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Original Article

Characterization of Selected Agricultural Waste Residues for Briquette Production

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Abstract

The global demand for renewable energy solutions has increased significant interest in utilizing waste biomass materials as a promising alternative to fossil fuels. In this study, briquettes were produced from carbonized agricultural wastes (coconut husk, corn cobs, and cowpea shells) using starch and clay as binders. Carbonization was performed by conventional heating at temperatures ranging from 300 to 500°C and residence times of 45-120 min. Agricultural residues and biochar were characterized by ultimate and proximate analyses. The characteristics of the briquette samples were assessed in terms of burning rate, compressive strength, shatter index, and calorific value. The factors - biochar type, binder type, and ratio - were used to evaluate the effects on the physiochemical properties of briquettes. Three different biochar-to-binder ratios were investigated: 70:30, 80:20, and 90:10. The results demonstrated that the briquettes exhibited a burning rate from 0.65 to 3.22 g/min, a time to boil water from 7.62 min to 23.56 min, a compressive strength from 11.38 to 39.66 N/mm^2, a shatter index from 77.45 - 99.81 %, and calorific values from 14.71 to 24.02 MJ/kg. Notably, briquettes with a 70:30 ratio achieved the highest shatter index of 99.81% and compressive strength of 39.66 N/mm^2, while those with a 90:10 ratio recorded the highest burning rate of 3.33 g/min, the quickest time to boil water of 7.62 min, and the highest calorific value of 24.02 MJ/kg. This study contributes to the utilization of agricultural waste residues, accentuating their potential as alternative fuel sources for local energy applications.

Categories: Renewable Energy in Agriculture, Environmental and Sustainable Engineering, Environmental Engineering

Keywords: biomass, coconut husk, corncob, cowpea shell, calorific value

Introduction

The need to explore green energy alternatives has never been more pressing than it is today. One promising area of research in this field is converting agricultural waste into useful energy sources. Every season of harvest leaves behind many non-edible parts of crops, like stalks, husks, shells, and fronds, which are collectively referred to as agricultural residue. In Ghana, these are from some of the most important crops, such as maize, cassava, yams, plantain, rice, cocoa pods, and oil palm. These residues have theoretical and technical potential of 28.92 million tonnes and 17.24 million tonnes, respectively [1]. However, this is not a problem that is unique to Ghana; globally, crop residue from some of the most important food crops like maize, rice, wheat, barley, soybean, and sugarcane is responsible for 3.7 Pg of dry matter annually, with maize, rice, and wheat alone responsible for more than 75% of this total quantity [2]. If not properly managed, this large quantity of waste can pose a significant threat. Improper management of waste materials such as rice straw, corn cobs, and sugarcane bagasse through open burning contributes to poor air quality [3]. Ineffective decomposition of waste materials also contributes to soil and water degradation, thereby causing ecological damage [4].

The lack of effective waste utilization represents not only a missed opportunity but also a factor contributing to ecological harm. This is where the potential of conversion technologies becomes relevant. Notably, advancements in thermochemical conversion methods, such as pyrolysis, have enabled the conversion of field residues into usable biochar [5,6]. One of the best uses of these processes, in terms of applicability and availability, is in the production of briquettes, which are solid blocks of compacted carbonized materials, including charcoal, wood shavings, forest waste, and agricultural waste, used in a variety of energy-related applications [7]. It is worth noting that, unlike traditional fuels such as wood and charcoal, agricultural waste-based briquettes are a more environmentally friendly option, thus proving to be a more beneficial option for a variety of uses [8]. Various research avenues have been explored to improve briquette production. These include investigations into the utilization of biochar and various types of binders such as molasses, cow dung, and horse dung to determine their effects on briquette production [9]. Others include investigations into the production of briquettes from biochar derived from water hyacinth, molasses, and Ethiopian soil in a ratio of 6:4:0 in the absence of organic materials [10]. In addition, environmentally friendly non-edible materials such as cassava peel starch, giant taro starch, and pine resin have been used in conjunction with waste coconut shell charcoal and cinnamon sawdust [11]. In terms of characterization, research has been conducted by Sunnu et al. [12] on charred briquettes from rice husks, corn cobs, and sugarcane bagasse. Others include investigations by Oyebamiji et al. [13] on briquettes from agricultural waste such as corn cob, cowpea shells, rice bran, sawdust, corn straw, dry leaf, and sugarcane peel. Falemara et al. [14] also conducted an assessment of the physical and combustion properties of briquettes from cowpea shells and corn cobs in addition to other biomass materials. However, there is a literature gap that integrates the characterization of the feedstock, the biochar, and the briquettes into a single framework. This study aimed to characterize selected agricultural residues for briquette production. Specifically, it focused on the characterization of agricultural waste residues and biochar, determined the physiochemical properties of produced briquettes, and evaluated the effects of binder type, biochar type, and mixing ratio on the briquettes.

Materials And Methods

Source of materials and preparation

The agricultural residues selected for this study were corncobs, coconut husks, and cowpea shells. Figure *1* shows the agricultural waste residues used for briquette production. Coconut husk was sourced from Tech Junction located in the Oforikrom District (6°41′N, 1°34′W) and was obtained from the fibrous husk of coconut residues through mechanical separation. Corncob and cowpea shell were collected from farms in Ejura within the Sekyedumase Municipal District (7°23′N, 1°22′W). The selected residues were subsequently sun-dried until a moisture content of 15% was achieved, with periodic sampling conducted to ensure uniform moisture reduction. This drying protocol aligns with international crop residue handling and grain storage recommendations [15].

