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Please cite this article as doi: 10.4081/jae.2026.2001

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Submitted: 11 October 2025

Accepted: 25 May 2026

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Contributions: **Jemmy J.S. Dethan**, Conceptualization, methodology, formal analysis, writing – original draft, writing – review and editing, supervision. **Fredrik J. Haba Bunga**, Data collection, experimentation, investigation, writing – original draft. **Godlief F. Neonufa**, Validation, resources, writing – review and editing.

All authors have read and agreed to the published version of the manuscript.

Conflict of interest: The authors declare no conflict of interest.

Availability of data and materials: The data that support the findings of this study are available from the corresponding author upon reasonable request.

Declaration of generative ai and ai-assisted technologies in the writing process: During the preparation of this manuscript, the corresponding author used Claude AI (Anthropic) to assist with language editing and improving the readability of the text. After using this tool, the authors reviewed and edited the content as necessary and take full responsibility for the content of the publication.

Funding: This research was funded by the Research Institute (Lembaga Penelitian) of Artha Wacana Christian University (Universitas Kristen Artha Wacana), Kupang, Indonesia.

Abstract

Particle size is a critical parameter influencing the quality of biomass briquettes, particularly in char-based systems where bonding mechanisms differ from raw biomass briquetting. This study investigates the effect of particle size on the physical, mechanical, and proximate properties of candlenut shell (*Aleurites moluccana*) char briquettes produced under controlled compaction conditions. Briquettes were prepared from three particle size fractions (0.84, 0.42, and 0.25 mm) using a constant binder content, moisture level, compaction pressure, and dwell time. The results show that reducing particle size led to improved bulk density and compressive strength, with finer briquettes exhibiting higher mechanical stability. Proximate analysis revealed a decrease in ash and volatile matter contents and an increase in fixed carbon as particle size decreased, indicating enhanced fuel quality for finer particles. Empirical relationships between particle size and briquette properties exhibited strong goodness-of-fit, with coefficients of determination (R^2) exceeding 0.95 within the tested range. These findings demonstrate that particle size significantly influences both the mechanical integrity and fuel characteristics of candlenut shell char briquettes. The results provide feedstock-specific experimental insights that can support process optimization in char-based briquetting systems, while emphasizing that the derived relationships are valid only within the investigated particle size range.

Key words: candlenut shell char; biomass briquettes; particle size; mechanical properties; proximate analysis; empirical modeling.

Introduction

The increasing demand for sustainable and low-carbon energy sources has intensified global interest in the utilization of agricultural residues as solid biofuels. Biomass briquettes offer an effective approach to improving the handling, storage, and energy density of loose biomass while supporting waste valorization and circular bioeconomy strategies. In many developing regions, including Indonesia, biomass-based solid fuels remain an important component of household and small-scale energy systems, emphasizing the need for reliable and efficient densification technologies suitable for agricultural applications.

Candlenut shells (*Aleurites moluccana* L.) are an abundant agro-industrial by-product generated during nut processing. Global candlenut production exceeds one million tons annually, resulting

in large quantities of shell residues with high fixed carbon content and favorable calorific properties. However, direct utilization of candlenut shells is constrained by their low bulk density and poor handling characteristics, making densification through briquetting a necessary preprocessing step for their effective use as solid biofuels.

Briquettes can be produced from either raw biomass or carbonized biomass (char), and these two systems differ fundamentally in their bonding mechanisms and material behavior. In raw biomass briquettes, inter-particle bonding is largely governed by the softening and flow of natural lignin under pressure and temperature. In contrast, char-based briquettes rely primarily on external binders, mechanical interlocking, and particle rearrangement, as carbonization largely eliminates the binding role of lignin. As a result, parameters such as particle size, binder distribution, and pore structure become more critical in determining the mechanical integrity and fuel-related properties of char briquettes. These differences imply that conclusions derived from raw biomass briquettes cannot be directly extrapolated to char-based systems.

