was supported by the post-hoc analysis rather than representing only a numerical difference.

The decrease in HHV at the highest starch concentration was particularly evident in A3B1 and A3B2. The lower HHV of these treatments coincided with their lower fixed carbon contents (63.35% and 61.00%, respectively), suggesting that excessive incorporation of binder material reduced the relative contribution of the carbon-rich charcoal fraction to the energy content. Thus, increasing binder concentration beyond the intermediate level did not provide an equivalent improvement in energy performance.

Interestingly, pine resin addition alone did not significantly affect HHV ($F_{1,12} = 0.121$, $p = 0.734$), whereas the significant starch $\times$ resin interaction ($F_{2,12} = 19.289$, $p < 0.001$) indicates that the effect of resin depended on the cassava starch concentration. This result is important because the numerical differences between resin-containing and resin-free treatments should not be interpreted as a uniform main effect of pine resin across all starch concentrations. Instead, the HHV response was dependent on the specific combination of starch and resin levels.

The contrasting responses of ignition time and HHV demonstrate that briquette combustion performance cannot be adequately characterized using a single fuel-quality parameter. Moisture, volatile matter, fixed carbon, density, mechanical strength, ignition behavior, and HHV collectively determine the functional performance of a solid biofuel. In this study, A2B2 combined moderate moisture ($5.35 \pm 0.13\%$), relatively high fixed carbon ($72.97 \pm 0.38\%$), the highest numerical density ($0.66 \pm 0.012 \text{ g cm}^{-3}$), the highest numerical compressive strength ($7.02 \pm 0.28 \text{ kg cm}^{-2}$), the shortest ignition time ($41 \pm 2.40 \text{ s}$), and the highest HHV ($7,080 \pm 35 \text{ kcal kg}^{-1}$).

These results indicate that 12% cassava waste starch combined with 5% pine resin provided the

most balanced performance across the measured fuel and physical properties. The formulation achieved rapid ignition without the substantial reduction in energy content observed at the highest starch concentration, while also maintaining high mechanical resistance. Importantly, A2B2 was statistically superior in HHV and ignition time based on the Tukey groupings, while its density and compressive strength were numerically highest but statistically comparable with selected high-binder treatments. Accordingly, A2B2 can be regarded as the most favorable formulation among those evaluated for further development as a solid biofuel. Nevertheless, its suitability for practical or industrial application requires further validation through combustion-emission testing, durability assessment, storage studies, and scale-up experiments. These additional assessments are particularly important because the present evaluation was conducted under controlled laboratory conditions and did not directly quantify combustion emissions, long-term durability, or operational performance at larger scales.

Overall briquette performance and optimum formulation

The performance of charcoal briquettes is governed by multiple interdependent physico-chemical, mechanical, and combustion properties; therefore, the selection of a suitable formulation should consider the balance among fuel quality, structural integrity, and combustion behavior rather than maximizing a single response. In the present study, varying cassava waste starch and pine resin concentrations produced distinct responses in proximate composition, density, compressive strength, ignition time, and HHV. These responses indicate that binder optimization involves inherent trade-offs between improved particle consolidation and the preservation of the carbon-rich fraction of the charcoal matrix.

One important trade-off involved moisture content and density. Increasing binder concentration generally increased moisture content while also promoting densification. The increase in moisture is consistent with the hydrophilic characteristics of starch, whereas the increase in density suggests improved particle consolidation during briquetting. Although higher density can improve structural integrity and volumetric energy storage, excessive moisture may reduce effective energy availability

because part of the combustion energy is consumed in water evaporation. A2B2 provided a favorable balance, with moderate moisture content ($5.35 \pm 0.13\%$) and the highest numerical density ($0.66 \pm 0.012 \text{ g cm}^{-3}$). However, its density was statistically comparable with A3B1 and A3B2 according to Tukey's HSD test. This combination suggests that the 12% starch + 5% resin formulation achieved effective densification without excessive moisture retention under the experimental conditions.

A second trade-off occurred between fixed carbon and volatile matter, which are important determinants of ignition and sustained combustion. Increasing binder concentration was associated with higher volatile matter and lower fixed carbon. The increase in volatile matter can facilitate ignition by providing combustible volatiles during the initial heating stage, whereas fixed carbon contributes more substantially to sustained heat release during subsequent combustion. A2B2 contained $72.97 \pm 0.38\%$ fixed carbon and $23.50 \pm 0.34\%$ volatile matter, representing a comparatively balanced composition among the tested treatments. Importantly, its fixed carbon content was statistically comparable with A1B1 ($73.86 \pm 0.42\%$), while its volatile matter content was statistically comparable with A1B2 ($22.80 \pm 0.27\%$) and A2B1 ($23.50 \pm 0.34\%$) according to the respective Tukey groupings. In contrast, the higher binder treatments A3B1 and A3B2 exhibited substantially lower fixed carbon contents ($63.35 \pm 0.57\%$ and $61.00 \pm 0.46\%$, respectively) accompanied by higher volatile matter contents ($27.60 \pm 0.48\%$ and $29.15 \pm 0.39\%$, respectively). This pattern indicates that excessive binder incorporation can alter the carbon-to-binder ratio in a manner that compromises energy-related properties.

