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Optimization of Physicochemical Properties of Rice Husk, Groundnut, and Coconut Shells Composite Briquettes, Using Cow Bone Catalyst

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***Abstract*: The rising demand for sustainable and cost-effective energy alternatives has heightened research efforts in biomass briquetting, with a particular focus on optimizing physicochemical properties to enhance fuel quality. This study was carried out to enhance and optimize the physicochemical characteristics of composite briquettes produced from readily available agricultural residues. The main objective was to formulate an optimized composite briquette consisting of rice husk, groundnut shell, and coconut shell, augmented by a novel heterogeneous catalyst obtained from bovine bone. A highly pure calcium (II) oxide (CaO) catalyst was effectively synthesized via calcination of bovine bone at 900 °C, with XRD analysis indicating a prominent peak intensity of 1701.99 µ, thereby corroborating the presence and purity of CaO. Briquette formulation was guided by a Simplex Lattice Mixture Design (SLMD), executed with Design Expert 13.0, facilitating a systematic assessment of biomass component interactions and the identification of optimal mixture proportions. Optimization was conducted using dual criteria: reducing ash content and enhancing calorific value. The model determined an optimal mixture consisting of 20% rice fiber, 30% groundnut shell, and 50% coconut shell. This formulation yielded the most advantageous physicochemical properties, attaining a high calorific value of 19.21 MJ/kg and an exceptionally low ash content of 5.61%, reflecting substantial enhancements compared to non-optimized or catalyst-free briquettes. The improved performance affirms the catalytic efficacy of CaO in facilitating more efficient combustion properties and enhancing briquette quality. This study demonstrates that bovine bone serves as an effective, readily available, and economical precursor for the synthesis of high-purity heterogeneous catalysts, thereby supporting waste-to-wealth initiatives and sustainable biofuel generation. The optimized composite briquette not only complies with but also closely correlates with international standards for solid biofuels, providing a practical alternative for domestic and small-scale industrial energy use. Future research may investigate variations in catalyst dosage, emission profiles, and the feasibility of large-scale production to further promote the adoption of catalyst-enhanced biomass briquettes.**

***Keywords*: Biomass briquette, Cow bone catalyst, CaO, Physicochemical optimization, SLMD, Calorific value.**

1. Introduction

The current global energy matrix, dominated by the combustion of fossil fuels, is the primary driver of air pollution, impacting over 90% of the global population. This reliance is linked to an estimated 5.13 million premature deaths worldwide each year, emphasizing the critical need for a transition to sustainable, localized, and cleaner energy sources [1], [2].

Biomass, particularly in the form of agricultural residues, offers a renewable, carbon-neutral solution, simultaneously providing energy and addressing waste management concerns [3]. However, raw biomass typically exhibits low volumetric energy density, high moisture content, and poor handling characteristics, which limit its large-scale adoption. Biomass briquetting is an essential densification process that transforms loose, low-density residues into uniform, high-density fuel blocks [4], [5]. This process significantly improves storage, transportation, and combustion efficiency.

This study employs composite briquetting, mixing rice husk, groundnut shell, and coconut shell to strategically balance fuel properties. The novelty lies in the inclusion of a heterogeneous catalyst derived from cow bone. Cow bone is an abundant, low-cost waste source rich in calcium compounds. Through high-temperature treatment, it yields Calcium Oxide (CaO), a powerful catalyst known to accelerate char conversion and enhance combustion completeness, thus optimizing the fuel's calorific value and dramatically reducing ash residue [6].

The primary challenge in producing solid biofuels is achieving consistent quality, particularly minimizing negative properties like the ash content and maximizing the energy content [7]. Simply blending materials does not guarantee optimal performance. This research overcomes this challenge by using a Simplex Lattice Mixture Design (SLMD), a statistically robust method, to precisely model the relationship between the proportions of the three biomass components and the cow bone catalyst to predict the ideal formulation [8].