Particle size is widely recognized as a critical preprocessing parameter influencing the physical integrity and handling performance of densified biomass fuels. Previous studies have shown that reducing particle size generally improves packing efficiency, inter-particle contact, and load transfer during compaction, leading to higher bulk density and compressive strength (Kaliyan and Vance Morey, 2009; Mani *et al.*, 2006; Pang *et al.*, 2019). However, the effects of particle size are not universal and may vary significantly depending on biomass type, degree of carbonization, binder characteristics, and compaction conditions.

Comparable particle-size-dependent trends in mechanical and proximate properties have been reported for briquettes derived from tropical agricultural residues, including nut shells and woody biomass, where finer particles promote improved densification behavior and enhanced carbon retention (Abineno *et al.*, 2024; Clauser *et al.*, 2021; Demirbaş *et al.*, 2004; Dethan, 2024). These findings highlight that particle size effects are feedstock- and process-dependent, reinforcing the need for material-specific investigations rather than generalized assumptions.

Despite extensive research on particle size effects in raw and partially dried biomass briquettes, systematic studies focusing on char-based briquettes remain limited. In particular, empirical modeling of particle size effects is often presented as broadly applicable, even though densification behavior is strongly influenced by feedstock properties and processing conditions. For candlenut shell char briquettes, quantitative experimental data linking particle size to mechanical and

proximate properties under controlled compaction pressure are still scarce, and feedstock-specific empirical relationships have not been sufficiently established.

Therefore, the objective of this study is to experimentally investigate the influence of particle size on the physical, mechanical, and proximate properties of candlenut shell char briquettes produced under controlled compaction conditions. By examining three discrete particle size levels and developing empirical relationships valid within the tested range, this work aims to provide feedstock-specific insights into densification behavior while avoiding overgeneralization. The results are expected to contribute to improved understanding of particle size effects in char-based briquetting systems and to support future optimization of agricultural biomass densification processes.

Materials and Methods

Raw candlenut shells were collected from a local nut processing facility in Lembata, Indonesia. The shells were first air-dried to a moisture content below 10% and then carbonized in a muffle furnace at 500 °C for 2 h under limited oxygen conditions to obtain char. After carbonization, the material was cooled in a desiccator and ground using a high-speed grinder. The resulting powder was sieved into three particle size ranges: 20 mesh (0.84 mm), 40 mesh (0.42 mm), and 60 mesh (0.25 mm), representing coarse, medium, and fine fractions, respectively.

Commercial cassava starch was used as a binder at 10 wt% of the total briquette mass, while distilled water was added to facilitate homogeneous mixing. Based on the proportion of added water and dry materials, the briquetting mixture had an estimated moisture content of approximately 12% (wet basis) at the time of compaction. This estimated moisture level lies within the range commonly reported as suitable for starch-bonded biomass briquettes, providing sufficient plasticity and promoting effective inter-particle bonding during densification.

Rationale for process parameter selection

The selection of three particle size classes (0.84, 0.42, and 0.25 mm) was intended to represent coarse, medium, and fine fractions commonly used in biomass densification studies. These ranges are widely reported to capture the transition from void-dominated packing (coarse particles) to enhanced inter-particle contact and bonding (fine particles) (Kaliyan and Vance Morey, 2009; Pang *et al.*, 2019).

A starch binder content of 10 wt% was selected based on previous studies indicating that 8-12 wt% starch provides an optimal balance between mechanical strength and fuel quality for carbonized biomass briquettes (Kaliyan and Vance Morey, 2009; Mani *et al.*, 2006; Sengar *et al.*, 2012). The addition of 10 wt% water was applied to activate starch gelatinization and improve binder distribution without causing excessive moisture-related defects.

The compaction pressure of 20 MPa was chosen as a representative medium-pressure regime, sufficient to achieve stable briquette formation while avoiding excessive energy consumption or die wear. Similar pressure levels have been successfully applied in the briquetting of nut shells and agricultural residues (Demirbaş *et al.*, 2004; Mani *et al.*, 2006; Pang *et al.*, 2019).

A dwell time of 60 s was applied to allow stress relaxation, binder flow, and particle rearrangement under constant load, ensuring uniform densification (Kaliyan and Vance Morey, 2009; Mani *et al.*, 2006; Pang *et al.*, 2019). Preliminary trials confirmed that shorter dwell times (<30 s) resulted in lower mechanical stability, while longer times (>90 s) did not yield significant additional strength improvements.