The observed responses are consistent with complementary contributions from cassava waste starch and pine resin, although the underlying mechanisms were not directly confirmed by instrumental characterization in this study. Gelatinized starch can promote particle cohesion and improve the continuity of the briquette matrix, whereas pine resin can contribute additional adhesion and combustible volatile constituents. The significant effects of binder formulation on compressive strength and ignition time are consistent with these proposed functions. However, the ANOVA results showed that pine resin addition alone did not significantly affect HHV

($F_{1,12} = 0.121$, $p = 0.734$), whereas its interaction with starch concentration was significant ($F_{2,12} = 19.289$, $p < 0.001$). Therefore, the contribution of pine resin to combustion performance should not be interpreted as a uniform main effect across all starch concentrations. Because SEM, FTIR, TGA, and XRD analyses were not conducted, these mechanisms should be interpreted as plausible explanations based on the measured properties rather than as experimentally verified mechanisms. The results nevertheless indicate that binder performance depends not only on adhesive capacity but also on the proportion of binder relative to the carbonaceous feedstock.

Considering the measured properties simultaneously, A2B2 (12% cassava waste starch + 5% pine resin) exhibited the most favorable performance profile among the formulations investigated. It combined relatively high fixed carbon ($72.97 \pm 0.38\%$), low ash content ($2.18 \pm 0.07\%$), moderate moisture ($5.35 \pm 0.13\%$), controlled volatile matter ($23.50 \pm 0.34\%$), the highest numerical density ($0.66 \pm 0.012 \text{ g cm}^{-3}$), the highest numerical compressive strength ($7.02 \pm 0.28 \text{ kg cm}^{-2}$), the shortest ignition time ($41 \pm 2.40 \text{ s}$), and the highest HHV ($7,080 \pm 35 \text{ kcal kg}^{-1}$). This combination indicates a favorable compromise between structural durability, ignition behavior, and energy content. Notably, A2B2 was statistically superior to the other treatments in terms of ignition time and HHV based on Tukey's HSD test, whereas its density was statistically comparable with A3B1 and A3B2, and its compressive strength was statistically comparable with A3B1 and A3B2. Although A1B1 had slightly lower moisture ($3.45 \pm 0.09\%$) and higher fixed carbon ($73.86 \pm 0.42\%$), its substantially lower density ($0.60 \pm 0.008 \text{ g cm}^{-3}$), compressive strength ($3.45 \pm 0.17 \text{ kg cm}^{-2}$), and longer ignition time ($128 \pm 4.20 \text{ s}$) limited its overall performance. Conversely, A3B1 and A3B2 did not provide additional improvements in physical performance sufficient to compensate for their lower fixed carbon ($63.35 \pm 0.57\%$ and $61.00 \pm 0.46\%$) and lower HHV ($6,730 \pm 40$ and $6,620 \pm 43 \text{ kcal kg}^{-1}$, respectively).

The identification of A2B2 as the most favorable formulation should therefore be understood as a comparative outcome within the experimental formulations evaluated, rather than as a universal optimum. This distinction is important because A2B2 was not statistically superior to every formulation for every individual response. Its selection resulted from the simultaneous

consideration of proximate composition, physical properties, ignition behavior, and energy content. The result is particularly relevant because it demonstrates that increasing binder concentration beyond the intermediate level does not necessarily improve briquette quality. Instead, an intermediate binder ratio can provide a more favorable balance between particle cohesion and preservation of the carbonaceous fraction. This multi-response perspective provides stronger support for formulation selection than reliance on density, compressive strength, fixed carbon, ignition time, or HHV individually.

From an application perspective, the A2B2 formulation demonstrates promising potential for the development of renewable solid biofuel from *P. edule* shell waste. The formulation simultaneously utilizes *P. edule* shell charcoal, cassava-processing residue, and pine resin, thereby providing an opportunity to valorize biomass resources that might otherwise have limited economic value. The combination of a relatively high density, high compressive strength, rapid ignition, and high HHV may support applications such as household cooking and small-scale thermal energy generation. However, the transition from laboratory-scale performance to practical or industrial application requires further validation, particularly through long-term storage and durability testing, combustion-emission assessment, ash characterization, life-cycle assessment, techno-economic analysis, and pilot-scale production trials.