The specific objectives are to prepare and physically characterize the heterogeneous catalyst CaO from cow bone via high-temperature calcination, to apply a Simplex Lattice

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Mixture Design to systematically produce composite briquettes and determine the optimum component mixing ratio that minimizes ash content and maximizes calorific value, and to perform comprehensive physicochemical characterization of the optimized composite briquette and validate the results against international biofuel standards. Sangotayo et al. [22] assessed the thermal efficacy of activated carbon derived from locally sourced agricultural residues, offering a sustainable alternative to imported adsorbents. The findings endorse waste-to-resource conversion, advance the development of cost-effective environmental remediation materials, and strengthen local industrial capacity. It also promotes the utilization of renewable biomass, supporting the development of energy-efficient purification, filtration, and adsorption technologies. This study aimed to enhance and optimize the physicochemical features of composite briquettes produced from widely available agricultural leftovers. The main objective was to create an optimal composite briquette made from rice husk, groundnut shell, and coconut shell, augmented by an innovative heterogeneous catalyst sourced from bovine bone.

2. Methodology

The three biomass feedstocks were obtained from local agricultural processing units within the market areas of Gambari and Ogbomosho, Oyo State, Nigeria. They were cleaned, dried, and ground to a particle size less than 1.0 mm. The cow bone sample was similarly obtained from butcher shops at Atenda market, Ogbomosho (8.14447°N, 4.24273°E). The bones were cleaned to remove any remaining meat, fat, and other organic matter, then washed thoroughly to remove any residual organic material. The cow bone precursor was ground finer, to 0.31 µm, to maximize the surface area before catalyst preparation [9].

Cassava starch was used as a binder. The binder constitutes 10% of the weight of each sample, to ensure ash content is minimal, as cassava starch increases ash content [10]. The starch solution was prepared by dissolving the dried cassava in water and heating the mixture while stirring inside an aluminum plate until a consistent paste was formed.

A. Catalyst Synthesis and Characterization

The drying oven is used for removing moisture from the ground cow bone, as well as drying the briquettes. Drying the briquettes to a suitable moisture content below 10% ensures durability and efficient combustion [11]. A thermostat oven is used, manufactured by Gulfex Medical and Scientific England, and the model is DHG 9023A. The briquettes were arranged on trays within the drying oven. Using a drying oven improves the quality and consistency of the briquettes, reduces drying time compared to natural air drying, and ensures that the briquettes are free from mold and microbial contamination. The cow bone powder was dried at 105 °C for 24 hours. It was then subjected to calcination in a Thermos Scientific furnace at a precise temperature of 900°C for 4 hours. This ensures the thermal decomposition of $CaCO_3$ to yield the active Calcium Oxide (CaO) catalyst. XRD of the cow bones sample was performed on a MiniFlex-based generator X-ray diffractometer using Cu-ka radiation fitted over a range from 20 to 80°, with a step size of 0.04 at a scanning speed of 3° per minute. The compounds and their relative intensities were determined for the uncalcined state of the cow bone sample at 100°C and the calcined state at 900°C.

B. Experimental Design and Briquetting

A Simplex Lattice Mixture Design (SLMD) was executed using Design Expert 13.0. This approach is ideal for optimizing mixtures where the response is a function of the component proportions ($X_1 + X_2 + X_3$ = 100%) [12]. Each vertex of the simplex represents a pure component, while points along the edges or within the simplex represent mixtures in defined ratios. This proportion was used to determine the weights of each sample that make up the formed composite biomass briquettes as shown in Table 1.

The predetermined blend for each run was weighed, mixed, and compacted using a direct manual compression technique to form briquettes of uniform size. The briquettes were then air-dried. A total of 13 briquette samples were produced, with 3 from each sample and 10 composite briquettes from their mixture. The samples are in Figure 1.