Briquette preparation

The prepared mixtures were compacted using a hydraulic press at a constant pressure of 20 MPa into cylindrical molds (diameter = 40 mm, height = 30 mm). This corresponded to a cross-sectional area of $1.26 \times 10^{-3} \text{ m}^2$, with the applied pressure (20 MPa) calculated from the ratio of load to mold area, equivalent to approximately 25.1 kN total load. Load cell calibration was verified prior to each test to ensure accuracy. Compressive strength (σ_c) was measured using a Universal Testing Machine (5 kN capacity, crosshead speed $1 \text{ mm} \cdot \text{min}^{-1}$), with load data recorded in newtons and converted to MPa using the briquette cross-sectional area. Each particle size treatment was replicated eight times ($n = 8$) to ensure statistical robustness. After pressing, the briquettes were oven-dried at 105 °C for 24 h to remove residual moisture, then stored in airtight containers for testing.

Physical and mechanical tests

Bulk density (ρ_b) was determined following the ASTM D2395-17 standard (ASTM International, 2017), using the ratio of briquette mass to geometric volume (cylindrical dimensions).

Compressive strength (σ_c) was measured using a Universal Testing Machine (UTM) with a 5 kN load cell at a crosshead speed of $1 \text{ mm} \cdot \text{min}^{-1}$. The maximum load at failure was divided by the cross-sectional area to obtain σ_c (MPa).

All tests were conducted at ambient conditions ($25 \pm 2^\circ\text{C}$, relative humidity $\approx 65\%$). Each reported value represents the mean $\pm$ standard deviation of eight replicates. Figure 1 illustrates the main equipment used in the preparation of candlenut shell briquettes.

The sieve shaker (a) was employed to classify the ground candlenut shell char into three particle size ranges 20, 40, and 60 mesh representing coarse, medium, and fine fractions. The hydraulic press (b) applied a uniform compaction pressure of 20 MPa to form cylindrical briquettes, ensuring consistent densification across treatments. The mold assembly (c) provided fixed dimensions (40 mm diameter $\times$ 30 mm height) for accurate volume measurement. This setup ensured reproducibility of briquette geometry, allowing direct comparison of physical and mechanical performance among particle size groups, as commonly required in biomass densification studies (Pang *et al.*, 2019; Setter and Oliveira, 2022).

Proximate analysis

Proximate analysis of the candlenut shell char briquettes was conducted in accordance with ASTM D3172-D3173 standards (ASTM International, 2013;). Moisture content was determined by oven-drying the samples at 105°C until constant mass was achieved. Ash content was measured by heating the samples in a muffle furnace at 750°C for 3 h. Volatile matter was determined by heating the samples at 950°C for 7 min under controlled conditions. Fixed carbon content was calculated by difference using the relation $\text{FC} = 100 - \text{MC} - \text{VM} - \text{Ash}$. All proximate analysis parameters were determined in triplicate for each particle size fraction to ensure data reliability.

Empirical model development

To establish quantitative relationships between particle size (x) and each response variable (ρ_b , σ_c , FC, VM, and Ash), two regression models were tested:

Logarithmic model:

- $Y = a + b \ln(x)$
- Power-law model: $\ln(Y) = a + b \ln(x)$ or equivalently $Y = e^a x^b$

Regression analysis was performed using Microsoft Excel (LINEST function). Model accuracy was evaluated using the coefficient of determination (R^2), root mean square error (RMSE), and 95% confidence intervals. In addition to goodness-of-fit metrics, statistical significance of the regression models was evaluated. Regression coefficients were tested using a two-tailed t -test at a 95% confidence level ($\alpha = 0.05$). All fitted models exhibited statistically significant slopes ($p < 0.05$), indicating that particle size had a significant effect on bulk density, compressive strength, ash content, volatile matter, and fixed carbon. The narrow confidence intervals further confirm the robustness of the estimated coefficients within the investigated particle size range.

