



## The Role of Clay Binder Proportion and Compression Intensity on Physical Attributes of Carbonized Rice Husk Briquette

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### ABSTRACT

This study evaluated the effects of clay binder ratio and compression level on the physical properties of carbonized rice husk briquettes for sustainable fuel optimization. Briquettes were produced using 0–20 % clay binder and compacted at 6, 12, and 18 mm in a factorial design. Bulk density, moisture content, volatile matter, fixed carbon, and ash content were measured, with both factors and their interaction significantly affecting all properties. Moisture content was lowest (4.02 %) at 5 % binder and 6 mm compaction, while volatile matter decreased from 24.48 % (0 % binder) to 13.41 % (20 % binder). Fixed carbon increased from 50.49 % to 68.27 % with increasing binder level, while ash content decreased from 20.21 % to 13.77 %. Higher binder ratios improved bulk density and durability, and increased compaction further enhanced density and strength across treatments. The optimum condition was achieved at 5–10 % clay binder and 12 mm compaction, providing a balance of fuel quality and storage stability. In summary, binder proportion and compression intensity are key determinants of carbonized rice husk briquette quality for sustainable rural energy applications.

### 1. Introduction

Rice husk is one of the most abundant agricultural residues globally, particularly in major rice-producing regions, with annual generation exceeding 150 million tons (Suryaningsih et al., 2018; Koskin et al., 2020; Kordi

et al., 2024). Despite its abundance, it is still largely treated as a low-value waste material, and its improper disposal through open burning contributes significantly to air pollution, environmental degradation, and public health risks (Elinge et al., 2019). However, its high cellulose, lignin, and silica content make it a promising

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feedstock for value-added applications, particularly in bioenergy and carbon-based materials (Saeed et al., 2021; Singh et al., 2024).

In developing countries such as Ethiopia, biomass accounts for approximately 87 % of household energy consumption, leading to severe environmental and health impacts, including deforestation and indoor air pollution-related mortality (Sanbata et al., 2014; Yongabi et al., 2016). In this context, converting rice husk into briquettes represents a critical pathway toward cleaner and more sustainable household energy. However, raw rice husk is inherently unsuitable for direct use due to its low bulk density, high ash content, and poor combustion characteristics, necessitating preprocessing such as carbonization to improve fuel quality (Kipngetch et al., 2023).

Although briquetting technology has been widely studied, existing research has predominantly focused on organic binders (e.g., starch, molasses) and isolated effects of processing parameters such as compaction or binder type (Ndindeng et al., 2015; Daniel et al., 2020). These approaches remain limited in two critical aspects: (i) they often neglect inorganic, low-cost binder alternatives, and (ii) they rarely investigate the interaction effects between binder proportion and compaction intensity, despite their known influence on briquette densification and combustion behavior.

In particular, the use of clay soil as a binder has received limited systematic investigation, even though it offers strong potential as a low-cost, widely available, non-food competing alternative capable of improving structural integrity (Rawat and Kumar, 2022). However, its combined effect with compaction intensity—and especially their interaction—remains poorly understood, with limited quantitative optimization reported in the literature. This represents a clear scientific and practical knowledge gap in biomass densification research.

To address this gap, the present study provides a systematic and statistically robust evaluation of the interactive effects of clay soil binder ratio and compaction level on the physical properties of carbonized rice husk briquettes. Unlike previous studies that typically consider these parameters in isolation, this work explicitly quantifies their interaction and identifies optimal operating conditions for balanced briquette performance. The key contribution of this study lies in: (i) the integrated assessment of clay binder-compaction interactions, (ii) optimization of key physical fuel quality parameters using a factorial experimental design, and (iii) the empirical validation of clay soil as a low-cost, non-food competing binder for high-performance briquette production. Accordingly, this study systematically investigates the combined effects of clay binder ratio and compaction level on the physical properties of carbonized rice husk briquettes to determine optimal processing conditions that balance density, durability, and combustion-related performance. The study aims to

assess these effects and identify optimal conditions for improved fuel quality. The findings contribute to the development of low-cost, efficient, and sustainable briquettes, supporting enhanced waste valorization and improved access to clean energy in resource-constrained settings.

## **2. Methodology**

### **2.1. Study Area**

The Fogera Plain, located in the South Gondar Zone of the Amhara Region, Ethiopia (11°42'N–12°03'N and 37°25'E–37°58'E), lies at an elevation of 1,500–2,500 m above sea level and is situated approximately 582 km from Addis Ababa, east of Lake Tana along the Bahir Dar–Gondar corridor. The area is predominantly flat (76 %), with minor undulating (13 %) and mountainous (11 %) terrain. The climate is characterized by mean annual temperatures of 10.3–18.8 °C and average rainfall of about 1,284 mm (Worku, 2014). This region is one of Ethiopia's major rice-producing areas, contributing approximately 70 % of national rice production (Alemu and Thompson, 2020). This has led to rapid growth in rice processing facilities, with more than 150 mills established and an average annual expansion rate of 34 % between 1997 and 2018 (Assaye and Alemu, 2020). These mills, ranging from small village-based custom mills to larger commercial facilities, are concentrated in key rice-growing areas such as Fogera, Gurafarda, and Gambella. Rice processing involves sequential operations, including cleaning, parboiling, drying, milling, polishing, and packaging. However, rice husk generated during milling is often improperly managed through open dumping or burning, or used as low-grade fuel. This practice creates environmental challenges such as air pollution, blockage of drainage systems, and street obstruction. During rainy seasons, accumulated husk can further exacerbate flooding risks in nearby settlements (Speight, 2020).

### **2.2. Materials Collection and Preparation**

The study utilizes three primary components to transform carbonized rice husk into charcoal briquettes: a carbonizer, a hammer miller, and a mold-making press.

- **Carbonizer:** This device features a frustum-shaped (or truncated pyramid) body perforated with 1 cm holes. Its square base measures 56 cm by 56 cm and is 25 cm high, topped by a welded 10 cm section before the truncated upper region. Centered at the top is a removable 10 cm-diameter, 100-cm-long air inlet tube that

screws in and out (Fig. 1a).

- Hammer Miller: Driven by an electric motor, this machine crushes biochar into powder. Operators manually feed carbonized material into a hopper; a steel rotor with hammers pulverizes it through a bottom screen into a collection chute (Fig. 1b).
- Mold Maker (Press): The briquette press utilizes a hydraulic jack with a steel frame and piston to compress a biochar-clay mix within rectangular molds. The hydraulic jack, based on Pascal's Law, amplifies force via a small piston pumping fluid into a cylinder to push a rectangular prism of clay. A 3.5 cm square prism piston, composed of 36 cubical elements, delivers the necessary hydraulic force for shaping the briquettes, with a release valve controlling lowering.

Additionally, the study utilized various raw materials and laboratory equipment, including carbonized rice husks, clay soil as a binding agent, and a temperature logger. It includes:

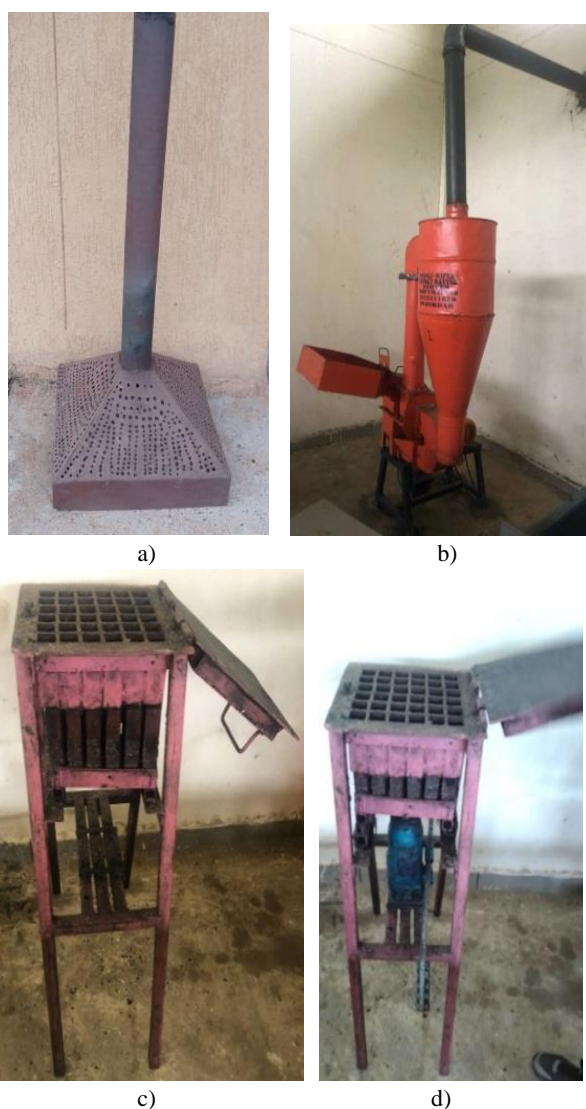
- Rice husk: Sourced from the FNRRTC milling workshop, sun-dried for 3-5 days, then carbonized in the carbonizer.
- Clay soil binder: Collected locally, dried, and passed through a 2 mm sieve to ensure uniformity.
- Equipment: A precision balance for weighing, a plastic bucket and spade for mixing biochar and clay, a leveling scoop for packing molds, a household cooking pot, and an oven for drying briquettes.