Taken together, the findings show that binder formulation is a critical engineering variable in determining the balance between briquette durability and fuel performance. Within the formulation range investigated, A2B2 (12% cassava waste starch + 5% pine resin) provided the most favorable integrated combination of physicochemical, mechanical, and combustion characteristics, particularly by achieving the lowest ignition time (41 ± 2.40 s) and highest HHV (7.080 ± 35 kcal kg⁻¹) while maintaining high fixed carbon ($72.97 \pm 0.38\%$) and favorable physical properties. This result strengthens the scientific contribution of the study by demonstrating that the combined use of cassava waste starch and pine resin can be evaluated as a multi-response binder system for valorizing *P. edule* shell biomass, while also recognizing the need for further mechanistic and scale-up investigations before broader industrial conclusions can be drawn.

DISCUSSION

Influence of bio-based binders on briquette quality

The overall quality of biomass briquettes is strongly influenced by the physicochemical characteristics and interactions of the binding agents used during densification. In the present study, cassava waste starch and pine resin jointly influenced the proximate composition, mechanical properties, and combustion performance of *Pan-gium edule* shell charcoal briquettes. Although the analytical methods and individual response variables used here are established in biomass briquette research, the present study provides new experimental evidence on the performance of an integrated dual-binder system combining cassava waste starch and pine resin with *Pan-gium edule* shell charcoal. This combination differs from conventional single-binder approaches by simultaneously exploiting the complementary functional characteristics of a starch-based binder and a resin-based binder. Unlike petroleum-derived synthetic binders, both materials are renewable, biodegradable, and readily available from agricultural and forestry resources, making them attractive alternatives for sustainable briquette production. The selection of appropriate natural binders is particularly important because binder composition directly affects particle cohesion, briquette durability, combustion behavior, and overall fuel quality (Olugbade et al., 2019).

Cassava waste starch primarily functions through gelatinization. During heating in the presence of water, starch granules absorb moisture, swell, rupture, and form a viscous polymeric gel that fills interparticle voids and binds adjacent charcoal particles through extensive hydrogen bonding. This gelatinized network enhances particle cohesion, reduces internal porosity, and improves stress transfer throughout the briquette matrix, thereby increasing density and compressive strength. In the present study, this interpretation is supported by the observed changes in density and compressive strength across the starch treatments, although direct microstructural confirmation was not conducted. Similar mechanisms have been reported for cassava-based binders used in coconut shell charcoal briquettes, where starch significantly improved structural integrity while maintaining acceptable combustion properties (Rudiyanto et al., 2023). Comparable observations have also

been reported for biomass densification systems, in which starch-based binders improve particle packing efficiency and mechanical stability by increasing the contact area between biomass particles (Tumuluru et al., 2011).

In contrast, pine resin contributes through a different mechanism. Resin is composed primarily of diterpene resin acids and volatile terpene compounds that soften during compaction and solidify upon cooling, thereby producing thermoplastic adhesion between adjacent charcoal particles. This proposed mechanism is interpreted cautiously because no SEM, FTIR, TGA, or XRD analyses were performed in the present study. Nevertheless, the observed reduction in ignition time and the variation in calorific value following resin addition are consistent with the expected contribution of resin-derived combustible constituents. This adhesive behavior may increase resistance to fracture while simultaneously supplying combustible volatile compounds that facilitate flame initiation during ignition. Consequently, pine resin may function as both a natural adhesive and an ignition promoter, distinguishing it from conventional starch binders. Similar improvements in ignition characteristics and briquette integrity following pine resin addition have been demonstrated by Pari et al. (2023), who reported enhanced initial ignition and calorific performance in resin-modified charcoal briquettes.

The superior performance of treatment A2B2 indicates that the combination of cassava waste starch and pine resin was associated with a complementary, rather than simply additive, response across several of the measured briquette properties. Gelatinized starch established the structural framework necessary for densification, whereas pine resin may have provided additional interparticle adhesion and contributed combustible volatile constituents that favored ignition characteristics. This complementary binder combination was associated with improvements in both mechanical and combustion-related properties, demonstrating that combining two natural binders can provide a favorable overall balance of briquette quality compared with the use of a single binder. Importantly, the contribution of the present study is not merely the identification of a practically suitable briquette formulation, but the experimental demonstration that binder composition governs trade-offs among structural and fuel-quality properties. The 12% cassava waste starch + 5% pine resin treatment (A2B2) provided

the most balanced multi-response performance among the tested formulations, rather than representing an optimum based on a single response variable. Similar synergistic effects of composite bio-based binders have recently been reported by Madhusanka et al. (2025), who concluded that mixed natural binders produced a better balance between structural integrity and fuel performance than individual binder systems. Furthermore, reviews on biomass densification emphasize that optimum binder formulation should maximize interparticle bonding while minimizing adverse effects on energy content and combustion efficiency (Sukarta et al., 2023).