Goodness-of-fit assessment and model limitations

Predicted values were compared against experimental results to verify the goodness of fit. The residuals were plotted against predicted values to detect systematic bias. The 95% confidence and prediction intervals were calculated to illustrate the uncertainty range (error band). Models with higher R^2 , indicating better variance explanation, and lower RMSE, reflecting reduced prediction error, were selected as optimal representations of each response variable.

It should be noted that the empirical models developed in this study are based on three discrete particle size levels. Therefore, the term “model validation” is used here in the context of goodness-of-fit assessment rather than independent predictive validation. The residual analysis and confidence intervals indicate that the selected models adequately describe the experimental data within the tested particle size range. However, extrapolation beyond this range should be performed with caution. Future studies incorporating a broader and more continuous particle size spectrum are recommended to enable full model validation and improve the generalizability of the proposed models.

Equipment and specifications

All instruments used in briquette production and testing are summarized in Table 1. The equipment includes devices for carbonization, grinding, particle-size classification, compaction, and mechanical and fuel property analyses. Calibration was performed prior to each test to ensure measurement accuracy and repeatability.

All equipment was operated under controlled laboratory conditions at ambient temperature ($27 \pm 2^\circ\text{C}$) and humidity ($65 \pm 5\%$). The hydraulic press and UTM were verified using standard load

calibration weights, while temperature instruments were cross-checked with a certified reference thermometer.

Results and Discussion

It should be clarified that moisture content was not treated as an experimental variable in this study. All briquettes were prepared using the same proportion of dry materials and added water, resulting in comparable initial moisture conditions across all particle size levels. Therefore, the observed variations in briquette properties are primarily attributed to particle size effects rather than moisture content differences.

Visual observation of briquettes

Figure 2 presents representative surface morphologies of candlenut shell briquettes produced at different particle sizes. Briquettes produced from coarse particles (20 mesh) exhibited rougher and more irregular surfaces, whereas those produced from finer particles (60 mesh) appeared smoother and more compact. These qualitative observations provide preliminary visual evidence of improved particle packing at smaller particle sizes and support the quantitative results discussed in subsequent sections.

Physical and mechanical properties

The integrated system shown in Figure 1 provides a controlled experimental framework for interpreting the effects of particle size on briquette structure and strength. Consistent particle size classification and uniform compaction pressure were maintained to minimize experimental variability, allowing the observed trends to be primarily attributed to particle size effects (Pang *et al.*, 2019; Setter and Oliveira, 2022).

The influence of particle size on briquette properties can be primarily explained by changes in packing behavior and inter-particle contact. Finer particles reduce internal void spaces, increase contact area, and enhance binder distribution, resulting in improved densification and bonding efficiency. This fundamental mechanism underlies the observed trends in mechanical strength, density, and fuel properties discussed in the following subsections.

Table 2 summarizes the average physical and mechanical properties of candlenut shell briquettes produced at different particle sizes.

Mechanical properties

Bulk density and compressive strength increased consistently with decreasing particle size. Bulk density increased from 0.68 g cm^{-3} at 0.84 mm particles to 0.75 g cm^{-3} at 0.25 mm, while compressive strength increased from 0.46 to 0.58 MPa. This trend follows the same densification mechanism described above, where finer particles promote more efficient packing and load transfer within the briquette matrix. As a result, the structural integrity of the briquettes improved by approximately 26.1% when particle size was reduced from coarse to fine fractions.

Fuel properties

Particle size also strongly influenced the fuel quality of the briquettes. As particle size decreased, ash content declined from 4.8% to 4.3% and volatile matter decreased from 27.5% to 26.2%, while fixed carbon increased from 61.4% to 64.7%. These trends reflect particle-size-dependent structural changes that affect devolatilization behavior during carbonization and are consistent with proximate-analysis-based observations reported in previous studies (Clauser *et al.*, 2021; Sengar *et al.*, 2012). Although lower ash and volatile matter contents may indirectly suggest improved fuel characteristics, no direct combustion or emission measurements were conducted in this study.

Overall, the results indicate that reducing particle size improves both mechanical durability and proximate fuel quality of candlenut shell briquettes under identical compaction conditions. Comparable particle-size-dependent trends in mechanical and proximate properties have been reported for char-based briquettes derived from tropical biomass residues, including Kesambi branches and torrefied biomass, where finer particles enhanced densification, compressive strength, and fixed carbon content (Abineno *et al.*, 2024; Dethan *et al.*, 2024).