Clay and starch were selected as binders for briquette production. Clay was obtained from farmlands on the Kwame Nkrumah University of Science and Technology (KNUST) campus in the Oforikrom district (6°41′N, 1°34′W), while starch was sourced from Kejetia Market located in the Central Business District, Kumasi (6°41′55″N, 1°37′09″W). The agricultural residues and selected binders were sorted and stored in ziplock bags. Briquette compaction was carried out using a screw press.

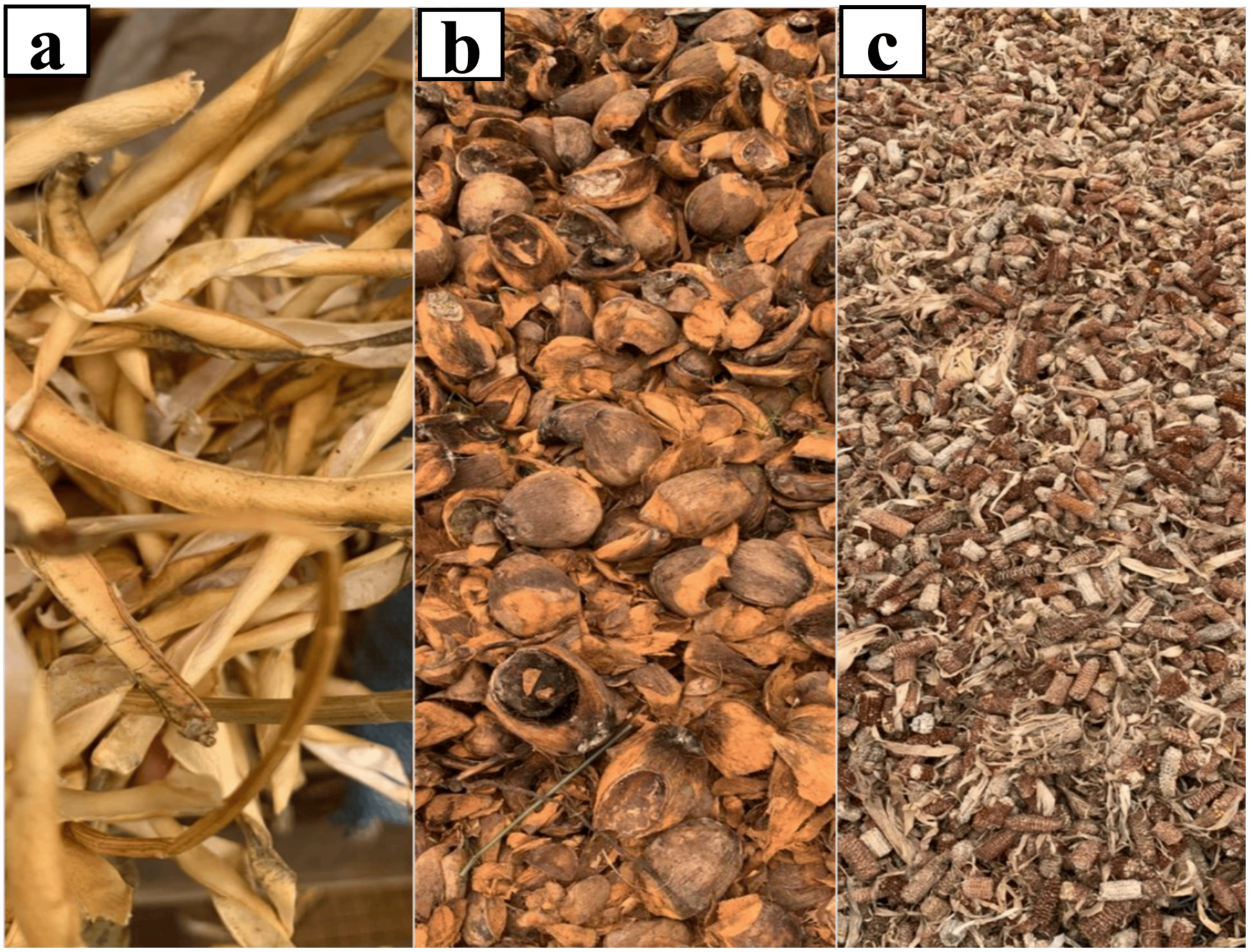

FIGURE 1: Agricultural waste residues. (a) cowpea shell; (b) coconut husk; (c) corn cob

Char production

The dried agricultural residues, namely corncob, cowpea shells, and coconut husks, were transformed into biochar through slow pyrolysis. The slow pyrolysis process was carried out by loading 2-4 kg of each residue into the kiln. The temperatures, 300°C-500°C, were monitored at regular intervals and gradually increased to the pyrolysis range. The residence time varied according to the type of residue. The cowpea shell residue took an average residence time of 45 min, the corncob took about 1 h and 30 min, and the coconut husk took an average residence time of 1 h and 45 min. The dried char was then crushed and sieved to ensure that it had the required particle size to enhance its surface area and interaction during briquetting. The size was regulated using sieves with apertures ranging from 0.42 mm to 0.25 mm, and all the materials were within this range. Achieving this fine particle size distribution is essential for maximizing density and enhancing briquette strength, as reported by Abineno et al. [16]. Also, briquettes produced from finer particles (below 1.2 mm) consistently demonstrate greater density and compressive strength than those fabricated from coarser fractions from the same source [16]. Biochar and binder mixtures were adjusted to 12% (w.b.) using an oven before compaction. This moisture content falls within the range used in literature [17].