These findings demonstrate that optimizing binder composition is fundamental because the binder system influences virtually every briquette property, including moisture retention, volatile matter, density, mechanical durability, ignition behavior, and calorific value. The present findings therefore extend previous single-binder and conventional-feedstock studies by providing experimental evidence for the relationship between dual-binder composition and multiple physicochemical, mechanical, and fuel responses in *Pangium edule* shell charcoal briquettes. Rather than acting solely as a physical adhesive, the combined cassava waste starch–pine resin system may influence both the internal microstructure and thermal decomposition behavior of the briquette. However, these mechanistic interpretations should be regarded as literature-supported explanations rather than direct instrumental evidence and should be further verified through SEM, FTIR, TGA, and XRD analyses in future studies. Consequently, binder selection should be regarded as a key engineering parameter in biomass briquette production, particularly when the objective is to achieve a balanced combination of fuel quality, structural integrity, and combustion efficiency suitable for sustainable bioenergy applications (Rudiyanto et al., 2023).

Trade-off between proximate composition and physical properties

An important outcome of this study is the identification of several trade-offs among briquette quality parameters. Improving one characteristic frequently influenced another, indicating that optimum briquette quality cannot be achieved by maximizing individual properties independently. Instead, overall performance depends on balancing chemical composition, structural integrity,

and combustion behavior. This multi-response optimization concept has become increasingly important in biomass densification research because the quality of solid biofuels is determined by the interaction among physical, chemical, and thermal properties rather than by a single performance indicator (Tumuluru et al., 2011).

One clear relationship was observed between moisture content and density. Increasing binder concentration generally enhanced particle consolidation and produced denser briquettes but simultaneously increased moisture retention because starch contains abundant hydroxyl groups capable of absorbing and retaining water. Although greater density improves handling durability, storage stability, and volumetric energy density, excessive moisture decreases thermal efficiency because additional energy is required to evaporate water before combustion can proceed efficiently. Similar relationships between binder concentration, moisture retention, and briquette densification have been reported for carbonized corncob and coconut shell briquettes prepared using cassava starch-based binders (Aransiola et al., 2019).

A second trade-off involved volatile matter and ignition behavior. Higher volatile matter promotes the early release of combustible gases during devolatilization, thereby reducing ignition time and facilitating flame propagation. However, excessive volatile matter may increase the rate of fuel consumption and shorten combustion duration. Conversely, briquettes with lower volatile matter generally require longer ignition periods despite exhibiting more stable char combustion. Therefore, an appropriate volatile matter content is essential to achieve a balance between ignition efficiency and sustained heat release (Nhuchhen and Salam, 2012).

The relationship between fixed carbon and heating value also deserves particular attention. Fixed carbon constitutes the primary energy-bearing fraction responsible for prolonged oxidation during the char combustion stage. Increasing binder content can replace part of the carbon-rich charcoal matrix with organic polymers, thereby decreasing the relative proportion of fixed carbon available for combustion. Because higher heating value is closely associated with combustible carbon content, excessive binder incorporation may reduce the energy density of the briquette despite improving its mechanical integrity (Baharuddin and Daud, 2026). Previous studies have similarly demonstrated that fixed carbon is one of

the strongest predictors of higher heating value in lignocellulosic biomass fuels (Nhuchhen and Salam, 2012; Daud et al., 2012).

Density also exhibited a close relationship with compressive strength because compact particle packing increases the contact area available for interparticle bonding and improves stress distribution throughout the briquette matrix. Nevertheless, excessive densification may potentially reduce internal pore connectivity and restrict oxygen diffusion during combustion, illustrating another engineering trade-off that should be considered during briquette design. Consequently, the optimum briquette should possess sufficient density to ensure mechanical durability while maintaining adequate porosity for efficient combustion. Similar conclusions have been reported in studies on biomass densification, where optimum briquette performance was consistently achieved at intermediate binder concentrations rather than at the maximum binder level (Aransiola et al., 2019).

These interactions help explain why treatment A2B2 exhibited the most favorable overall performance among the tested formulations. Although treatment A1B1 contained lower moisture and higher fixed carbon, its relatively lower density and compressive strength and longer ignition time reduced its practical applicability. Conversely, treatments containing the highest binder concentration (A3B1 and A3B2) achieved relatively high mechanical performance but experienced reductions in fixed carbon and higher heating value due to the greater contribution of binder material to the briquette matrix. Therefore, the preferred formulation should be selected based on a balanced assessment of overall fuel performance rather than on the maximization of individual quality parameters (Tumuluru et al., 2011).

Combustion mechanism of cassava starch-pine resin briquettes

The combustion behavior of biomass charcoal briquettes proceeds through a series of overlapping stages, including moisture evaporation, devolatilization, volatile combustion, char oxidation, and burnout. The duration and intensity of each stage are strongly influenced by the physicochemical characteristics of the briquette, particularly its proximate composition, internal pore structure, and binder composition. Consequently, natural binders not only improve mechanical integrity but also may modify thermal

decomposition pathways and combustion behavior (Nhuchhen and Salam, 2012).