Empirical model validation

Table 3 presents the empirical regression models describing the relationships between particle size and the mechanical and proximate properties of candlenut shell briquettes. All models exhibited high coefficients of determination ($R^2 \geq 0.95$) and low RMSE values (≤ 0.45), indicating that particle size is a dominant factor governing briquette properties within the investigated range.

Bulk density (ρ_b) increased with decreasing particle size following a logarithmic relationship ($\rho_b = 0.7369 - 0.06556 \ln x$, $R^2 = 0.993$) as shown in Figure 3. The logarithmic trend suggests diminishing densification gains at the finest particle size, indicating a threshold beyond which further size reduction yields marginal improvements. This behavior reflects the same particle size-controlled packing mechanism discussed earlier.

Kaliyan and Vance Morey (2009) Compressive strength (σ_c) followed a power-law relationship with particle size ($\sigma_c = 0.444x^{-0.190}$, $R^2 = 0.997$), increasing from 0.46 MPa at 0.84 mm to 0.58 MPa at 0.25 mm (Figure 4). The observed increase in compressive strength follows the same densification and bonding mechanism described above and is consistent with previous observations for agricultural residue briquettes (Mani *et al.*, 2006; Pang *et al.*, 2019).

Ash and volatile matter contents decreased as particle size decreased, described by power-law models with $R^2 \approx 0.96$. These trends are consistent with particle-size-related structural effects influencing devolatilization behavior, as commonly reported in proximate analysis studies (Demirbaş *et al.*, 2004; Sharma *et al.*, 2024).

Fixed carbon exhibited an inverse relationship with particle size ($FC = 61.48 x^{-0.072}$, $R^2 = 0.977$), indicating enhanced carbon retention at smaller particle size (Figure 5). This inverse relationship complements the observed reduction in volatile matter and reflects the interconnected nature of proximate components. It should be emphasized that the empirical models developed in this study are valid only within the tested particle size range (0.25–0.84 mm). Extrapolation beyond this range should be avoided.

Technical implications for briquette design

From a technical perspective, the results highlight the importance of particle size control in briquette manufacturing. Finer particle sizes (≤ 0.42 mm) improve packing efficiency and mechanical strength under constant compaction pressure, contributing to more robust briquettes with reduced susceptibility to handling damage. The accompanying changes in proximate

composition suggest that particle size optimization can also influence fuel characteristics, although direct combustion performance was not evaluated in this study.

The empirical relationships developed herein provide a practical basis for selecting suitable particle size ranges during feedstock preprocessing to balance mechanical durability and fuel quality in small-scale biomass briquetting systems.

Conclusions

This study examined how particle size influences the physical, mechanical, and proximate properties of candlenut shell briquettes produced under controlled compaction conditions. The results show a clear and consistent trend: reducing particle size from coarse (0.84 mm) to fine fractions (0.25 mm) is associated with higher bulk density and compressive strength, along with lower ash and volatile matter contents and higher fixed carbon.

These changes can be mainly attributed to particle-size-dependent packing behavior. Finer particles promote better inter-particle contact and more efficient densification under constant pressure, which in turn leads to stronger and more compact briquettes. The findings confirm that particle size is a critical preprocessing parameter in biomass briquetting and plays an important role in determining both mechanical integrity and material characteristics.

Empirical relationships were developed to describe the observed trends between particle size and briquette properties. These relationships should be regarded as preliminary and applicable only within the tested particle size range and experimental conditions. Their primary purpose is to support interpretation of the experimental results rather than to provide generalized predictive tools.

Some limitations of this study should be noted. Only three discrete particle size levels were investigated, moisture content was not treated as an independent variable, and fuel performance was evaluated indirectly through proximate analysis without direct combustion or emission testing. Future work should consider a wider and continuous particle size range, as well as direct combustion performance measurements, to further strengthen and extend the applicability of the findings.

Overall, this study provides a clearer understanding of the role of particle size in candlenut shell briquetting and offers a solid experimental basis for future research on biomass densification and solid biofuel development.