### 2.3. The procedure of the Carbonization of Rice Husk

Rice husks were collected from the FNRRTC rice processing workshop and initially sun-dried for two days to reduce moisture content to approximately 14%. The dried husks were then subjected to slow pyrolysis using a metal drum carbonizer operating under limited oxygen conditions. Carbonization was conducted at an estimated temperature range of 350-450 °C for 2-3 hours. During this process, the biomass was gradually thermally decomposed until the emission of white smoke subsided, indicating the completion of volatile release and formation of stable carbonaceous material.

The resulting carbonized rice husk (CRH) was carefully removed from the carbonizer, cooled under ambient conditions to prevent spontaneous ignition, and stored in airtight containers to avoid moisture absorption and contamination. The carbonization system (Fig. 2a) was operated with controlled airflow to maintain partial oxidation conditions and ensure consistent char quality (Fig. 2b). The cooled CRH was then size-reduced using a carbonized rice husk hammer mill (Fig. 2c) to obtain a uniform fine powder suitable for briquetting. For briquette preparation, 2 kg of milled CRH was mixed with clay soil binder at predetermined ratios, followed by the addition of approximately 2 liters of water to facilitate homogeneous blending. The mixture was thoroughly stirred to ensure uniform distribution of binder and moisture (Fig. 2d).

The prepared mixture was then transferred into prism-shaped steel molds with a square base of 3.5 mm and a height of 100 mm. Compaction was carried out using a hydraulic jack press (Fig. 2e), applying compression to achieve final briquette heights ranging from 6 to 18 mm, depending on the treatment level. After compaction, the briquettes were allowed to rest for approximately 24 hours to stabilize internal bonding and allow partial moisture redistribution. Excess surface water was drained for 3-5 minutes before drying. Finally, the formed briquettes—composed of carbonized rice husk and clay binder—were arranged on drying trays and sun-dried



**Figure 1.** Components for Carbonized Rice Husk (CRH) Briquetting Process: carbonizer (a), Hammer miller (b), mold maker (c), and mold maker with hydraulic jack (d) (Fetene and Tikuneh, 2025)

(Fig. 2f) until reaching a stable condition suitable for storage and testing.

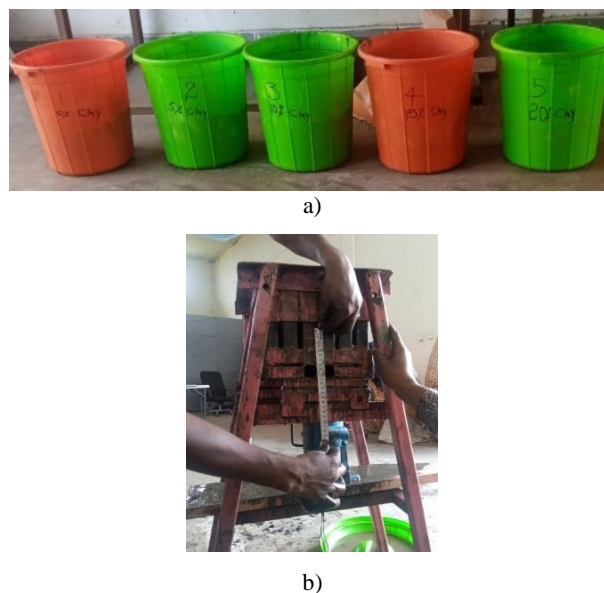


**Figure 2.** Process of carbonized Rice Husk Briquetting: Preparation of carbonizing of rice husk (a), cooling and storing of bio char (b), bio char grinding using a hammer mill (c), mixing of bio char powder with clay and water (d), molding of the mix using a mold maker and hydraulic jack (e), and bio char charcoal briquettes (f) (Fetene and Tikuneh, 2025)

## 2.4. Experimental Design

This study examined the impact of clay content and compaction level on the physical properties of carbonized rice husk (CRH) charcoal briquettes. A completely randomized design was used in a factorial experiment, with five clay ratios (0 %, 5 %, 10 %, 15 %, and 20 %) (Fig. 3a) and three compaction levels (6 mm, 12 mm, and 18 mm), resulting in 45 experimental units (3 replicates x 5 clay ratios x 3 compaction levels). Briquettes were created by mixing CRH with clay slurry at the specified ratios, then compacted using a manual hydraulic press (Fig. 3b) with a 50 mm cylindrical mold. After compaction, briquettes were air-dried (48-72

hours) and oven-dried (105 °C, 24 hours) (Chaney, 2010, Onukak et al., 2017; Fetene and Tikuneh, 2025). The study assessed the effects of clay ratio and compaction level on the briquettes' bulk density, moisture content, ash content, volatile matter, and fixed carbon.



**Figure 3.** Treatment setup: factors of clay soil ratio (a) and factors of compression level (b)

## 2.5. Variables and Data Collection

This study examined the characteristics and quality of carbonized rice husk briquettes, focusing on physical properties such as bulk density, fixed carbon, ash content, volatile matter, and moisture content.

### 2.5.1. Independent Variables

Independent variables included the properties of rice husk, the clay soil binding ratio, and the compression level. The rice husk was sourced from a local FNRRTC rice processing workshop, cleaned to remove impurities, and dried to reduce moisture for better carbonization. (Fetene and Tikuneh, 2025) Clay soil was chosen as the binding material due to its availability. When mixed with carbonized rice husk powder, it creates a cohesive mixture for briquettes, ensuring structural integrity during burning. Different studies have used varying clay soil ratios, including Celestino et al. (2023) at 2.4 % to 6.9 %, Daniel et al. (2020) at 0 % to 40 %, and Chukwunke et al. (2021) at 10 %. This study utilized a clay soil to carbonized rice husk ratio from 0 % to 20 % and 100 % to 80 % (see Fig. 3a) for optimal binding and energy content. Effective compression is essential for creating durable briquettes. The hydraulic jack controlled compaction to achieve the desired density, while insufficient or excessive compaction can lead to fragile briquettes or combustion issues (Daniel et al., 2020;

Anggraeni et al., 2021; Fetene and Tikuneh, 2025). Various studies reported different compaction levels, including Demirel (2023) at 25 MPa to 150 MPa and others ranging from 25 kPa to 630.6 kPa. This study adopted compression levels of 6 mm, 12 mm, and 18 mm, in line with existing research.

### 2.5.2. Physical Property Analysis

The observed dependent variables of the charcoal briquettes are mainly the physical characteristics of carbonized rice husk briquettes, which are conducted and determined using the equation provided below. The produced briquettes were evaluated for the following physical properties:

- **Moisture contents (%):** The percentage moisture content was determined by weighing randomly selected three sample briquettes from each treatment, placing them in a crucible of known mass and heating in an oven set at  $105\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$  for 1 hour using the formula (Chaney, 2010; Onukak et al., 2017; Fetene and Tikuneh, 2025).

$$\text{PMC} = \frac{W_1 - W_2}{W_2} \cdot 100 \quad (1)$$

Where:  $W_1$ : the initial weight of the briquette sample,  $W_2$ : the final weight of the briquette sample

- **Bulk Density ( $\text{g}/\text{cm}^3$ ):** Measured by the mass-to-volume ratio of the briquettes. The three samples of briquettes were randomly selected, and the mass of the sample briquettes was determined by weighing them before and after drying, while the volume of the briquettes was obtained by measuring their length, width, and height. Hence, the bulk density of sample briquettes was determined through the equation (Chaney, 2010; Onukak et al., 2017; Fetene and Tikuneh, 2025).

$$bd = \frac{m}{v} \quad (2)$$

Where:  $bd$ : bulk density ( $\text{g}/\text{cm}^3$ ),  $m$ : briquette mass (g), and  $v$ : the volume of briquetting ( $\text{cm}^3$ )

- **Volatile Matter (%):** The volatile matter (VM) was determined by randomly selecting three samples from each treatment by placing the briquette sample in a crucible and exposing it to a furnace at a temperature of  $550\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$  for 8 minutes, followed by weighing the sample after cooling, and then estimating using the formula (Chaney, 2010; Onukak et al., 2017; Fetene and Tikuneh, 2025).

$$\text{VM} = \frac{W_2 - W_3}{W_3} \cdot 100 \quad (3)$$

Where:  $W_2$ : represents the weight of the oven-dried sample (g),  $W_3$ : is the weight of the sample after 8 minutes in the furnace at  $550\text{ }^{\circ}\text{C}$  (g).