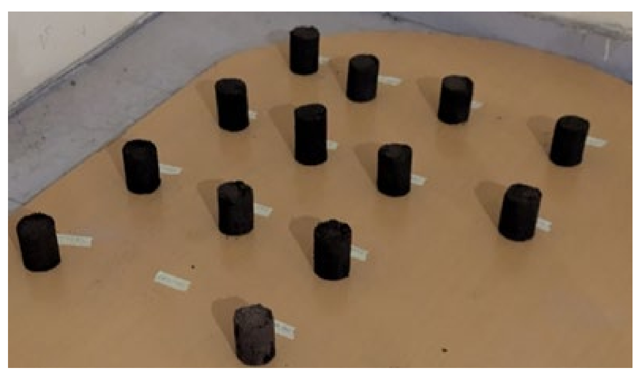


Fig. 1. Formed composite briquettes

Table 1
Proportion and weight of the biomass samples

Runs	Coconut Shell Proportion (%)	Coconut Shell Weight (grams)	Groundnut Shell Proportion (%)	Groundnut Shell Weight (grams)	Rice Husk Proportion (%)	Rice Husk Weight (grams)
1	42	75.6	32	57.6	26	46.8
2	40	72	40	72	20	36
3	45	81	30	54	25	45
4	50	90	30	54	20	36
5	40	72	35	63	25	45
6	44	79.2	33	59.4	23	41.4
7	40	72	30	54	30	54
8	47	84.6	31	55.8	22	39.6
9	41	73.8	37	66.6	22	39.6
10	45	81	35	63	20	36

C. Physicochemical Properties Characterization (Proximate Analysis)

1) Calorific Value

The calorific value of each briquette was determined using a bomb calorimeter. The heat of combustion was determined by burning a small sample (2g) of the briquette in an oxygen atmosphere, at a pressure of 3 MPa in the instrument to ensure perfect combustion. The released heat gave rise to a deflection in its galvanometer [13]. The same procedure was repeated for the reagent, benzoic acid of known specific calorific value. The difference in the deflection made by a sample of the briquette and the deflection made by a sample of benzoic acid was used to determine the calorific value.

Given that:

Mass of Benzoic Acid = M_b g

Calorific Value of 1 gramme of Benzoic Acid = 6.32kcal/g

Heat released from Benzoic Acid, C_b = 6.32 x M_1 Kcal (1)

Galvanometer deflection without sample = T1

Galvanometer deflection of Benzoic Acid = T_2 - T_1 (2)

Calibration constant, $k = \frac{6.32\ X\ W_1}{T_2 - T_1}$ (3)

This experiment is repeated five times, and the average value of k is determined.

Furthermore, the mass of the sample used = 0.25 g

Galvanometer deflection made by the sample = T_3

Thus, the galvanometer deflection of the sample = T_3 - T_1 (4)

Heat released by the sample = $(T_3 - T_1)$ y Kcal (5)

Calorific value of the sample = $\frac{(T_3 - T_1)\text{ y Kcal}}{0.25}$ Kcal/g (6)

Calorific value in MJ/Kg = $\frac{(T_3 - T_1)\text{ y Kcal}}{0.25}$ x 0.23866 MJ/kg (7)

Percentage by mass of the water content of the analytical sample for general analysis determined in accordance with ISO 18134-3.

2) Moisture Content

The moisture content was determined by placing 2 grammes of the sample in a crucible. The crucible with the sample was then transferred into the oven set at 105°C to dry for 24 hours [14]. The crucible with the sample was removed from the oven and transferred to a desiccator, then cooled for ten minutes and weighed.

Percentage Moisture Content, $PMC = \frac{W_1 - W_2}{W_2}$ (8)

W_1 is the initial weight of the briquette sample

W_2 is the final weight

3) Volatile Matter

The briquettes' percentage volatile matter content was determined using a Lenton furnace. The residue of the dry sample from moisture content determination was preheated at 300°C for 2 hours to drive off the volatile constituents of the sample. The resulting sample was further heated at 470 ºC for 2 hrs, to remove the volatile matter, just before the sample turned to ashes, and then cooled in a desiccator [15]. The crucible with a known weight and its content was weighed, and the percentage of weight loss, which is the percentage volatile matter, was calculated using the equation below.