During the initial heating stage, free and bound moisture are removed from the briquette matrix. Moisture content plays a critical role because a portion of the supplied thermal energy must first be consumed for water evaporation before pyrolysis reactions can proceed. Briquettes with lower moisture content therefore generally tend to ignite more readily and exhibit higher thermal efficiency, whereas excessive moisture delays temperature elevation and prolongs ignition time (Nhuchhen and Salam, 2012). In the present study, the moderate moisture content observed in treatment A2B2 ($5.35 \pm 0.13\%$) coincided with the shortest ignition time (41 ± 2.40 s), while treatment A1B1, which had the lowest moisture content ($3.45 \pm 0.09\%$), exhibited the longest ignition time (128 ± 4.20 s). This indicates that ignition behavior was not determined by moisture content alone but was also influenced by binder composition and other fuel properties.

Following moisture removal, devolatilization becomes the dominant process. During this stage, cassava starch and pine resin undergo thermal degradation, releasing combustible volatile compounds that initiate flame formation. Pine resin is particularly rich in diterpene resin acids and terpene hydrocarbons, which can contribute to the formation of combustible volatiles during heating. The evolution of these combustible gases may accelerate ignition and promote flame establishment. Similar observations were reported by Pari et al. (2023), who demonstrated that pine resin addition significantly reduced ignition time while improving the calorific performance of charcoal briquettes through the contribution of terpene-derived volatiles. These findings provide a plausible explanation for the improved ignition characteristics observed in briquettes containing pine resin compared with formulations prepared using starch as the sole binder.

As volatile compounds are progressively consumed, combustion transitions to the oxidation of fixed carbon, which represents the principal heat-generating stage of charcoal combustion. Fixed carbon is oxidized more slowly than volatile compounds and therefore contributes to combustion duration and sustained heat release. Numerous studies have demonstrated a positive relationship between fixed carbon content and higher heating value because carbon oxidation

provides a substantial proportion of the thermal energy released during char combustion (Yin et al., 2008). Consequently, maintaining an adequate fixed carbon fraction while preserving sufficient volatile matter is essential for simultaneously achieving rapid ignition and prolonged combustion stability.

Binder composition also affects combustion through its potential influence on the briquette microstructure. Gelatinized cassava starch may improve particle packing by filling interparticle voids, whereas thermoplastic pine resin may reinforce particle adhesion and reduce fragmentation during burning. Improved particle cohesion could promote a more uniform combustion front and minimize premature structural failure of the briquette. However, excessive binder addition may reduce pore connectivity, thereby restricting oxygen diffusion into the interior of the briquette and slowing oxidation reactions. Similar interactions between briquette density, porosity, oxygen transport, and combustion efficiency have been reported for densified biomass fuels, where an optimum balance between mechanical strength and internal permeability is required to maximize combustion performance (Gilvari et al., 2019).

The superior combustion performance of treatment A2B2 demonstrates that optimizing binder composition influences combustion through multiple complementary mechanisms rather than through a single property. The moderate proportion of cassava waste starch (12%) and pine resin (5%) was associated with sufficient volatile matter for rapid ignition, a relatively high fixed carbon content ($72.97 \pm 0.38\%$) for sustained heat release, and favorable physical properties for maintaining briquette integrity. This balanced combination resulted in the shortest ignition time (41 ± 2.40 s) and the highest higher heating value (7.080 ± 35 kcal kg⁻¹) among the evaluated formulations. However, because the present study did not directly characterize pore structure, volatile release, or combustion kinetics, the proposed mechanisms should be regarded as plausible interpretations supported by the observed fuel properties and previous literature rather than as directly verified mechanisms. Such a systems-based interpretation is increasingly emphasized in recent biomass combustion research because it provides a more comprehensive framework for optimizing renewable solid fuels (Pio et al., 2026).

Optimization of overall briquette performance

Selection of the optimum briquette formulation should not rely on a single quality parameter because individual fuel properties frequently exhibit opposing trends. Instead, briquette optimization requires simultaneous consideration of proximate composition, mechanical durability, combustion behavior, and practical applicability. This multi-response optimization approach recognizes that improvements in one characteristic are often accompanied by compromises in another, making overall fuel performance a more meaningful criterion than isolated quality indicators (Ali et al., 2024). In the present study, this approach is particularly relevant because the dual-binder system combines cassava waste starch and pine resin, which exert complementary effects on structural integrity and combustion-related properties. Thus, the optimum formulation was evaluated based on the overall balance among multiple measured responses rather than on a single indicator of briquette quality.

Although treatment A1B1 exhibited the lowest moisture content and the highest fixed carbon content, these favorable chemical characteristics did not translate into superior overall briquette performance. The relatively low density and compressive strength observed in A1B1 indicate insufficient interparticle bonding, which may reduce resistance to breakage during handling, transportation, and storage. In addition, the relatively longer ignition time suggests that the absence of pine resin limited the availability of volatile combustible compounds required for rapid flame establishment. This contrast provides experimental evidence that favorable proximate composition alone is insufficient to define an optimal briquette formulation and that structural and ignition characteristics must be considered simultaneously. These findings demonstrate that excellent proximate composition alone cannot ensure satisfactory engineering performance because fuel utilization depends equally on structural integrity and combustion behavior (Aransiola et al., 2019).