- **Fixed Carbon (%):** Calculated by subtracting the sum of moisture, volatile matter, and ash from 100%. The fixed carbon was also determined by the randomly selected three sample values from each treatment and computed using the following equation (Chaney, 2010; Onukak et al., 2017; Fetene and Tikuneh, 2025).

$$\text{FC}(\%) = 100 - (\text{VM} + \text{MC} + \text{AC}) \quad (4)$$

- **Ash Content (%):** Measured by combusting a known mass of briquette in a furnace at  $750\text{ }^{\circ}\text{C}$  for 3 hours. Three sample briquettes were randomly selected from each treatment and first measured the weight, then placed in a crucible of known mass and oven-dried until a constant mass was achieved. Subsequently, these samples were subjected to heating in a furnace at  $555\text{ }^{\circ}\text{C}$  for 4 hours and weighed after cooling, followed by weighing the percentage loss in mass of the sample using the following formula (Chaney, 2010; Onukak et al., 2017; Fetene and Tikuneh, 2025).

$$\text{Ashcontent}(\%) = \frac{D}{B} \cdot 100 \quad (5)$$

Where:  $D$ : the weight of the ash (Furnace-dried) in grams,  $B$ : the weight of the oven-dried sample in grams.

### 2.6. Data Analysis

Experimental data were analyzed using analysis of variance (ANOVA) in R and Statistix-10 to evaluate the effects of binder ratio and compaction level on the physical properties of the briquettes, including their interaction effects. When significant differences were detected, mean separation was performed using the least significant difference (LSD) test at a 5% probability level. In addition, Tukey's HSD test ( $\alpha=0.05$ ) was applied to confirm pairwise differences among treatment means and to group them into statistically distinct classes. The optimal treatment combination was identified based on overall performance across measured properties.

## 3. Result and Discussion

The evaluation of the physical properties of carbonized rice husk briquettes highlighted the complex relationship between the clay soil binder ratio and the degree of compaction. These two factors significantly influence key physical characteristics of the briquettes, including

bulk density, moisture content, volatile matter, fixed carbon, and ash content. The findings from this experiment provide valuable insights into how these variables interact, offering a basis for optimizing briquette quality to meet specific performance requirements and application contexts. The detailed results and their practical implications are discussed in the following sections.

### 3.1. Bulk Density of Briquettes

Bulk density is a key quality parameter in briquetting, defined as the mass per unit volume of compacted material, and it directly influences handling, transportation efficiency, storage stability, and combustion performance. Higher bulk density indicates improved structural integrity and resistance to mechanical degradation, whereas lower values reflect increased porosity and fragility. These observations are consistent with previous studies highlighting the importance of densification in biomass briquettes (Deshannavar et al., 2018; Aransiola et al., 2019). The ANOVA results showed that both compression level and clay content significantly affected bulk density ( $p < 0.05$ ), with a significant interaction effect ( $p < 0.05$ ), indicating that the effect of compression depends on binder proportion. This confirms the need for joint optimization of both factors. Clay content had a stronger effect ( $F = 16.10$ ,  $p < 0.0001$ ) than compression level ( $F = 3.98$ ,  $p = 0.0302$ ), while the interaction was highly significant ( $F = 13.28$ ,  $p < 0.0001$ ) (Table 1). The non-significant replication effect ( $p = 0.5748$ ) indicates good experimental consistency and minimal variability among blocks (Dormohamadi and Rahimnia, 2020). Bulk density ranged from 0.780 to 1.321 g/cm<sup>3</sup>. The highest value was recorded at 12 mm compression with 20 %

clay, while the lowest occurred at 12 mm compression with 5 % clay. This suggests that increased binder content enhances particle bonding, while moderate compression promotes densification through improved particle rearrangement. Coefficients of variation were low (0.59-14.03 %), indicating good precision, with the lowest variability (0.77 %) observed at 12 mm compression and 10 % clay, reflecting uniform briquette formation. Tukey’s HSD test ( $\alpha = 0.05$ ) confirmed significant differences among treatment means, grouping them into distinct statistical classes. The highest bulk density ( $1.321 \pm 0.024$  g/cm<sup>3</sup>) differed significantly from the lowest ( $0.780 \pm 0.067$  g/cm<sup>3</sup>), reinforcing the strong combined influence of binder content and compression level on briquette densification.

Optimizing bulk density enhances storage and handling efficiency, reduces transportation costs, improves combustion performance, and lowers ash residue, thereby increasing the sustainability and economic value of carbonized rice husk briquettes. These findings are consistent with previous studies emphasizing the need to optimize both binder ratio and compaction pressure for producing durable, high-performance briquettes (Olugbade et al., 2019; Daniel et al., 2020; Saeed et al., 2021; Yusuf et al., 2023; Fetene and Tikuneh, 2025).

The interaction plot (Fig. 4a) illustrates a clear combined effect of compression level and clay binder content. At 6 mm compression, bulk density showed little variation across binder ratios (0-20%), whereas at 12 mm and 18 mm compression, bulk density increased progressively with higher binder content. The diverging trends confirm a significant interaction effect, indicating that compression and binder content do not act independently.

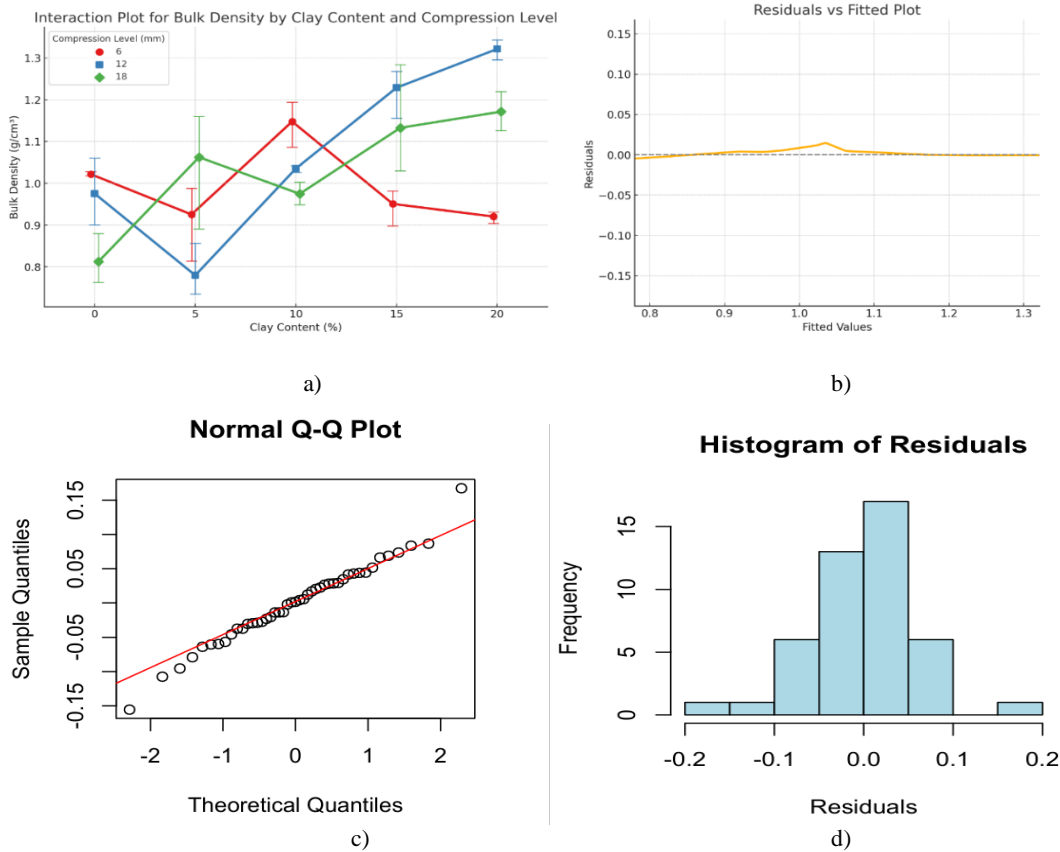
Model adequacy was confirmed through residual diagnostics. The Residuals vs. Fitted plot (Fig. 4b)

**Table 1**

Mean Bulk Density (g/cm<sup>3</sup> ± Standard Deviation), Coefficient of Variation (CV %), and Tukey Groupings for Carbonized Rice Husk Briquettes at Different Compression Levels and Clay Binder Ratios