Volatile matter (%) $= \frac{\text{Final Weight}}{\text{original weight}}$ x 100 (9)

4) Density

Density is the mass per unit volume of the briquette sample and is calculated using:

Density = $\frac{\text{Dry Mass of Briquette Volume of Briquette}}{\text{Volume of Briquette}}$ (10)

Dry mass is the mass of the briquette after drying.

5) Ash Content

Ash content of the sample briquettes was determined using a furnace residue, and they were heated in a furnace ignited at 59 $°C0°C$,-, for 2hrs and transferred into a desiccator to cool down the material that was turned into white ash and weighed. The same process was repeated three times at an interval until the weight is constant. The weight was recorded as the final weight of the ash [15]. The percentage ash content was calculated using the equation below:

Ash content (%) = $\frac{Weight\ of\ Ash}{Initial\ weight\ of\ dry\ sample}$ x 100 (11)

6) Fixed Carbon

Fixed carbon is described as the percentage of the briquette that consists of non-volatile carbon; this is calculated by parameters determined earlier [16]. According to ASTM method D3172-07a, the percentage weight of fixed carbon is given as:

PC = 100 – (MC + VM + AS). (12)

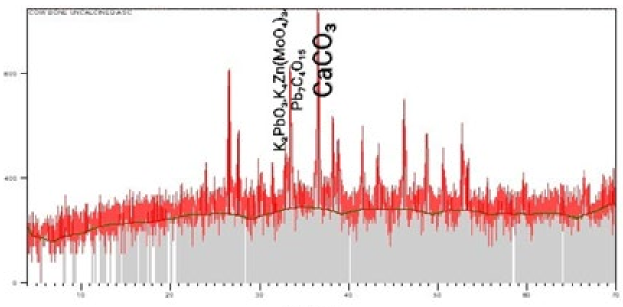
MC = percentage moisture content, VM = percentage volatile matter, AS = percentage ash content.

3. Results and Discussion

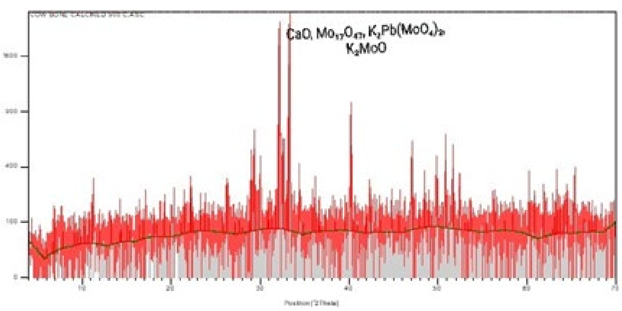
A. Catalyst Characterization

The X-Ray Diffraction (XRD) analysis provided conclusive evidence of the phase change. Initially, the uncalcined pattern was dominated at the peak by Aragonite ($CaCO_3$). The calcined pattern exhibited a sharp, intense peak at $2\theta = 32.228°$ (Intensity 2277.65 U), confirming the presence of pure Calcium Oxide (CaO). The effective conversion of the cow bone sample to the active catalyst is visually represented in Figure 2 (a) and (b).

The high concentration of calcium oxide observed at such an elevated temperature (900°C) can be attributed to the complete decomposition of organic molecules during calcination. This process leaves behind stable inorganic constituents, which become oxidized, resulting in an increased presence of compounds such as calcium oxide in the cow bones sample [17].



(a)



(b)

Fig. 2. Cow Bone Compound Intensity-Count (U) vs Position (2θ) (a) Uncalcined (b) Calcined

B. Optimization of the Biomass Composite Briquettes

A total number of 10 composite briquettes were produced, and for optimization, the ash content and calorific value were utilized, owing to their critical roles in defining both the energy potential of the briquettes. Subsequent analyses of other key physicochemical parameters were conducted on the optimized briquette sample.