Conversely, formulations containing the highest binder concentration (A3B1 and A3B2) produced stronger particle cohesion through increased starch gelatinization and resin-assisted adhesion. However, excessive incorporation of binder partially replaced the carbon-rich charcoal matrix with organic polymers, leading to higher

moisture and volatile matter contents, reduced fixed carbon, and ultimately lower higher heating values. This phenomenon illustrates the law of diminishing returns commonly observed in biomass densification systems, whereby increasing binder concentration beyond an optimum threshold yields only marginal improvements in mechanical properties while simultaneously reducing fuel quality (Nhuchhen and Salam, 2012). Therefore, the results indicate that increasing binder concentration does not necessarily produce proportional improvements in overall fuel performance, highlighting the importance of identifying an appropriate binder balance.

Treatment A2B2 achieved the most balanced combination of physicochemical and combustion properties because the moderate proportion of cassava waste starch (12%) and pine resin (5%) provided sufficient interparticle bonding without substantially reducing the combustible carbon fraction. The optimized binder ratio produced relatively high fixed carbon (72.97%), low ash content (2.18%), moderate moisture content (5.35%), the highest density (0.66 g cm^{-3}), the highest compressive strength (7.02 kg cm^{-2}), the shortest ignition time (41 s), and the highest higher heating value ($7.080 \text{ kcal kg}^{-1}$) among the evaluated formulations. However, Tukey's HSD test indicated that the density of A2B2 was statistically comparable to A3B1 and A3B2, while its compressive strength was statistically comparable to A3B2. Therefore, A2B2 should be regarded as the formulation exhibiting the most favorable overall numerical performance profile rather than being statistically superior to all other treatments for every individual property.

Importantly, A2B2 represents a multi-response optimum within the experimental range rather than an optimum based on a single response variable. Its superior overall performance provides new experimental evidence that an intermediate combination of starch and resin can balance structural and fuel-quality requirements in *Pangium edule* shell charcoal briquettes. These results indicate that the complementary contributions of the two renewable binders were associated with a favorable compromise between structural durability and energy performance. This finding extends conventional single-binder approaches by demonstrating the potential value of a dual-binder formulation for simultaneously addressing mechanical and combustion-related requirements. Similar optimization strategies

have been reported in biomass briquette research, where intermediate binder concentrations consistently provide better overall fuel quality than either insufficient or excessive binder addition (Rudiyanto et al., 2023).

From an engineering perspective, the superiority of A2B2 highlights the importance of adopting a multi-criteria evaluation framework for biomass briquette development. Practical briquette performance is determined not only by laboratory measurements of individual parameters but also by the combined effects of mechanical stability, energy density, ignition behavior, and combustion efficiency during real-world utilization. Consequently, selecting formulations based on a balanced combination of these characteristics provides a more reliable basis for product development than relying on a single indicator such as fixed carbon or calorific value alone. Scientifically, the present findings demonstrate that binder composition is an important determinant of the trade-offs among physicochemical, mechanical, and combustion properties in an underutilized *Pangium edule* shell charcoal feedstock. This systems-oriented optimization strategy is increasingly recommended in recent biomass fuel studies because it better reflects the complex interactions governing solid biofuel performance and facilitates the development of commercially viable renewable energy products (Tumuluru et al., 2011). Nevertheless, the identification of A2B2 as the most favorable formulation is limited to the laboratory-scale conditions and formulation range evaluated in the present study. Additional investigations involving combustion emissions, storage stability, durability during transportation, life-cycle assessment, techno-economic evaluation, and pilot-scale production are required before broader industrial implementation can be recommended.

Comparison with previous studies and engineering implications

The present findings are generally consistent with previous studies demonstrating that natural binders improve briquette quality while reducing dependence on petroleum-derived synthetic adhesives. Rudiyanto et al., (2023) reported that cassava-derived binders enhanced the compressive strength and structural integrity of coconut shell charcoal briquettes through improved particle adhesion. Likewise, Pari et al., (2023), demonstrated

that pine resin accelerated ignition and increased the calorific performance of biomass briquettes by supplying volatile terpene compounds during thermal decomposition. More broadly, reviews on biomass densification have consistently emphasized that natural binders improve particle cohesion and mechanical durability while maintaining acceptable fuel quality when applied at appropriate concentrations (Sanchez-Roque et al., 2025). These previous studies provide an important basis for understanding the individual functions of starch- and resin-based binders; however, they do not directly address their combined application to *Pangium edule* shell charcoal within a single factorial experimental system.