Compression Level (mm)	Clay Content (%)	Mean ± SD (g/cm <sup>3</sup> )	CV (%)
12	20	1.321 <sup>a</sup> ± 0.024	1.82
12	15	1.229 <sup>ab</sup> ± 0.063	5.13
18	20	1.171 <sup>abc</sup> ± 0.047	4.01
6	10	1.148 <sup>abcd</sup> ± 0.056	4.88
18	15	1.133 <sup>abcde</sup> ± 0.134	11.83
18	5	1.061 <sup>bcd</sup> ± 0.149	14.03
12	10	1.035 <sup>bcd</sup> ± 0.008	0.77
6	0	1.022 <sup>bcde</sup> ± 0.006	0.59
12	0	0.980 <sup>cdef</sup> ± 0.080	8.21
18	10	0.970 <sup>cdef</sup> ± 0.027	2.77
6	15	0.951 <sup>def</sup> ± 0.047	4.94
6	5	0.925 <sup>ef</sup> ± 0.097	10.49
6	20	0.920 <sup>ef</sup> ± 0.014	1.52
18	0	0.813 <sup>fg</sup> ± 0.061	7.52
12	5	0.780 <sup>g</sup> ± 0.067	8.59

Note: Means with different letters in the Tukey Group column are significantly different at the 5 % level according to Tukey’s HSD test. Values are shown as mean bulk density (g/cm<sup>3</sup>) ± standard deviation (SD); CV (%) indicates the coefficient of variation within each treatment.



**Figure 4.** Diagnostic and interaction plots for Bulk density ( $\text{g/cm}^3$ ): (a) Interaction plot of mean Bulk density ( $\text{g/cm}^3$ ) by clay content (%) and compression level (mm) with error bars ( $\pm$  SD); (b) Residuals vs. fitted values plot displaying random scatter, supporting homogeneity of variances; (c) Normal Q-Q plot indicating approximate normality of residuals; (d) Histogram of residuals demonstrating a roughly normal distribution

showed randomly scattered residuals around zero, indicating homoscedasticity. The Normal Q-Q plot (Fig. 4c) demonstrated approximate normality, with points closely following the reference line, while the histogram of residuals (Fig. 4d) exhibited a near bell-shaped distribution without noticeable skewness or multimodality.

### 3.2. Moisture Contents of Briquettes

The results show that both compression level and clay content significantly affect moisture content in carbonized rice husk briquettes, with compression having a highly significant effect ( $p = 0.001$ ) and clay content also significant ( $p = 0.008$ ). However, their interaction was not significant ( $p = 0.75$ ), indicating that both factors act independently (Table 2). Moisture content is a key determinant of briquette combustion performance and storage stability (Jani, 2016).

Moisture trends varied across treatments. At 6 mm compression, moisture initially decreased at 5 % clay, increased at 15 %, and declined again at 20 %. At 12 mm, it increased up to 10 % clay before decreasing, while at 18 mm it peaked at 5 % clay and then declined. These variations are associated with changes in porosity and

binder behavior under different compaction levels (Aliyu et al., 2021).

The lowest moisture content occurred at 5 % clay and 6 mm compression, while the highest was observed at 10 % clay and 12 mm compression. The overall mean moisture content (4.46 %) indicates favorable storage and combustion characteristics, consistent with previous studies (Daniel et al., 2020; Chukwuneka et al., 2021; Lubwama et al., 2022). Although compression had a statistically significant effect, differences among compression levels were relatively small in practical terms. In contrast, clay content showed a clearer influence, with around 5 % clay yielding the lowest moisture content, suggesting an optimal range for moisture reduction.

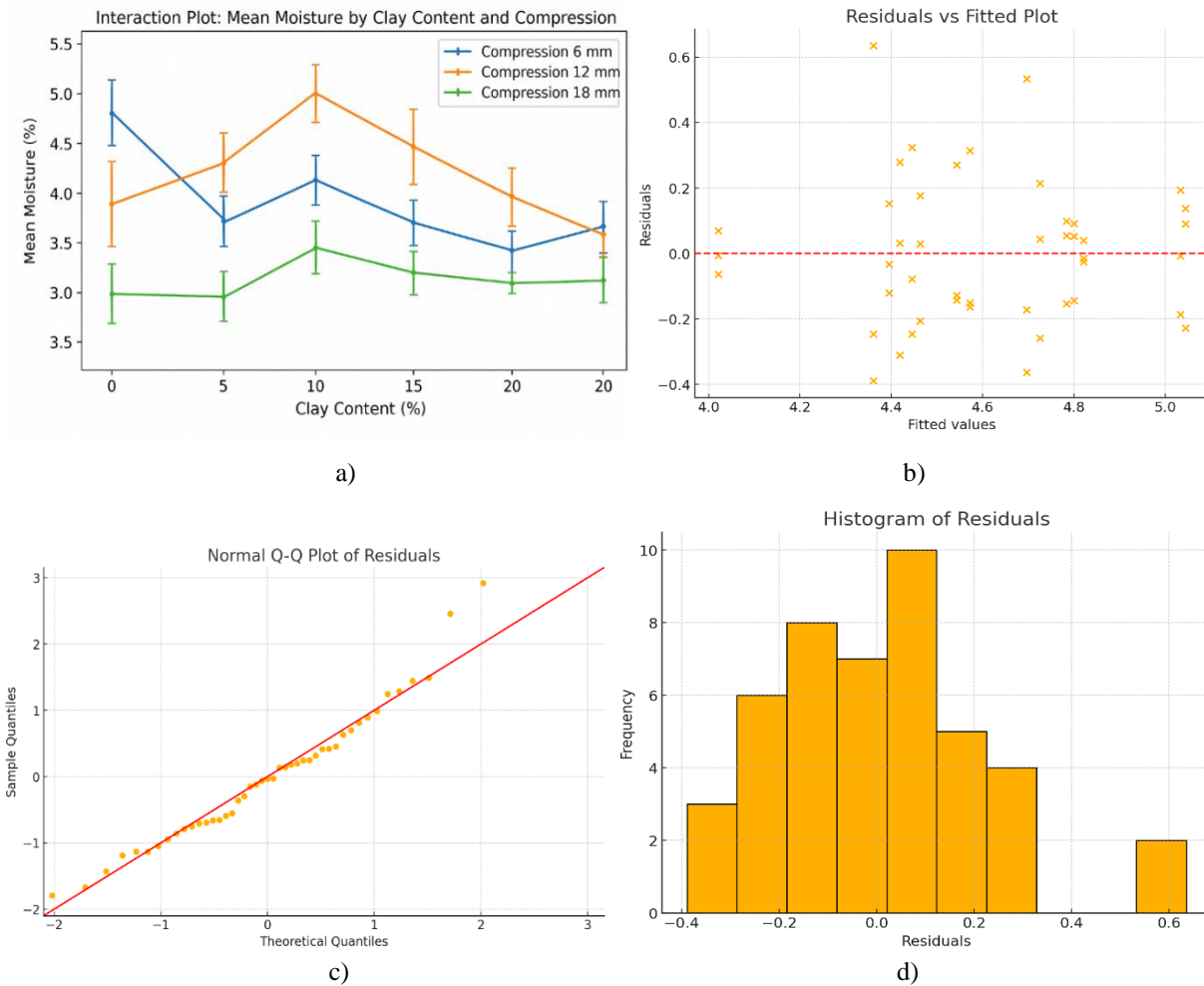
Coefficients of variation indicated good repeatability across treatments. The interaction plot (Fig. 5a) supports these findings, showing mostly parallel trends and overlapping error bars, confirming the lack of significant interaction. Moisture content generally decreased at higher clay levels ( $\geq 10$  %) and was highest at 12 mm compression, while 18 mm compression tended to reduce moisture slightly. In summary, clay content had a stronger influence than compression, and both factors operated independently.

**Table 2**

Mean Moisture Content (%) ± Standard Deviation (SD), Coefficient of Variation (CV, %), and Tukey Groupings for Carbonized Rice Husk Briquettes at Different Compression Levels and Clay Binder Ratios

Compression Level (mm)	Clay Content (%)	Mean Moisture (%) ± SD	CV (%)
6	0	4.821 <sup>a</sup> ± 0.029	0.60
6	5	4.051 <sup>d</sup> ± 0.043	1.06
6	10	4.697 <sup>ab</sup> ± 0.682	14.52
6	15	5.034 <sup>a</sup> ± 0.609	12.10
6	20	4.543 <sup>bc</sup> ± 0.211	4.65
12	0	4.464 <sup>cd</sup> ± 0.132	2.96
12	5	4.395 <sup>cd</sup> ± 0.140	3.19
12	10	5.045 <sup>a</sup> ± 0.386	7.65
12	15	4.783 <sup>ab</sup> ± 0.081	1.69
12	20	4.419 <sup>cd</sup> ± 0.292	6.60
18	0	4.361 <sup>d</sup> ± 0.497	11.40
18	5	4.800 <sup>ab</sup> ± 0.362	7.54
18	10	4.726 <sup>ab</sup> ± 0.114	2.41
18	15	4.445 <sup>cd</sup> ± 0.220	4.95
18	20	4.573 <sup>bc</sup> ± 0.264	5.77

Note. The table shows the mean moisture content (%) with standard deviation (SD), Tukey HSD grouping, and coefficient of variation (CV, %) for carbonized rice husk briquettes at different compression levels (6, 12, and 18 mm) and clay contents (0% to 20%). Means sharing the same letter within the Tukey Group column are not significantly different at  $p < 0.05$ .