Proximate analysis

Agricultural residues and biochar were characterized by proximate analysis namely: Volatile matter, Fixed carbon, Moisture content, and Ash content. Moisture content was measured in accordance with ASTM standard procedures [18]. A total of 1 g of sample was weighed in a pre-dried crucible. It was then dried in an oven from 105°C to 110°C to constant mass for 2 h. The sample was cooled in a desiccator and reweighed. Equation (1) was used to calculate moisture content:

$$\%M.C = \frac{M_i - M_d}{M_i} \times 100 \tag{1}$$

Where M_i = Initial mass of sample and M_d = Dry mass of sample

Ash content was determined following the method described by Tanko et al. [19]. The procedure involved weighing the initial sample, then subjecting it to combustion in a furnace at 550°C for 4 h to achieve complete ashing. Both the initial and final ash weights were recorded using a digital balance, and the percentage ash content (%AC) was calculated using Equation (2):

$$\%A.C = \frac{W_f}{W_i} \times 100 \tag{2}$$

Where W_i = Weight of the ash residue and W_f = Weight of original sample

Volatile matter content was measured according to ASTM D3175-20 [20]. A total of 1 g of sample was weighed in a silica crucible. It was then placed in a furnace, heated to 900 ± 5°C and held for exactly 7 min. The sample was then cooled in a desiccator and then reweighed. The same procedure was used for both agricultural residue and biochar. Equation (3) was used to calculate the volatile matter of sample:

$$\%V.M = \frac{W_i - W_f}{W_i} \times 100 \tag{3}$$

Where W_i = the initial weight of the sample and W_f = the final weight of the sample

Fixed carbon was obtained by subtracting the total values of the parameters from 100% using Equation (4):

$$\%F.C = 100\% - (\%M.C + \%V.M + \%A.C) \tag{4}$$

Ultimate analysis

Elemental analysis of carbon, nitrogen, hydrogen, and sulfur was performed using the Eurovector CHNS analyzer in accordance with ASTM standards [21,22]. The process involved weighing 2 mg into a tin sample, which is then combusted in a furnace at high temperature around 1000°C in the presence of oxygen. The combustion products (CO_2, H_2O, N_2, SO_2) were separated and detected using gas chromatography. All elemental components were calculated by the analyzer. Equation (5) was used to calculate the oxygen content of sample:

$$\%O = 100\% - (\%C + \%N + \%S + \%H) \tag{5}$$

Briquetting

Bello et al. [23] conducted a comparative analysis of hydraulic piston press and screw press methods, finding that the screw press produced briquettes with enhanced physical properties and superior combustion characteristics. Briquettes produced using the screw press method attained higher densities, ranging from 1 g/cm^3 to 1.4 g/cm^3, thereby increasing their overall energy density [23]. Briquettes produced using a screw press had a height of 30 mm and a diameter of 50 mm. The production of briquettes used a screw press with a die of 50 mm diameter, which is the most standard for lab-scale and commercial boilers [24,25]. After compaction, the briquettes were air-dried at ambient temperature for 48 h and then oven-dried at 105°C for 20 h to achieve an optimum moisture content of 8% as reported by Abdel Aal et al. [26].

The resulting briquettes were cylindrical in shape, with a diameter of 50 mm and a height of 30 mm, in accordance with the specifications recommended by Obi et al. [27] for achieving optimal combustion characteristics. The briquetting process from the raw materials collection to compaction and densification is as seen in Figure *2*.

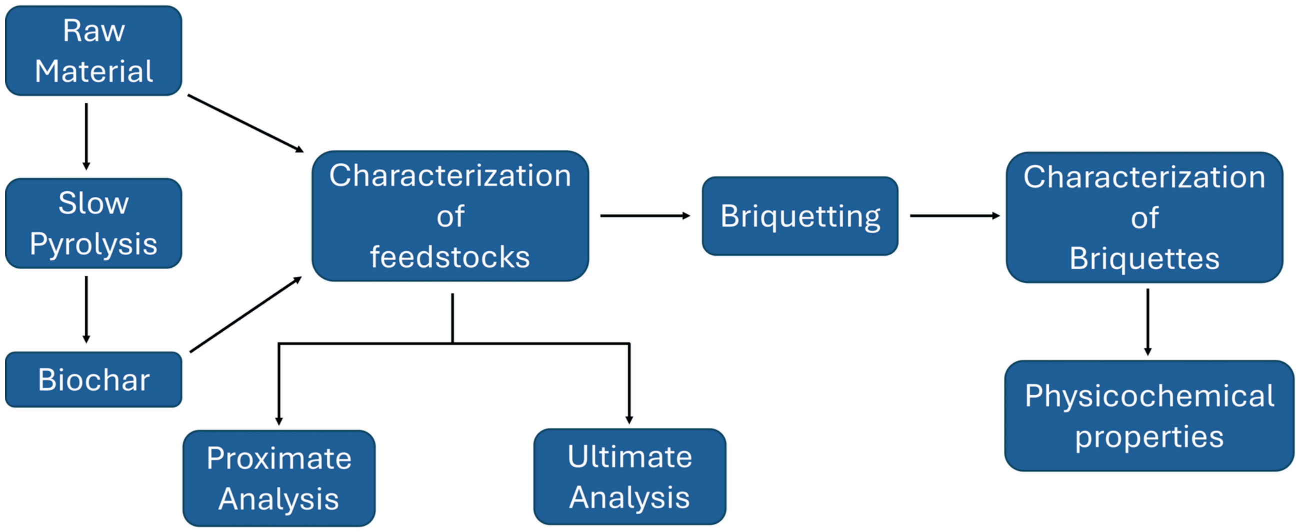

FIGURE 2: Schematic diagram of the characterization process

Physicochemical properties of briquettes

Calorific value was determined using an oxygen bomb calorimeter. A sample of 1 g was used, and the value was obtained from the temperature difference between ignition of the sample and its completion of burning, considering the calorimetric coefficient of the calorimetric equipment. Compressive strength was evaluated using a Universal Testing Machine with a load capacity of 1000 kN, following the ASTM D2166-85 standard [28]. Each briquette sample was placed flat on the horizontal metal plate of the machine, and the upper crosshead was lowered at a controlled rate until the sample fractured. Compressive strength was then calculated using equation (6).