1) Ash content

The results obtained from the characterization of the individual residue of coconut shell, groundnut shell, and rice husk (for ash content) were 3.78%, 3.87% and 13.13% as presented in Table 1. The relatively high ash content in rice is explained by its high silica content [18]. The values are consistent with previous authors [19], [4], [20], [21].

The results obtained from the 10 composite briquettes are presented in Table 3.0. The lowest ash content from the experimental data is 5.58, at 20% rice husk, 40 % groundnut shell, and 40 % coconut shell, while the highest ash content is 6.53, at 30% rice husk, 30% groundnut shell, and 40% coconut shell. The coefficient of determination R^2 of the ash content (AC) is 0.9631, indicating a strong correlation; the predicted R^2 of 0.9372 is in reasonable agreement with the adjusted R^2 of 0.9525; the adequate precision is 25.637, which indicates a high signal-to-noise ratio, and confirms that the model is useful for the design space as presented in Table 4.

The model is linear, as the p-value for linear is < 0.0001 as shown in Table 5. The relatively high model F-value of 91.24 as shown in Table 6, implies the model is significant and that there is only a 0.01% chance that the Model F-value could occur due to noise.

The coded equation is given as:

$$\text{Ash Content} = +6.49A + 5.60B + 5.61C \quad (13)$$

Table 2
Experimental data of characterized pure briquette samples

Briquette Samples	Ash Content (%)	Calorific Value (MJ/Kg)	Moisture Content (%)	Volatile Matter (%)	Carbon (%)	Fixed Carbon (%)	Sulfur (%)
Coconut shell	3.78	21.5	7.32	64.5	51.12	24.4	0.15
Groundnut shell	3.87	19.59	10.53	63.3	45.4	22.3	0.395
Rice husk	13.13	13.02	9.89	60.95	46.83	16.03	0.535

Table 3
Experimental data of characterized composite briquette samples

Run	Component A: Rice Husk	Component B: Groundnut Shell	Component C: Coconut Shell	Response 1 Ash Content (%)	Response 7 Calorific Value (MJ/Kg)
1	26	32	42	6.02	18.76
2	20	40	40	5.58	19.08
3	25	30	45	6.03	18.89
4	20	30	50	5.61	19.21
5	25	35	40	6.04	18.98
6	23	33	44	5.98	19.1
7	30	30	40	6.53	18.57
8	22	31	47	5.77	19.04
9	22	37	41	5.79	18.92
10	20	35	45	5.6	19.17

Table 4
Ash Content's (a) Fit statistics (b) Fit summary (c) Sequential model sum of squares

(a)

Std. Dev.	Mean	C.V. %	R²	Adjusted R²	Predicted R²	Adeq. Precision
0.0633	5.9	1.07	0.9631	0.9525	0.9372	25.6371

(b)

Source	Sequential p-value	Lack of Fit p value	Adjusted R²	Predicted R²	
Linear	< 0.0001		0.9525	0.9372	Suggested
Quadratic	0.9512		0.9231	0.4143	
Special Cubic	0.6439		0.9057	-0.0219	
Cubic					
Sp Quartic vs Quadratic	0.585		0.9296	-8.924	
Quartic vs Cubic					Aliased
Quartic vs Sp Quartic					Aliased

(c)

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Mean vs Total	347.51	1	347.51			
Linear vs Mean	0.7314	2	0.3657	91.24	< 0.0001	Suggested
Quadratic vs Linear	0.0021	3	0.0007	0.1077	0.9512	
Sp Cubic vs Quadratic	0.0021	1	0.0021	0.2622	0.6439	
Cubic vs Sp Cubic	0.0239	3	0.008			
Quartic vs Cubic	0	0				Aliased
Sp Quartic vs Quadratic	0.02	3	0.0067	1.12	0.585	
Quartic vs Sp Quartic	0.0059	1	0.0059			Aliased
Total	**348.27**	**10**	**34.83**			