**Figure 5.** Diagnostic and interaction plots for Moisture Content (%): **(a)** Interaction plot of mean moisture content by clay content (%) and compression level (mm) with error bars ( $\pm$  SD); **(b)** Residuals vs Fitted plot showing random scatter supporting homogeneity of variances; **(c)** Normal Q-Q plot indicating approximate normality of residuals; **(d)** Histogram of residuals showing roughly normal distribution (Fetene and Tikuneh, 2025)

In order to validate the ANOVA model for moisture content, residual diagnostics were performed. The interaction plot (Fig. 5a) indicates a combined effect of compression level and clay binder content. The Residuals vs Fitted plot (Fig. 5b) showed randomly distributed residuals around zero, indicating homoscedasticity, which is a key assumption for ANOVA validity (Kutner et al., 2005; Montgomery, 2017). The Normal Q-Q plot (Fig. 5c) revealed that residuals closely followed the theoretical line with only minor deviations at the tails, suggesting approximate normality (Ghasemi and Zahediasl, 2012; Field, 2024).

Similarly, the histogram (Fig. 5d) exhibited a near bell-shaped distribution, further supporting the normality assumption (Razali and Wah, 2011). Collectively, these diagnostics confirm that ANOVA assumptions were satisfied, supporting the reliability of the moisture content analysis.

### 3.3. Volatile Matter of Briquettes

The two-way ANOVA showed that both compression level (mm) and clay content (%) significantly influenced volatile matter in carbonized rice husk briquettes ( $p < 0.05$ ), with a significant interaction effect ( $p = 0.0027$ ). Among the main effects, clay content had a highly significant influence ( $p < 0.001$ ), whereas compression level alone was not significant ( $p \approx 0.29$ ) (Table 3), indicating that clay content is the dominant controlling factor. Tukey’s HSD test revealed clear treatment groupings. Briquettes without clay (0 %) consistently exhibited the highest volatile matter (22.87 %-24.48 %), forming group “a.”

Increasing clay content resulted in a progressive reduction in volatile matter, with 5-10 % clay forming

group “b,” while 20 % clay at 6 mm compression produced the lowest value (13.41 %), classified as group “c.” The significant interaction indicates that the effect of clay depends on compression level, with the strongest reduction occurring at lower compression, highlighting the need for joint optimization of both factors. The distribution of mean values ( $\approx 13$  %-24 %) showed a slight right skew, driven by higher volatile matter at zero clay and low compression. Coefficients of variation ranged from 0.85 % to 11.92 % (mean  $\approx 5.77$  %), indicating good repeatability. Lower variability at higher clay levels suggests improved particle cohesion and sample homogeneity.

Volatile matter, representing the fraction of organic compounds released during combustion, strongly influences ignition behavior, flame stability, and energy release (Shiferaw et al., 2017; Ngene et al., 2024). While higher volatile matter promotes easier ignition, it may reduce overall heating value (Inegbedion and Ikpoza, 2022). The observed reduction with increasing clay content is consistent with previous studies (Chukwunke et al., 2021; Lubwama et al., 2022), while the mean value ( $\sim 17.86$  %) aligns with reported ranges (Daniel et al., 2020; Celestino et al., 2023).

Notably, intermediate conditions (12 mm compression with 10 % clay) maintained a balanced volatile fraction, suggesting a compromise between ignition performance and structural stability. Generally, clay content exerted a stronger influence than compression level, emphasizing that volatile matter is primarily governed by binder proportion. These findings underscore the importance of jointly optimizing binder content and compression conditions to achieve balanced briquette performance in terms of ignition, combustion behavior, and structural integrity.

**Table 3**

Mean Volatile Matter Content (%  $\pm$  Standard Deviation), Coefficient of Variation (CV%), and Tukey Groupings for Carbonized Rice Husk Briquettes at Different Compression Levels and Clay Binder Ratios

Compression Level (mm)	Clay Content (%)	Mean Volatile Matter (%) $\pm$ SD	CV (%)
6	0	24.48 <sup>a</sup> $\pm$ 0.53	2.17
18	0	23.53 <sup>a</sup> $\pm$ 1.52	6.46
12	0	22.87 <sup>a</sup> $\pm$ 1.83	8.00
18	5	19.41 <sup>b</sup> $\pm$ 0.82	4.23
12	10	18.55 <sup>b</sup> $\pm$ 1.31	7.06
12	5	17.35 <sup>b</sup> $\pm$ 0.60	3.46
6	5	17.27 <sup>b</sup> $\pm$ 1.28	7.41
18	10	16.69 <sup>bc</sup> $\pm$ 1.99	11.92
18	20	16.76 <sup>bc</sup> $\pm$ 0.69	4.12
18	15	15.93 <sup>bc</sup> $\pm$ 0.38	2.38
12	20	15.36 <sup>bc</sup> $\pm$ 0.13	0.85
12	15	15.39 <sup>bc</sup> $\pm$ 0.58	3.77
6	15	15.46 <sup>bc</sup> $\pm$ 0.23	1.49
6	10	15.36 <sup>bc</sup> $\pm$ 0.73	4.75
6	20	13.41 <sup>c</sup> $\pm$ 0.29	2.16

Note: Means with different letters in the Tukey Group column are significantly different at the 5 % level according to Tukey’s HSD test. Values are shown as mean volatile matter content (%)  $\pm$  standard deviation (SD); CV (%) indicates the coefficient of variation within each treatment.

The interaction plot (Fig. 6a) indicates that volatile matter decreases with increasing clay content across all compression levels (6, 12, and 18 mm). The highest values ( $\approx 22.5\text{-}24.5\%$ ) were observed at 0% clay, followed by a marked decline at 5-10% clay, with the steepest reduction occurring at 6 mm compression.

At higher clay contents (15-20%), volatile matter values stabilized and converged across compression levels, with the lowest values recorded at 6 mm and 20% clay. This pattern confirms a significant interaction effect, where clay content reduces volatile matter more strongly at lower compression, while higher compression slightly moderates this effect, likely due to increased densification. Similar trends of decreasing volatile matter with increasing binder content have been reported in biomass briquetting studies (Chukwunke et al., 2021; Lubwama et al., 2022), while compaction effects on volatile retention have also been noted (Olugbade et al., 2019). Residual diagnostics (Fig. 6b-d) validated the ANOVA assumptions. The Normal Q-Q plot showed residuals closely aligned with the reference line,

indicating approximate normality (Razali and Wah, 2011; Ghasemi and Zahediasl, 2012), while the histogram exhibited a near bell-shaped distribution centered around zero.