Compared with previous studies that generally evaluated either a single binder or a single performance aspect, the present study integrates an underutilized lignocellulosic biomass resource (*Pangium edule* shell charcoal) with two renewable bio-based binders and simultaneously evaluates their influence on proximate composition, physical properties, and combustion performance. The scientific contribution of the present study therefore lies not in the use of novel analytical methods, but in generating new experimental evidence on the performance of this specific biomass–dual-binder system and the relationship between binder composition and multiple briquette quality attributes. This integrated assessment enables the identification of a favorable formulation based on overall briquette performance rather than on isolated performance indicators. The results further demonstrate that combining cassava waste starch and pine resin produced complementary effects across the measured responses: starch was associated with improved densification and mechanical performance, whereas pine resin was associated with shorter ignition time and additional interparticle bonding. The observed performance of A2B2 (12% cassava waste starch + 5% pine resin) further indicates that an intermediate dual-binder formulation can provide a favorable balance between structural integrity and fuel performance. This finding extends previous single-binder approaches by demonstrating the potential of a complementary dual-binder strategy for an underutilized tropical biomass feedstock. Such complementary mechanisms have received limited attention in previous studies, particularly for underutilized tropical forest biomass, although the proposed mechanisms should be interpreted cautiously because direct instrumental confirmation

by SEM, FTIR, TGA, or XRD was not undertaken in the present study.

From an engineering perspective, the A2B2 formulation (12% cassava waste starch + 5% pine resin) showed a combination of properties that indicates potential suitability for household cooking, small-scale agro-industrial heating, institutional kitchens, and decentralized bioenergy systems. It exhibited a density of $0.66 \pm 0.012 \text{ g cm}^{-3}$, compressive strength of $7.02 \pm 0.28 \text{ kg cm}^{-2}$, ignition time of $41 \pm 2.40 \text{ s}$, and HHV of $7.080 \pm 35 \text{ kcal kg}^{-1}$. Its relatively high density and compressive strength may improve resistance to abrasion during packaging, storage, and transportation, while its favorable ignition characteristics and higher heating value may contribute to effective thermal utilization during end use. However, the physical superiority of A2B2 should be interpreted in light of the Tukey HSD results: its density was statistically comparable with A3B1 and A3B2, while its compressive strength was statistically comparable with A3B2. Similar engineering considerations have been identified as critical quality attributes for commercial biomass briquettes because mechanical durability directly affects logistics, whereas combustion properties determine thermal performance and user acceptance. However, these potential applications should not be interpreted as evidence of commercial-scale readiness, because the present study was conducted at laboratory scale and did not evaluate long-term durability, continuous combustion performance, emissions, production economics, or scale-up behavior.

Beyond its technical performance, the present study also contributes to sustainable biomass utilization by simultaneously valorizing three renewable resources: *P. edule* shell waste, cassava processing residues, and pine resin. The replacement of petroleum-derived synthetic adhesives with renewable bio-based binders supports circular bioeconomy principles by extending the value chain of agricultural and forestry by-products while reducing waste generation. Furthermore, converting locally available biomass residues into high-value solid biofuels contributes to renewable energy diversification, although the magnitude of the associated environmental and socioeconomic benefits cannot be quantified from the present experimental data alone. The approach is nevertheless relevant to rural energy diversification and aligns conceptually with several sustainable development goals (SDGs), particularly

SDG 7 (Affordable and Clean Energy), SDG 12 (Responsible Consumption and Production), and SDG 13 (Climate Action). Similar sustainability benefits have been highlighted in studies examining biomass waste valorization for renewable energy production. A quantitative assessment of these environmental and socioeconomic benefits would require dedicated life-cycle and techno-economic analyses.

Although the present study provides a comprehensive evaluation of proximate composition, physical properties, and combustion performance, further investigations are necessary to strengthen the engineering applicability and mechanistic interpretation of the proposed briquette formulation. Future studies should include microstructural characterization using scanning electron microscopy (SEM) to investigate binder distribution and pore morphology, Fourier transform infrared spectroscopy (FTIR) to identify chemical interactions between charcoal and bio-based binders, thermogravimetric analysis (TGA) to evaluate combustion kinetics and thermal stability, and X-ray diffraction (XRD) to examine mineralogical changes during carbonization. In addition, combustion emission measurements (CO , CO_2 , NO_x , SO_2 , and particulate matter), life cycle assessment (LCA), and techno-economic analysis should be incorporated to comprehensively evaluate the environmental sustainability and commercial feasibility of large-scale production. Pilot-scale production and repeated independent trials should also be conducted to determine process robustness, product consistency, storage durability, and scalability under conditions closer to industrial operation. Such integrated assessments have become increasingly important in recent biomass energy research because they bridge laboratory-scale characterization with industrial implementation and sustainable bioenergy development.

Future research directions and broader implications

The findings of this study provide a basis for further investigation of dual bio-based binder systems for biomass briquette production. The identification of A2B2 as the most favorable formulation within the tested treatment range provides an initial reference point for subsequent optimization rather than a definitive formulation for large-scale application. Future studies should therefore build upon the present findings by expanding the

experimental range and incorporating additional response variables that are relevant to practical fuel utilization.