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Table 1. Equipment and specification used for briquette production and testing

Equipment	Model / Specification	Function
Muffle furnace	Nabertherm L5/11, 500°C ± 5°C	Carbonization of candlenut shells
Hydraulic press	Manual type, 2-ton capacity (≈ 20 MPa)	Briquette compaction
Universal testing machine (UTM)	Shimadzu AGS-X, 5 kN, crosshead speed = 1 mm min ⁻¹	Measurement of compressive strength
Sieve shaker	Retsch AS200, 20–60 mesh range	Particle size classification
Oven	Memmert UN55, 105°C ± 2°C	Drying of briquettes and samples
Analytical balance	A&D FX-300i, 0.001 g accuracy	Sample mass measurement
Caliper	Mitutoyo CD-15CP, ± 0.01 mm accuracy	Dimensional measurement
Thermocouple and data logger	Omega HH147U, K-type	Monitoring temperature during carbonization

Table 2. Average physical and mechanical properties of candlenut shell briquettes at different particle.

Particle size (mesh)	Mean particle size (mm)	Bulk density (g cm ⁻³)	Compressive strength (MPa)	Ash (%)	Volatile matter (%)	Fixed carbon (%)
20	0.84	0.68	0.46	4.8	27.5	61.4
40	0.42	0.73	0.52	4.5	26.8	62.9
60	0.25	0.75	0.58	4.3	26.2	64.7

Table 3. Empirical models for mechanical and fuel properties of candlenut shell briquettes.

Parameter	Model equation	R ²	RMSE	Trend	Interpretation
ρ_b (g cm ⁻³)	$\rho_b = 0.7369 - 0.06556 \ln(x)$	0.993	0.0027	↓ particle size → ↑ ρ_b	Finer particles improve packing and reduce voids, producing denser briquettes.
σ_c (MPa)	$\sigma_c = 0.444x^{-0.190}$	0.997	0.0025	↓ particle size → ↑ σ_c	Smaller particles enhance bonding and load transfer, yielding stronger briquettes.
Ash (%)	$Ash = 4.395x^{0.295}$	0.959	0.08	↓ particle size → ↓ Ash	Coarser particles retain more inorganics; finer powders burn more completely and cleanly.
VM (%)	$VM = 25.75x^{0.118}$	0.958	0.44	↓ particle size → ↓ VM	Coarser particles retain more volatiles; finer powders release volatiles more completely.
FC (%)	$FC = 61.48x^{-0.072}$	0.977	0.43	↓ particle size → ↑ FC	Smaller particles enhance carbonization and combustion stability, producing higher fixed carbon.



Figure 1. Equipment employed in briquette production: sieve shaker (left); hydraulic press (middle); briquette mold system (right).



Figure 2. Candle nut shell briquettes produced at different particle sizes: 20 mesh (left); 40 mesh (middle); 60 mesh (right).

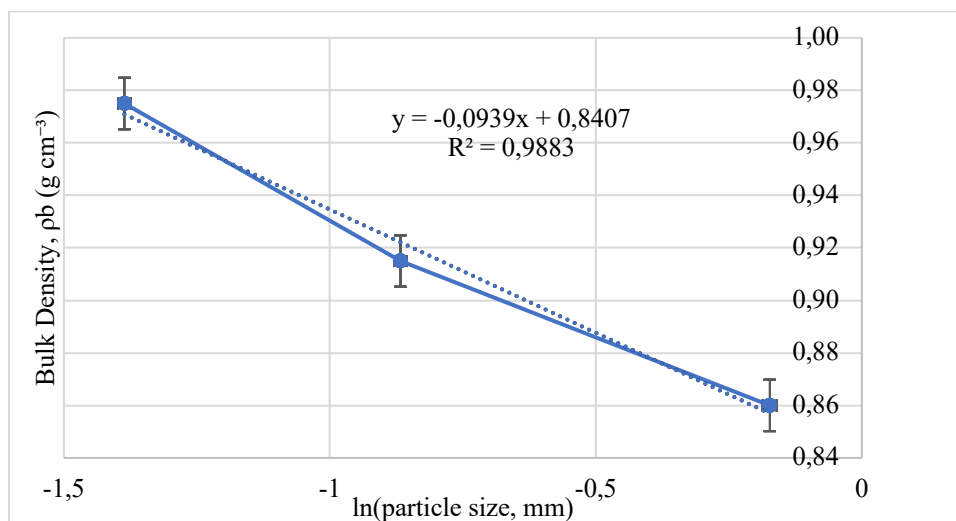


Figure 3. Relationship between $\ln$ (particle size) and bulk density (ρ_b) of candle nut shell briquettes showing logarithmic trend.