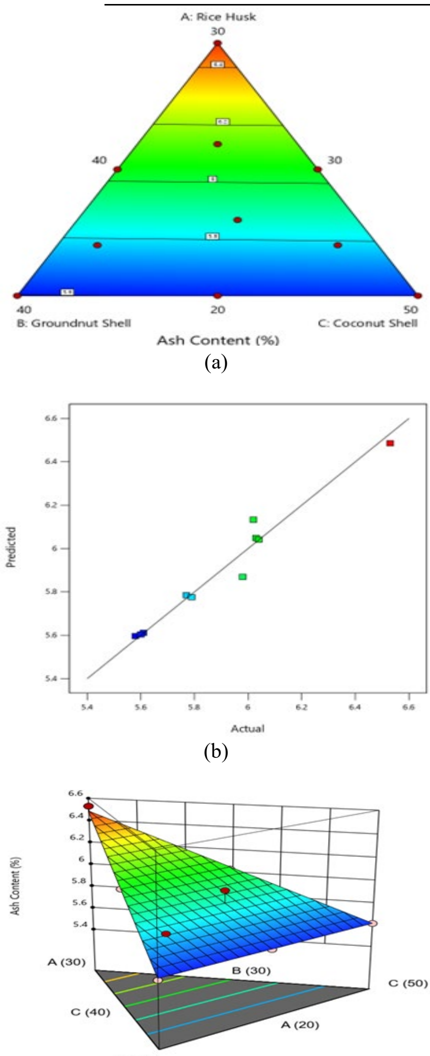


Fig. 3. Graph for calorific value: (a) Contour model, (b) Predicted vs Actual model, (c) 3D surface model

2) Calorific Value

Subsequently, the results obtained for calorific values for the coconut shell, groundnut shell, and rice husk are 21.5, 19.59, and 13.02, as seen earlier in Table 1. It is observed that the ash content and calorific value have an inverse relationship, as the increase in ash content results in a decrease in calorific value and vice versa, as seen in Figure 4.0d [22].

The highest calorific value of the composite briquette is 19.21 MJ/kg, where the proportion of rice husk, coconut shell, and groundnut shell is 20%, 30% and 50% (Table 3). This high calorific value is due to the large proportion of coconut shell and groundnut shell in the mixture. The lowest calorific value of 18.57 MJ/kg is obtainable at 30% rice husk, 30% groundnut shell, and 40% coconut shell.

3) Statistical analysis for the composite briquettes – Calorific Value

The coefficient of determination R^2 of the calorific value (CV) is 0.8742, indicating a strong correlation; the predicted R^2 of 0.7948 is in reasonable agreement with the adjusted R^2 of 0.8383; the adequate precision is 14.1389, which indicates a high signal-to-noise ratio, and confirms that the model is useful for the design space (Table 5a). The model is linear, as the p-value for linear is < 0.0007 (Table 5b). The relatively high model F-value of 24.32 implies the model is significant, and the chance of it occurring due to noise is 0.07% (Table 5c).

The coded equation is given as:

$$\text{Calorific Value} = 18.60A + 19.10B + 19.21C \quad (14)$$

C. Numerical Validation Studies of the Composite Catalytic Briquette Produce

The numerically optimized selected results by the software Design Expert version 13.0 were based on the highest desirability of 1.00 [9]. In this study, the optimum values suggested at desirability of 1 for ash content and calorific value were 5.642 % and 19.184 MJ/kg (Figure 4 and 5).