A particularly important direction is the development of a systematic multi-response optimization framework in which fuel quality, mechanical durability, ignition behavior, emissions, and processing efficiency are evaluated simultaneously. Such an approach would enable researchers to determine whether the performance balance observed for A2B2 can be maintained under different feedstock characteristics and processing conditions. Comparative studies involving other agricultural and forestry residues would also help establish the broader applicability of dual-binder systems.

Further research should also move from laboratory characterization toward application-oriented validation. Controlled combustion trials, long-term storage tests, transportation durability assessments, and pilot-scale production would provide evidence of performance under conditions closer to actual utilization. Particular attention should be given to combustion emissions and ash behavior because these parameters are important for determining the environmental and operational suitability of briquettes for household and small-scale thermal applications.

Mechanistic investigations using SEM, FTIR, TGA, and XRD would provide complementary evidence for understanding the structural, chemical, and thermal responses associated with starch-resin incorporation. These analyses could clarify how the two binders interact with the charcoal matrix and help distinguish physical adhesion from chemical or thermally induced interactions. Finally, future studies should integrate life-cycle assessment and techno-economic analysis with experimental performance data. Such integration would enable evaluation of whether the environmental benefits associated with valorizing *P. edule* shell waste, cassava residues, and pine resin remain favorable when energy consumption, binder preparation, drying, transportation, emissions, and production costs are considered. This progression from laboratory-scale characterization to mechanistic understanding, process optimization, and system-level assessment would strengthen the evidence required for evaluating the technical, environmental, and economic feasibility of *P. edule*-based briquettes at larger scales.

CONCLUSIONS

This study demonstrated that the formulation of cassava waste starch and pine resin substantially influenced the physicochemical, mechanical, and combustion performance of *Pangium edule* shell charcoal briquettes. Across the tested formulations, moisture content ranged from 3.45–7.15%, volatile matter from 20.67–29.15%, ash from 2.01–2.71%, and fixed carbon from 61.00–73.86%. Density varied from 0.60–0.66 g cm⁻³, while compressive strength ranged from 3.45–7.02 kg cm⁻². Ignition time varied from 41–128 s and HHV from 6.620–7.080 kcal kg⁻¹. These results confirm that binder formulation is an important determinant of briquette quality because changes in binder proportion simultaneously affect chemical composition, structural integrity, and combustion behavior. Among the six formulations evaluated, A2B2 (12% cassava waste starch + 5% pine resin) provided the most favorable performance profile. This formulation produced 5.35% moisture, 23.50% volatile matter, 2.18% ash, and 72.97% fixed carbon, together with the highest density (0.66 g cm⁻³), highest compressive strength (7.02 kg cm⁻²), shortest ignition time (41 s), and highest HHV (7.080 kcal kg⁻¹). However, the interpretation of these results should account for the statistical comparisons: A2B2 was not statistically different from A3B1 and A3B2 in density, and its compressive strength was statistically comparable with A3B2. The performance of A2B2 was therefore best described as the most favorable formulation based on the integrated response profile, rather than as a formulation that was statistically superior for every individual parameter. Accordingly, A2B2 should be regarded as the most favorable formulation within the experimental range investigated, rather than as a universally optimum formulation. The findings further indicate that increasing binder concentration beyond the intermediate level did not provide proportional improvements in briquette performance. The highest starch treatments, A3B1 and A3B2, exhibited lower fixed carbon contents of 63.35% and 61.00%, respectively, and lower HHVs of 6.730 and 6.620 kcal kg⁻¹, despite maintaining relatively high mechanical properties. This trade-off demonstrates that excessive binder incorporation can improve particle consolidation while simultaneously reducing the proportion of the carbon-rich fraction available for energy generation. The results therefore support a multi-response approach to binder selection rather than optimization

based on a single parameter such as fixed carbon, density, compressive strength, or HHV. From a resource-utilization perspective, the study demonstrates the potential to valorize *P. edule* shell waste together with cassava-processing residue and pine resin into a value-added solid biofuel. The use of renewable bio-based binders provides an alternative to petroleum-derived adhesives and offers a pathway for integrating agricultural and forestry residues within a circular bioeconomy framework. Nevertheless, the present study does not directly quantify the environmental or economic benefits of this approach. The present evidence is also limited to laboratory-scale characterization, and the proposed roles of starch gelatinization and pine-resin adhesion remain mechanistic interpretations rather than directly verified mechanisms. Future research should therefore combine microstructural and spectroscopic characterization (SEM, FTIR, TGA, and XRD) with combustion-emission measurements, durability and storage testing, life-cycle assessment, and techno-economic analysis. These investigations are needed to establish the underlying binder–charcoal interactions and determine whether the favorable performance observed for A2B2 can be maintained under pilot- and industrial-scale production conditions. Such validation would provide a stronger scientific and engineering basis for assessing the environmental sustainability, economic feasibility, and practical deployment potential of *P. edule*-based charcoal briquettes.

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