$$\sigma = \frac{P}{A} \tag{6}$$

Where P = load at failure and A = the cross-sectional area of the briquette

The shatter index of briquettes was determined in accordance with ASTM D440-86 [29]. Each sample was initially weighed, then subjected to three consecutive drops from a fixed height of 2 m. Following each drop, the briquette was screened using a 2.5 mm sieve shaker, and the weight of particles retained in the sieve was recorded, as described by Tanko et al. [19]. The shatter index (K) was calculated using the corresponding Equation (7).

$$\%K = \frac{S_1}{S_2} \times 100 \tag{7}$$

Where S_1 = weight of the briquette after the drop and S_2 = weight of the briquette before the drop

To conduct the water boiling test (WBT), a stainless steel cup containing a fixed volume of water (100 cm^3) at room temperature of 25°C was placed on a briquette stove loaded with 100 g briquette samples as described in the procedure by Odusote et al. [30]. A stopwatch was used to record the duration from ignition to complete combustion of the briquettes. A thermometer was used to measure the temperature of the water when it reached the boiling point (100°C).

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The burning rate is defined as the rate at which a given mass of bio-coal briquettes is consumed until only ash remains [31]. A weighed mass of 200 g was placed in a stove and burnt to ash. The burning rate was determined using Equation (8):

$$Burning\ rate\left(\frac{g}{\min}\right) = \frac{W_i - W_f}{T} \tag{8}$$

Where W_i = Weight of original briquette samples, W_f = Weight of briquette samples after complete burning and, T = time.

Experimental design

Table 1 shows the treatments for briquette production. The experiment employed a 3 × 2 × 3 factorial design. The factors and their respective levels were biochar type at three levels (Cowpea shell char, cocopeat char, corncob char), binder type at two levels (starch and clay), and ratios at three levels (90:10, 80:20, and 70:30). The reason for the selected ratios was based on recent literature. Lomunyak et al. [32] examined the use of Acacia charcoal dust with binder proportions of 10%, 15%, 20%, 25% and 30%. Also, Ayaa et al. [33] investigated five different nanocomposite briquette formulations with biochar-to-binder ratios of 90:10, 80:20, 70:30, 60:40, and 50:50. Additionally, Parthipan et al. [34] explored briquettes produced from composite dry fallen leaves powder mixed with starch binder in ratios of (100:10, 90:20, 80:20, 70:30, 60:40, and 50:50). Each experimental trial was carried out three times (n = 3) under equal experimental conditions for purposes of achieving reproducibility. For each treatment, the average value of the three trials was taken into account for analysis. All factors resulted in 18 treatment combinations (n = 18).

Treatments	Biochar type	Binder type	Biochar: Binder Ratio
T1	Corncob char	Clay	90:10
T2	Corncob char	Clay	80:20
T3	Corncob char	Clay	70:30
T4	Cowpea shell char	Clay	90:10
T5	Cowpea shell char	Clay	80:20
T6	Cowpea shell char	Clay	70:30
T7	Coconut husk char	Clay	90:10
T8	Coconut husk char	Clay	80:20
T9	Coconut husk char	Clay	70:30
T10	Corncob char	Starch	90:10
T11	Corncob char	Starch	80:20
T12	Corncob char	Starch	70:30
T13	Cowpea shell char	Starch	90:10
T14	Cowpea shell char	Starch	80:20
T15	Cowpea shell char	Starch	70:30
T16	Coconut husk char	Starch	90:10
T17	Coconut husk char	Starch	80:20
T18	Coconut husk char	Starch	70:30

TABLE 1: Treatments for briquette production

Results And Discussion

Proximate analysis

Agricultural residues and biochar were characterized by proximate analysis (volatile matter, fixed carbon, ash content, and moisture content). Figure *3* and Figure *4* show the results of proximate analysis for both residue and biochar samples. These results indicate the highest volatile matter of 22.5% for biochar. This decline can be attributed to the loss of volatile compounds during pyrolysis. Previous studies have demonstrated that increasing the pyrolysis temperature reduces volatile matter in biochar. Liu et al. [35] reported a decline in volatile matter with increasing temperature from 300°C to 700°C. This occurs because higher temperatures enhance the decomposition of volatile components into smaller, low-molecular-weight liquids and gases [36]. In contrast, biochar samples exhibited significantly higher fixed carbon content of 71% than the maximum recorded for the residue samples of 15%. Increasing pyrolysis temperatures facilitates the removal of volatile components, thereby increasing the concentration of fixed carbon in the biochar. For example, a study on crop residues reported that fixed carbon content rose from 47.51% to 71.69% as the pyrolysis temperature increased from 300°C to 600°C [35]. Similarly, Liu et al. [37] found that the carbon sequestration capacity of spent mushroom substrate biochar improved with higher pyrolysis temperatures, reaching 60.6% at 600°C, which aligns with the present study.