Table 5
Ash content's (a) Fit statistics (b) Fit summary (c) Sequential model sum of squares

(a)

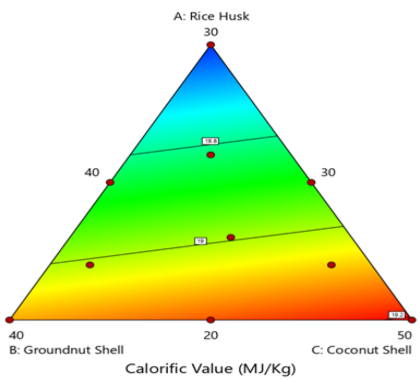
Std. Dev.	Mean	C.V. %	R²	Adjusted R²	Predicted R²	Adeq Precision
0.0787	18.97	0.4148	0.8742	0.8383	0.7948	14.1389

(b)

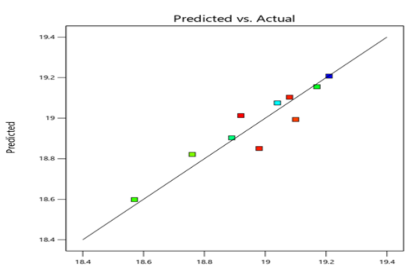
Source	Sequential p-value	Lack of Fit p-value	Adjusted R²	Predicted R²	
Linear	0.0007		0.8383	0.7948	Suggested
Quadratic	0.779		0.7786	0.33	
Special Cubic	0.8555		0.7086	-0.356	
Cubic					
Sp Quartic vs Quadratic	0.8993		0.4269	-79.7828	
Quartic vs Cubic					Aliased
Quartic vs Sp Quartic					Aliased

(c)

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Mean vs Total	347.51	1	347.51			
Linear vs Mean	0.7314	2	0.3657	91.24	< 0.0001	Suggested
Quadratic vs Linear	0.0021	3	0.0007	0.1077	0.9512	
Sp Cubic vs Quadratic	0.0021	1	0.0021	0.2622	0.6439	
Cubic vs Sp Cubic	0.0239	3	0.008			
Quartic vs Cubic	0	0				Aliased
Sp Quartic vs Quadratic	0.02	3	0.0067	1.12	0.585	
Quartic vs Sp Quartic	0.0059	1	0.0059			Aliased
Residual	0	0				
Total	**348.27**	**10**	**34.83**			



(a)



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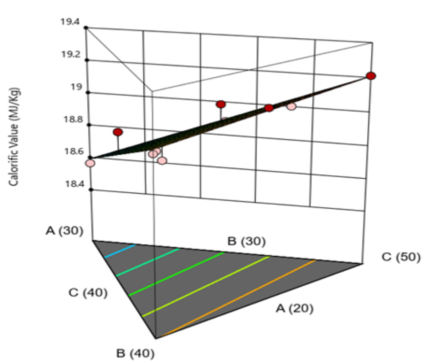

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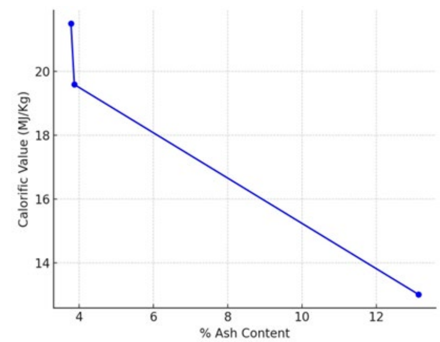


(d)

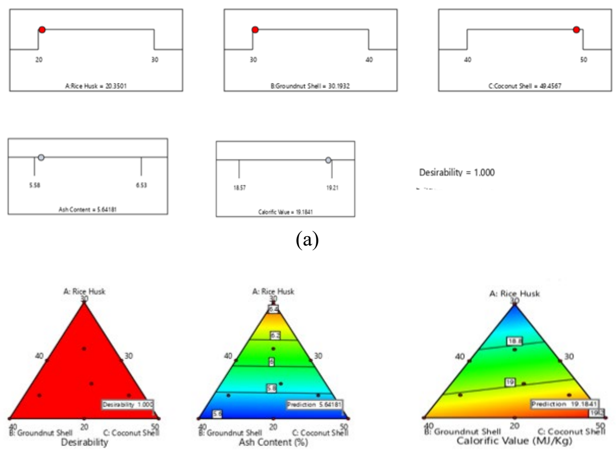
Fig. 4. Graph for calorific value: (a) Contour model, (b) Predicted vs Actual model, (c) 3D Surface model, (d) vs Ash content