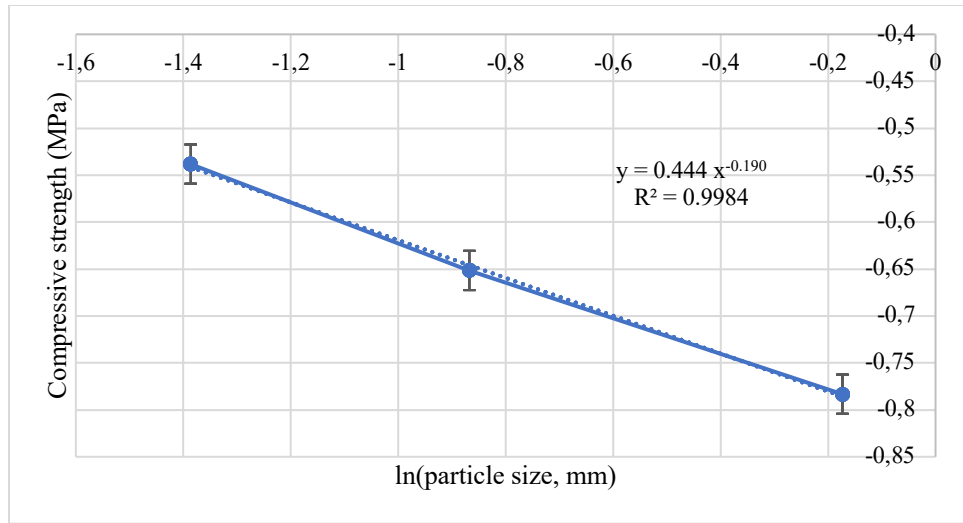


Figure 4. Relationship between ln (particle size) and compressive strength (σ_c) of candlenut shell briquettes.

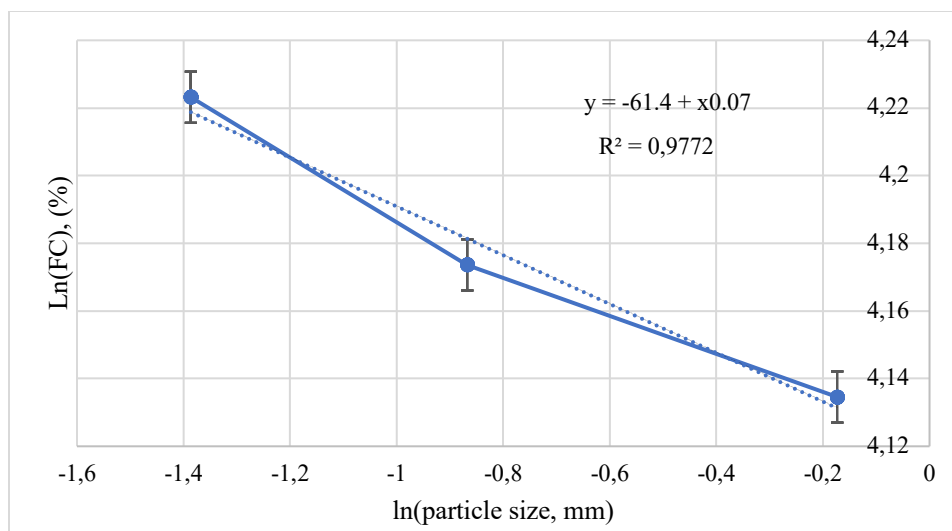


Figure 5. Relationship between ln(particle size) and fixed carbon (%) of candlenut shell briquettes.