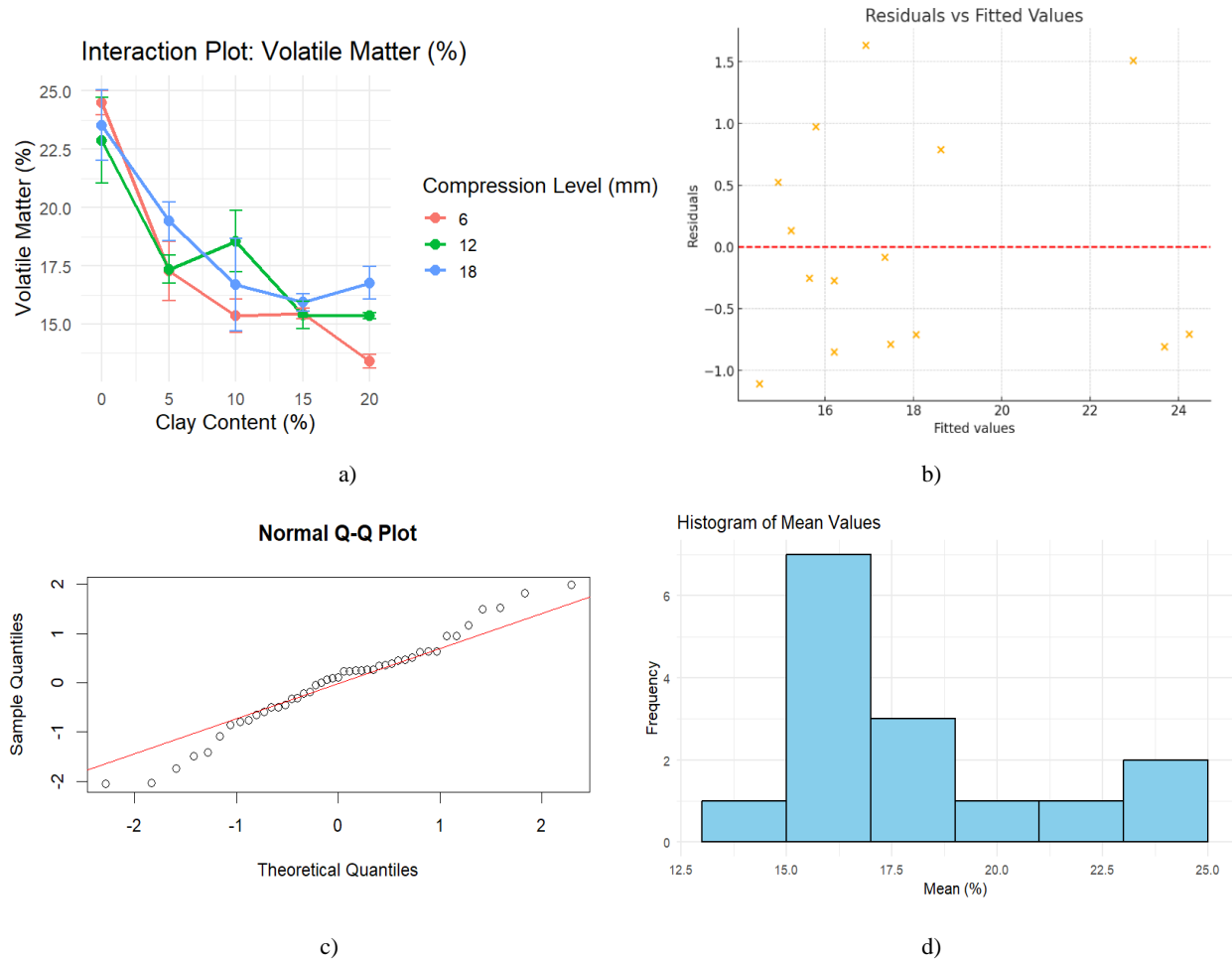
The Residuals vs Fitted plot showed no systematic pattern, confirming homoscedasticity and independence (Kutner et al., 2005; Montgomery, 2017; Fetene and Tikuneh, 2025).

Collectively, these results support the validity of the ANOVA model and justify subsequent post-hoc analysis.

### 3.4. Fixed Carbon of Briquettes

The ANOVA results indicated that both compaction pressure (CP) and clay binder ratio (BR) significantly affected the fixed carbon content of carbonized rice husk briquettes.

Clay content had a highly significant effect ( $p < 0.001$ ), accounting for the largest share of variability (Sum Sq = 1051.4), while compaction pressure was also significant ( $p = 0.003$ ) but contributed less (Sum Sq = 39.0).



**Figure 6.** Diagnostic and interaction plots for Volatile Matter (VM, % wt): (a) Interaction plot of mean volatile matter by clay content (%) and compression level (mm) with error bars ( $\pm$  SD); (b) Residuals vs. fitted values plot displaying random scatter, supporting homogeneity of variances; (c) Normal Q-Q plot indicating approximate normality of residuals; (d) Histogram of residuals demonstrating a roughly normal distribution (Fetene and Tikuneh, 2025)

A significant CP · BR interaction ( $p = 0.008$ ) confirms that the influence of clay content depends on the applied compression level. The interaction plot shows that fixed carbon increases with clay content at all compression levels, with the most pronounced improvement at 6 mm compression. At higher compression levels (12 and 18 mm), the increase in fixed carbon tends to plateau beyond 10-15 % clay, likely due to enhanced densification promoting volatile expulsion during compaction.

Overall, the results indicate that lower compression combined with higher binder content favors greater carbon retention. Low residual variance and coefficients of variation ( $\approx 0.3-6\%$ ) confirm strong measurement reliability and experimental consistency (Table 4). The lowest variability occurred at higher clay levels (15-20 %), particularly at 20 % clay and 6 mm compression, which also produced the maximum fixed carbon ( $68.27\% \pm$  low CV). In contrast, slightly higher variability (4-6 %) was observed at low clay levels (0-5 %), reflecting reduced structural stability when binder content is insufficient. These findings are consistent with previous studies (Ikelle et al., 2014; Lubwama et al., 2020; Nazari et al., 2020; Adu-Poku et al., 2022; Sam Obu et al., 2022), which highlight the importance of optimizing binder ratio and compaction conditions to enhance fixed carbon and overall fuel quality. Collectively, the results demonstrate that increasing clay content—particularly at lower compaction pressures—improves fixed carbon yield, structural stability, and combustion potential, thereby enhancing the suitability of briquettes as a sustainable biofuel (Narzary et al., 2023). The interaction plot (Fig.

7a) shows that fixed carbon increases with rising clay content at all compression levels, with the most pronounced improvement at 6 mm compression, where values increase from approximately 50 % to nearly 68 % at 20 % clay. At higher compression levels, the increase is less pronounced, with a plateau observed beyond 10-15 % clay. Similar trends of increasing fixed carbon with higher binder content have been reported in biomass briquetting studies, where improved bonding and reduced volatile losses enhance carbon retention (Chukwunke et al., 2021; Lubwama et al., 2022; Sam Obu et al., 2022). The observed plateau at higher binder levels is also consistent with findings that excessive binder addition may yield diminishing returns in fuel improvement (Nazari et al., 2020). Error bars indicate generally good repeatability, with slightly higher variability at intermediate clay levels (5-10 %).

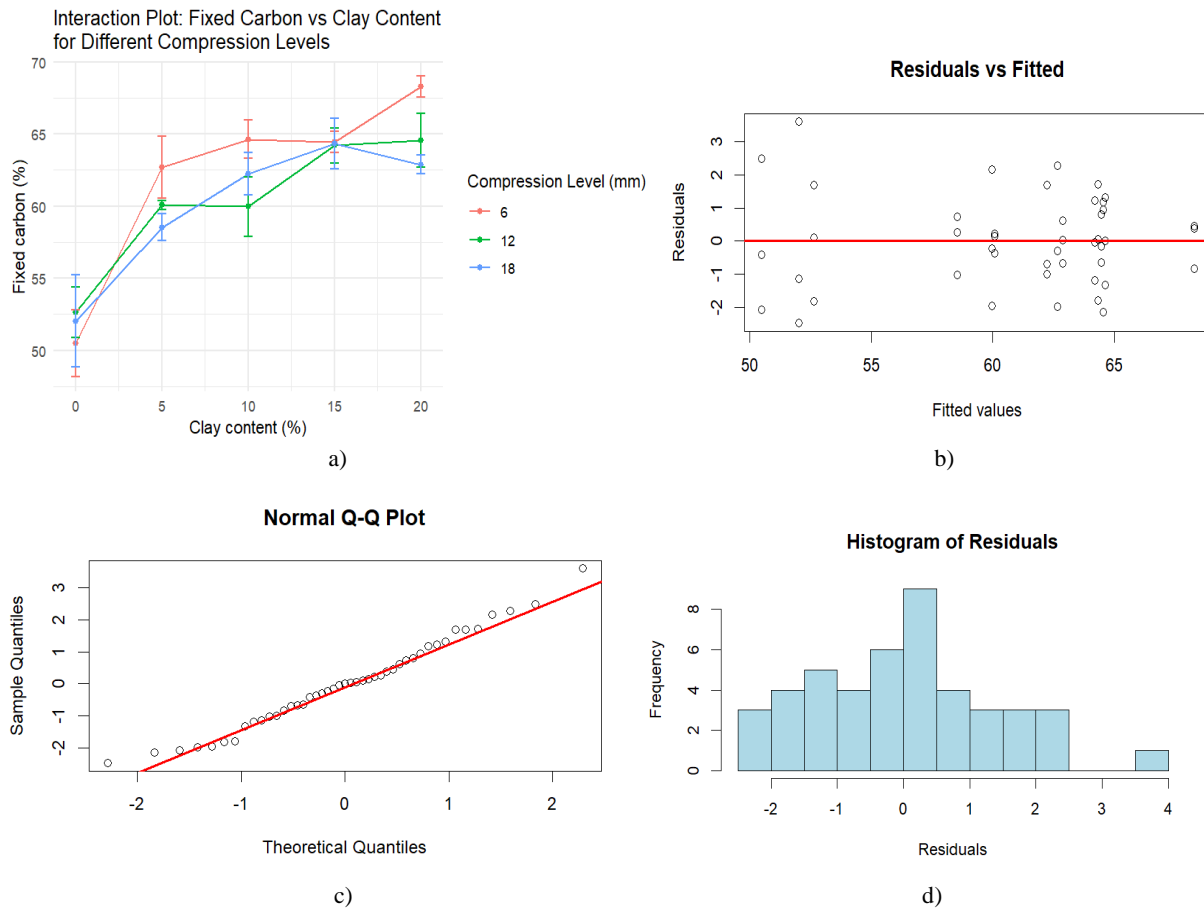
These results suggest that optimizing clay content in combination with lower compression can maximize fixed carbon and improve fuel quality. Residual diagnostics confirmed the validity of the ANOVA model. Residuals were randomly distributed around zero (Fig. 7b), the Q-Q plot indicated approximate normality (Fig. 7c), and the histogram showed a near-symmetric distribution (Fig. 7d). Similar diagnostic approaches for validating ANOVA assumptions are well established in statistical literature (Razali and Wah, 2011; Ghasemi and Zahediasl, 2012; Montgomery, 2017). Collectively, these results confirm that model assumptions were satisfied and support the reliability of the statistical inferences.