Calculation suggested by the design software 13.0 was used to produce the briquettes. These briquettes produced were characterized, and the results obtained for ash contents and calorific value were 5.65 % and 19.201 MJ/kg. The validation of the numerically optimized results, selected by the software, was carried out by investigating or determining the percentage error between the predicted and actual values. The percentage difference between the actual and the selected values of the briquette produced for ash content and calorific value was 0.1416 % and 0.0885 %.

A commercially obtained calcium oxide was used to produce composite briquettes of rice husk, coconut shell, and groundnut shell. This further validates the efficiency and economic importance of the heterogeneous catalyst developed from agricultural residue. The results obtained for ash content and calorific value were 5.652, 19.211, obtained by the optimum conditions as suggested by the software. The results indicate that the cow bone heterogeneous catalyst developed produces the same effect as the commercially obtained calcium oxide catalyst.

Table 6
Validation of numerical solution of optimized briquettes

Parameter	Numerical - Optimized Data	Experimental Data - Heterogeneous Catalyst.	Percentage Error - Heterogeneous (%)	Experimental Data - Commercial Catalyst.	Percentage Error (Commercial) (%)
Ash Content (%)	5.642	5.65	0.1416	5.652	0.0354
Calorific Value (MJ/kg)	19.184	19.201	0.0885	19.211	0.0521



(a)

(b)

Fig. 5. All Responses: (a) Numerical solution ramps (b) Numerical solution contour

D. *Physicochemical Properties of the Optimized Briquettes*

The physicochemical properties of the optimized briquettes determined earlier at 20% rice husk, 30 % coconut shell, 50 % groundnut shell are given in term of ash content, moisture content volatile matter, carbon fixed, carbon, sulphur and calorific value as 5.61 %, 8.71%, 63.55%, 48.55%, 22.07%, 0.302% and 19.201 MJ/kg respectively. The properties have shown improvement in the properties of the individual briquettes, with the moisture content of 8.71% less than the moisture content of groundnut shell and rice husk, as seen earlier (Table 6.0). The sulphur content is also relatively low compared to the high values in groundnut shell and rice husk.

4. Conclusion

This research effectively demonstrated the manufacturing, optimization, and validation of composite briquettes composed of rice husk, groundnut shell, and coconut shell, augmented with a heterogeneous catalyst obtained from bovine bone. XRD analysis verified the successful conversion of cow bone into high-purity calcium oxide (CaO) at 900 °C, demonstrating complete organic component decomposition and exhibiting pronounced crystallinity. The catalytic efficacy of cow-bone-derived CaO was comparable to that of commercial CaO, confirming its potential as an economical and sustainable catalyst.

Optimization conducted with Design Expert software determined the optimal mixture comprising 20% rice husk, 30% groundnut shell, and 50% coconut shell, resulting in a low ash content of 5.61% and a calorific value of 19.21 MJ/kg. Statistical modeling for ash content and calorific value yielded high R^2 values, significant model F-values, and satisfactory precision, confirming the robustness of the predictive models. Numerical optimization further evidenced a strong concordance between predicted and experimental outcomes, with percentage discrepancies under 0.2%, thereby validating the robustness of the model.

The physicochemical assessment of the optimized composite briquette demonstrated compliance with appropriate standards, evidenced by decreased moisture content, enhanced volatile matter levels, and reduced sulfur concentration relative to individual residues. These findings collectively verify that agricultural residues, supplemented with cow-bone-derived CaO, can generate high-quality, energy-efficient briquettes appropriate for both domestic and industrial use.

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