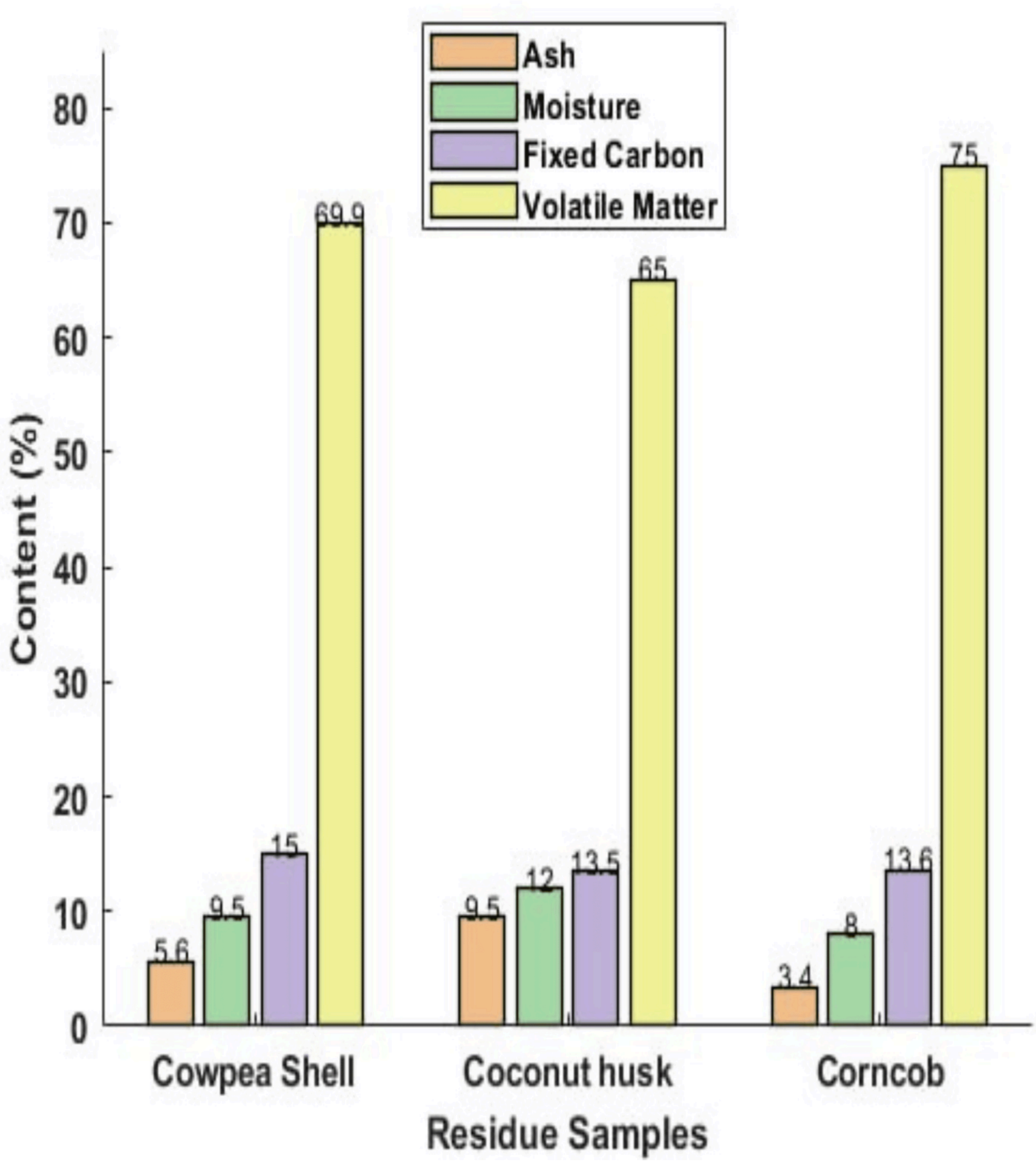

FIGURE 3: Proximate analysis of residue samples

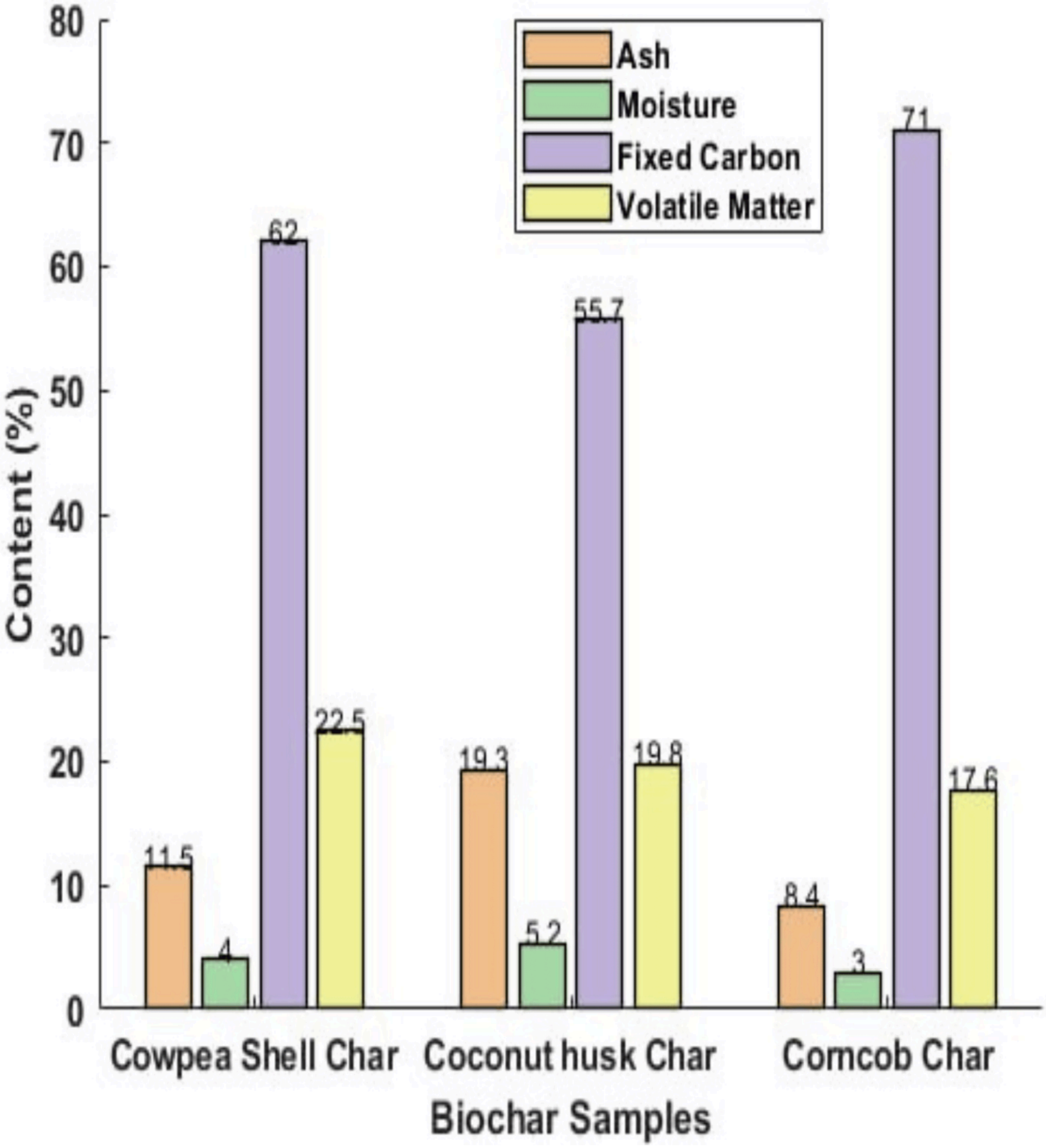

FIGURE 4: Proximate analysis of biochar samples

Ultimate analysis

Characterization of agricultural residues and biochar was based on their elemental composition. Ultimate analysis facilitates efficient biomass fuel design, quality control, and energy potential assessment for applications in power generation and biorefineries, with models achieving high accuracy through optimal element combinations, such as carbon with nitrogen or oxygen [38]. Figure *5* and Figure *6* show the results of ultimate analysis for both residue and biochar samples. Residue samples exhibited the highest overall oxygen content of 49.9%, while biochar samples reached a maximum oxygen content of 27.8%, which indicates a decline in oxygen content. In contrast, carbon content increased following pyrolysis, with biochar samples achieving a maximum of 78.0%, compared to 49.9% in the residue samples. Both samples generally displayed low sulfur and nitrogen levels. Santos et al. [39], in their study on biochar-based fertilizers, noted that biomass feedstocks typically contain low nitrogen and sulfur, which are further diminished during pyrolysis. Similarly, Lavie et al. [40] reported refuse-derived biochar briquettes containing low nitrogen from 0.44% to 0.52%, and sulfur from 0.09% to 0.21% content. These findings are consistent with the results of the present study.