**Table 4**

Mean Fixed Carbon Content ( $\% \pm$  Standard Deviation), Coefficient of Variation (CV %), and Tukey Groupings for Carbonized Rice Husk Briquettes at Different Compression Levels and Clay Binder Ratios

Compression level (mm)	Clay content (%)	Mean Fixed Carbon (%) $\pm$ SD	CV (%)
6	0	50.49 <sup>d</sup> $\pm$ 2.37	4.70
18	0	52.03 <sup>d</sup> $\pm$ 3.07	5.90
12	0	52.64 <sup>d</sup> $\pm$ 1.79	3.40
12	5	60.06 <sup>c</sup> $\pm$ 0.33	0.55
6	5	62.67 <sup>bc</sup> $\pm$ 2.39	3.81
18	5	58.54 <sup>c</sup> $\pm$ 1.73	2.95
18	20	62.87 <sup>bc</sup> $\pm$ 0.70	1.11
12	10	59.29 <sup>c</sup> $\pm$ 2.13	3.59
18	10	62.90 <sup>bc</sup> $\pm$ 1.58	2.51
6	10	64.63 <sup>ab</sup> $\pm$ 1.39	2.15
12	15	64.20 <sup>ab</sup> $\pm$ 1.21	1.89
18	15	64.34 <sup>ab</sup> $\pm$ 1.94	3.02
6	15	64.45 <sup>ab</sup> $\pm$ 0.99	1.54
12	20	64.54 <sup>ab</sup> $\pm$ 1.88	2.91
6	20	68.27 <sup>a</sup> $\pm$ 0.19	0.28

Note: Means with different letters in the Tukey Group column indicate significant differences at the 5% level (Tukey’s HSD test). Values are presented as mean fixed carbon content ( $\% \pm$  standard deviation (SD)); CV (%) shows the relative variability within each treatment group.



**Figure 7.** Diagnostic and interaction plots for Fixed carbon (%): (a) Interaction plot of mean Fixed carbon (%) by clay content (%) and compression level (mm) with error bars ( $\pm$  SD); (b) Residuals vs. fitted values plot displaying random scatter, supporting homogeneity of variances; (c) Normal Q-Q plot indicating approximate normality of residuals; (d) Histogram of residuals demonstrating a roughly normal distribution

### 3.5. Ash Content of Briquettes

The ANOVA results showed that both compression level (CP) and clay content (BR) significantly influenced ash content in carbonized rice husk briquettes. Compression level had a significant effect ( $F(2,30) = 4.09, p = 0.027$ ), while clay content had a highly significant effect ( $F(4,30) = 29.85, p < 0.0001$ ), indicating that binder ratio is the dominant factor in reducing ash content (Table 5). However, the  $CP \times BR$  interaction was not significant ( $F(8,30) = 0.71, p = 0.68$ ), suggesting that the effect of clay content is consistent across all compression levels (Saeed et al., 2021; Sunnu et al., 2023).

Ash content decreased markedly with increasing clay addition, with the largest reduction observed when any clay was added compared to 0 %. Tukey’s HSD test showed that 0 % clay ( $20.07 \pm 1.29$  %, group “a”) was significantly higher than 5 % clay ( $17.73 \pm 1.26$  %, group “b”), while 10-20 % clay ( $\approx 15.4$ - $15.9$  %) formed a statistically similar lower group (“c”). This indicates diminishing returns beyond 5-10 % clay, consistent with previous studies emphasizing the dominant role of binder composition in controlling ash content (Lubwama et al.,

2022; Celestino et al., 2023). For compression, briquettes compacted at 6 mm ( $17.73 \pm 2.86$  %) and 12 mm ( $17.89 \pm 2.11$  %) differed significantly, while 18 mm ( $17.59 \pm 2.40$  %) overlapped both groups, indicating a smaller but measurable effect of compression on ash content. Given the non-significant interaction, the decreasing trend with increasing clay content remained consistent across all compression levels.

As shown in Fig. 8a, ash content decreases progressively with increasing clay ratio from 0 % to 20 %, with only minor variation across compression levels.

The relatively high ash content is attributed to the presence of silica ( $SiO_2$ ) in both rice husk and clay, yielding an average ash content of 16.74 %, consistent with Celestino et al. (2023) and Fetene and Tikuneh, (2025). Since high ash content can reduce combustion efficiency and increase emissions, optimizing binder ratio and compression remains essential for improving fuel quality (Ikelle et al., 2014; Lubwama et al., 2022).

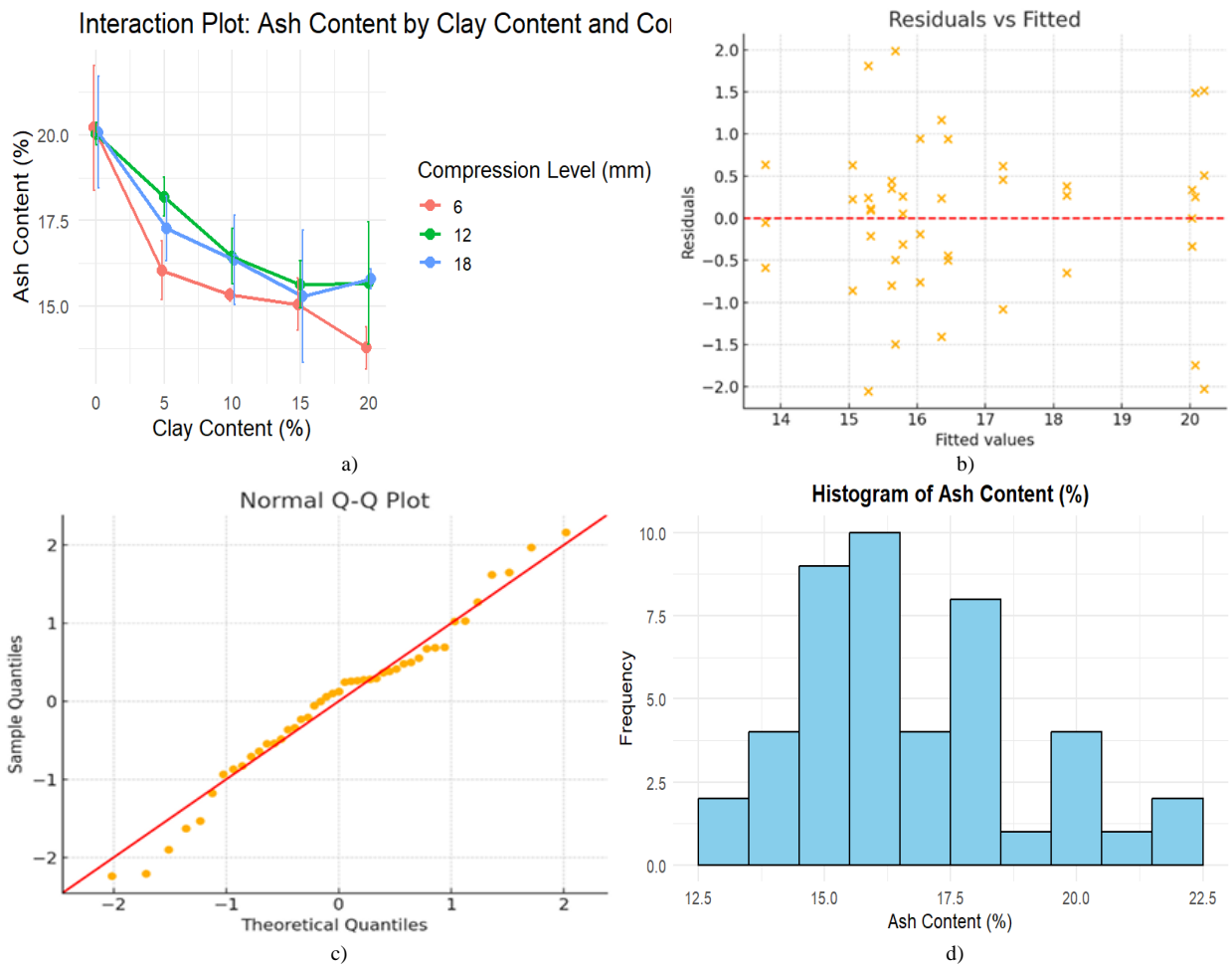
In conclusion, moderate clay addition (around 5-10 %) is sufficient to significantly reduce ash content, while higher levels provide limited additional benefit, indicating diminishing returns at elevated binder ratios.

**Table 5**

Mean Ash Content (% ± Standard Deviation), Coefficient of Variation (CV %), and Tukey Groupings for Carbonized Rice Husk Briquettes at Different Compression Levels and Clay Binder Ratios

Compression Level (mm)	Clay content (%)	Mean Ash Content (% ± SD)	CV (%)
6	0	20.54 <sup>a</sup> ± 1.78	8.67
6	5	16.71 <sup>b</sup> ± 0.86	5.15
6	10	15.32 <sup>c</sup> ± 0.16	1.05
6	15	15.05 <sup>c</sup> ± 0.67	4.45
6	20	13.77 <sup>d</sup> ± 0.61	4.43
12	0	20.03 <sup>a</sup> ± 0.34	1.70
12	5	18.19 <sup>b</sup> ± 0.48	2.64
12	10	16.45 <sup>c</sup> ± 0.76	4.62
12	15	15.63 <sup>c</sup> ± 0.67	4.29
12	20	15.68 <sup>cd</sup> ± 1.69	10.78
18	0	20.08 <sup>a</sup> ± 1.63	8.11
18	5	17.59 <sup>b</sup> ± 0.86	4.89
18	10	16.35 <sup>bc</sup> ± 1.06	6.48
18	15	15.28 <sup>cd</sup> ± 1.96	12.83
18	20	15.79 <sup>cd</sup> ± 0.29	1.84

Note: Means with different letters (a, b, c, d) indicate significant differences according to Tukey’s HSD test at  $p < 0.05$ . Values are presented as mean ash content (%) ± standard deviation (SD); CV (%) is the coefficient of variation within each treatment.



**Figure 8.** Diagnostic and interaction plots for ash content (%): (a) Interaction plot of mean ash content by clay content (%) and compression level (mm) with error bars ( $\pm$  SD); (b) Residuals vs. fitted values plot displaying random scatter, supporting homogeneity of variances; (c) Normal Q-Q plot indicating approximate normality of residuals; (d) Histogram of residuals demonstrating a roughly normal distribution

The interaction plot (Fig. 8a) shows that ash content decreases as clay content increases from 0 % to 20 % across all compression levels. The greatest reduction occurs with the initial addition of clay (0 % to 5 %), after which the effect gradually levels off at higher clay contents.

The 6 mm compression level exhibits the steepest decline, suggesting a stronger response of ash content to clay addition under lower compaction. Similar reductions in ash content with increasing binder proportion have been reported in biomass briquette studies, where improved binding reduces combustible impurities and alters mineral retention behavior (Saeed et al., 2021; Lubwama et al., 2022; Celestino et al., 2023).

The largely parallel trends confirm the non-significant interaction observed in the ANOVA, indicating that the effect of clay content is consistent across compression levels. Overlapping error bars at higher clay contents further support the lack of significant differences beyond 10 % clay. Model diagnostics (Fig. 8b-d) confirmed that ANOVA assumptions were adequately met.

The Residuals vs Fitted plot showed randomly distributed residuals around zero, indicating constant variance (Kutner et al., 2005; Montgomery, 2017).

The Normal Q-Q plot indicated that residuals closely followed the reference line with only minor tail deviations, suggesting approximate normality (Razali and Wah, 2011; Ghasemi and Zahediasl, 2012).

The histogram of residuals showed a unimodal distribution centered around 15-18 %, with mild skewness but no extreme outliers. Collectively, these diagnostics support the validity of the ANOVA model and the appropriateness of parametric analysis for this dataset.

## 4. Conclusion and Recommendations

### 4.1. Conclusion

This study used a 3 · 5 factorial design to evaluate the effects of compaction level (6, 12, and 18 mm) and clay soil binder ratio (0-20 %) on the physical properties of carbonized rice husk briquettes. The main findings are summarized as follows:

- Bulk density was significantly affected by the interaction between binder ratio and compaction level, ranging from 0.780 g/cm<sup>3</sup> (5 % clay, 12 mm) to 1.321 g/cm<sup>3</sup> (20 % clay, 12 mm).
- Moisture content was influenced by both factors independently, with low values (~4.02-4.04 %) indicating favorable storage and combustion characteristics.
- Volatile matter decreased with increasing clay content, from 24.48 % (0 % clay, 6 mm) to 13.41 % (20 % clay, 6 mm).
- Fixed carbon increased with higher binder ratios,

rising from 50.49 % to 68.27 %, indicating improved carbon retention.

- Ash content declined with increasing clay content, from 20.21 % to 13.77 %, enhancing fuel quality.

Collectively, the results show that clay binder ratio and compaction level strongly influence briquette quality. Higher clay content with moderate compaction improves density, fixed carbon, and ash reduction, while lower binder levels enhance volatile matter and ignition characteristics, highlighting the need for joint optimization for balanced bioenergy performance. Study limitations: Calorific value and other energy-related parameters were not measured and should be included in future work. In addition, compaction was controlled via piston displacement due to equipment constraints; applied pressure was estimated where possible but not directly measured in all cases, which should be considered when interpreting and comparing the results.

### 4.2. Recommendations

Based on the findings, the following recommendations are proposed for researchers, producers, and policymakers:

#### i. Optimization of binder ratio and compaction

- For high-density, high-carbon briquettes: use 15-20 % clay binder with moderate compaction (~12 mm).
- For easy ignition: use ≤5 % binder with lower compaction (6 mm).

#### ii. Alternative binders

- Investigate locally available binders such as starch, cassava flour, molasses, and other agricultural residues, and compare them with clay in terms of cost, availability, durability, and combustion performance.

#### iii. Thermal and combustion evaluation

- Conduct calorific value analysis, combustion testing, and emissions assessment to better quantify energy efficiency and environmental performance.

#### iv. Economic feasibility and adoption

- Evaluate production costs, supply chain practicality, and user acceptance to support scalable and economically viable implementation.

#### v. Scale-up and field validation

- Pilot large-scale production and assess storage stability, transport performance, and end-user usability under real operating conditions.

In conclusion, optimizing binder ratio and compaction significantly improves briquette quality and sustainability. Further research on alternative binders, combustion performance, and field validation will enhance the development of affordable and cleaner biomass fuel options for rural and peri-urban communities.

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# Uticaj udela glinenog veziva i intenziteta kompresije na fizička svojstva briketa od karbonizovane pirinčane ljuske

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## I Z V O D

Ova studija procenjuje uticaj udela glinenog veziva i nivoa kompresije na fizička svojstva briketa od karbonizovane pirinčane ljuske u cilju optimizacije održivog goriva. Briketi su proizvedeni uz dodatak 0-20 % glinenog veziva i presovani pri nivoima kompresije od 6, 12 i 18 mm, prema faktorskom eksperimentalnom planu. Određivani su nasipna gustina, sadržaj vlage, sadržaj isparljivih materija, sadržaj fiksnog ugljenika i sadržaj pepela, pri čemu su oba faktora, kao i njihova međusobna interakcija, značajno uticali na sva ispitivana svojstva.

Najniži sadržaj vlage (4,02 %) postignut je pri dodatku 5 % veziva i kompresiji od 6 mm, dok se sadržaj isparljivih materija smanjio sa 24,48 % (bez dodatka veziva) na 13,41 % (pri 20 % veziva). Sadržaj fiksnog ugljenika povećao se sa 50,49 % na 68,27 % sa povećanjem udela veziva, dok se sadržaj pepela smanjio sa 20,21 % na 13,77 %. Veći udeo veziva poboljšao je nasipnu gustinu i mehaničku postojanost briketa, dok je povećanje stepena kompresije dodatno unapredilo gustinu i čvrstoću kod svih ispitivanih uzoraka.

Optimalni uslovi postignuti su pri dodatku 5-10 % glinenog veziva i kompresiji od 12 mm, čime je obezbeđena ravnoteža između kvaliteta goriva i stabilnosti pri skladištenju. Rezultati ukazuju da su udeo veziva i intenzitet kompresije ključni parametri koji određuju kvalitet briketa od karbonizovane pirinčane ljuske za održive primene u ruralnim energetske sistemima.