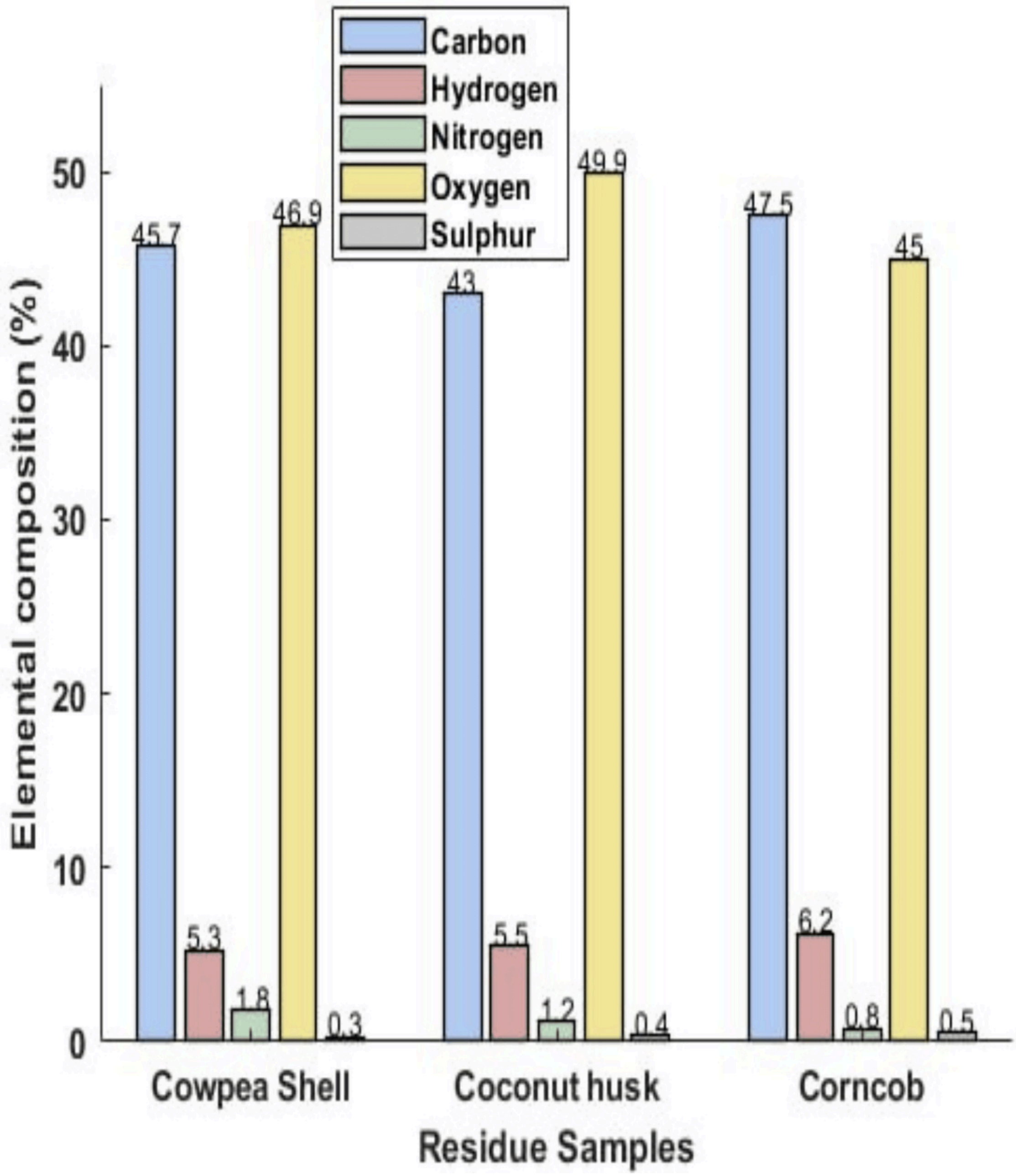

FIGURE 5: Ultimate analysis of residue samples

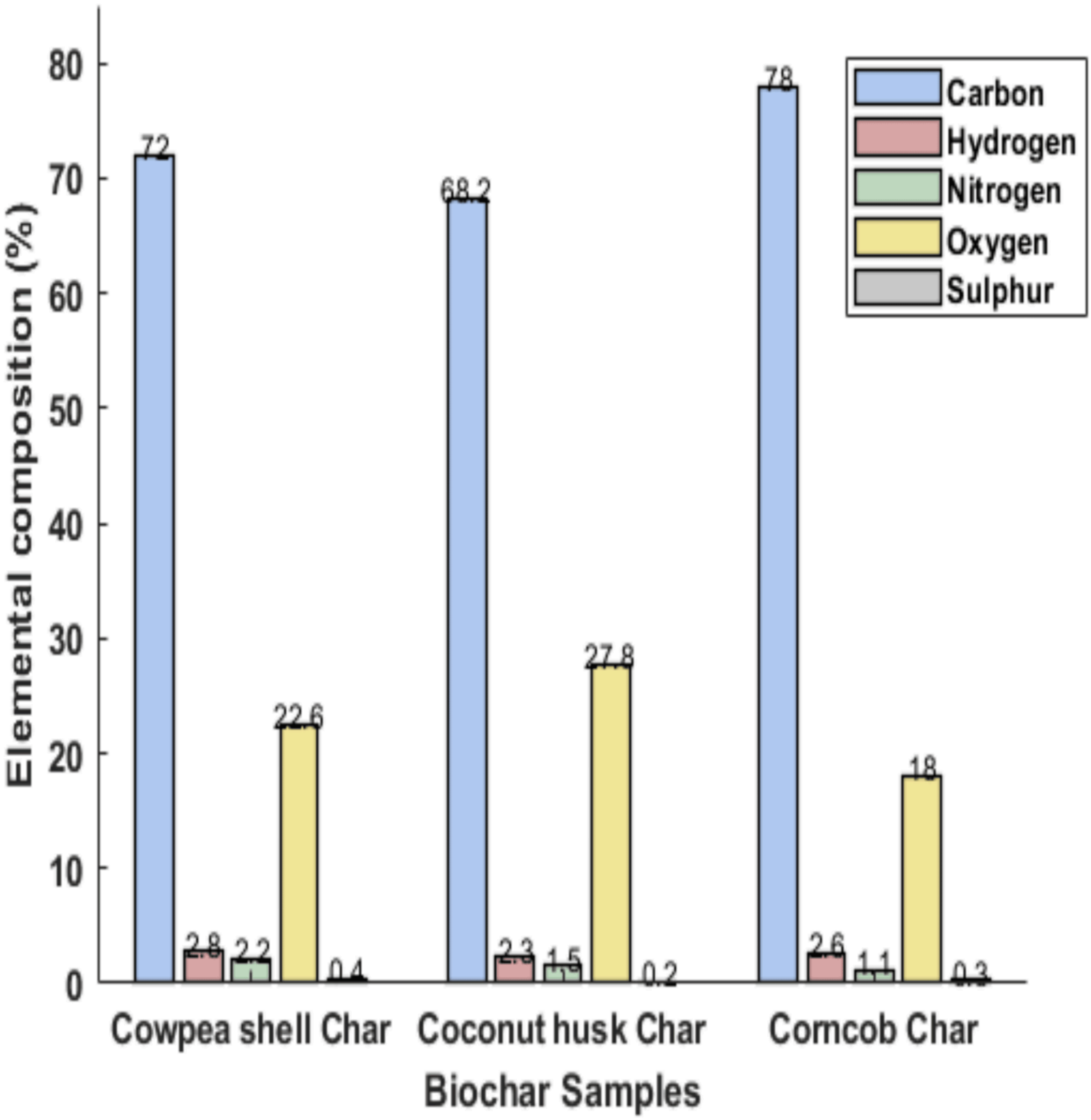

FIGURE 6: Ultimate analysis of biochar samples

Briquette characterization

Briquettes were characterized by physicochemical properties (responses), namely: calorific value, compressive strength, shatter index, WBT, and burning rate. Table 2 shows the mean and standard error values for all properties.

Calorific value is defined as the amount of heat energy produced per unit mass of a fuel. The highest calorific value for bio-briquettes had a mean value of 20.04 ± 0.74 MJ/kg and the lowest mean value obtained was 17.52 ± 0.49 MJ/kg. Ali et al. [41] reported calorific values ranging from 16.20 to 19.80 MJ/kg for hybrid bio-briquettes produced from a mixture of sawdust, sugarcane bagasse, and paddy straw. Kipngetich et al. [42] also had calorific values of 15.80-28.40 MJ/kg from carbonized rice husk briquettes.

Compressive strength of a briquette measures its resistance to crushing or breaking under pressure during storage and transportation. The highest mean value for compressive strength for biochar briquettes was 22.99 ± 4.47 N/mm^2, and the lowest mean value of compressive strength recorded was 21.99 ± 2.28 N/mm^2. Ramírez-Ramírez et al. [43] investigated the physical and thermal properties of hardwood biomass briquette biofuels and reported compressive strength values ranging from 10.3 N/mm^{-1} to 98.6 N/mm^{-1}.

Shatter Index is defined as resistance to breakage when a mass of an object is dropped. The highest mean shatter index value obtained was 87.86 ± 5.15% compared to the lowest mean shatter index value of 85.44 ± 2.46%. Fadele et al. [44] characterized briquettes from forest wastes using optimization approach and reported an optimum shatter index value of 99.86%.

WBT is a standardized method to determine the time a fuel will take to boil water to 100°C. The fastest mean value the briquettes took to boil water was 13.41 ± 1.61 min, while the slowest time it took had a mean value of 15.66 ± 1.81 min. Senchi and Kofa [45] investigated carbonized rice husk briquettes produced with starch and gum Arabic as binders in varying ratios. Their findings indicated that briquettes bonded with 25% gum Arabic required 22 min to boil 1 L of water, whereas those with a 45% gum Arabic ratio required 18 min [41]. Similarly, Senchi and Kofa [45] reported that starch-bonded briquettes with a 25% ratio took 20 min, while those with a 45% ratio boiled 1 L of water in 14 min. Burning rate measures the rate at which fuels combust. The highest burning rate of briquettes recorded was (2.19 ± 0.04) g/min and the lowest burning rate mean value obtained was 1.92 ± 0.10 g/min [46]. The burning rate of the fuel briquettes and wood charcoal ranged from 3.16 ± 0.06 g/min for carbonized shell powder with starch as the binder to 3.03 ± 0.05 g/min [46]. All mean values are consistent with those reported in recent literature.

Properties	Briquettes					
	Cowpea shell char		Coconut husk char		Corncob char	
	Mean	SE	Mean	SE	Mean	SE
Calorific Value (MJ/kg)	17.52	0.49	20.04	0.74	19.79	2.64
Compressive strength (N/mm^2)	22.99	4.47	21.99	2.28	22.84	5.96
Shatter Index (%)	86.89	5.43	87.86	5.15	85.44	2.46
Water Boiling Test (mins)	15.66	1.81	14.89	1.99	13.41	1.61
Burning rate (g/min)	2.19	0.04	1.92	0.10	1.94	0.16

TABLE 2: Properties of briquettes and their respective mean values

Effects of factors on responses

Analysis of Variance (ANOVA) was employed to evaluate the effects of factors (biochar type, binder type, and mixing ratios) on responses (calorific value, compressive strength, shatter index, WBT, and burning rate). Tukey HSD test was also used to generate parameters highlighting significant differences in groups. However, calorific value and compressive strength were considered for inferential analysis. Present studies consistently emphasize these parameters, as they are key requirements for energy performance and structural integrity [47-49].

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Char type	104.72	2	20.94	10.16	0.0192	Significant
Binder type	29.41	1	29.41	14.27	0.0026	Significant
Ratios	52.24	2	26.12	12.67	0.0011	Significant
Residual	24.74	12	2.06			
Cor total	129.46	17				

TABLE 3: ANOVA for calorific value against biochar type, binder type, and ratios

ANOVA, Analysis of Variance

Char type	N	Mean	Grouping	
Coconut husk char	6	20.00	A	
Corncob char	6	19.76	A	
Cowpea shell char	6	17.49		B

TABLE 4: Grouping information using Tukey method and 95% confidence for char type

Binder type	N	Mean	Grouping	
Starch	9	20.36	A	
Clay	9	17.81		B

TABLE 5: Grouping of binder types using Tukey method with a 95% confidence level

Mixing ratios	N	Mean	Grouping	
90:10	6	21.14	A	
80:20	6	19.14	A	B
70:30	6	16.97		B

TABLE 6: Mixing ratios grouped using Tukey method at a 95% confidence level

Table *3* presents the ANOVA results for calorific value, indicating that char type, binder type, and mixing ratios all had a significant effect ($p < 0.05$) on the calorific value of the briquettes. According to Table *4*, coconut husk char and corncob char belong to the same statistical group, while cowpea shell char forms a separate group, signifying that cowpea shell char is significantly different from the other two. Biochar type may play a predominant role in determining calorific value. Biochar types with different values of carbon content may alter the calorific value of biochar briquettes. Biochar derived from hardwoods typically has a higher fixed carbon content and lower volatile matter, resulting in calorific values that often exceed 28-30 MJ/kg [50]. In contrast, biochar produced from agricultural residues such as rice husk or empty fruit bunch (EFB) tends to have higher ash content, which serves as a non-combustible diluent, thereby lowering the effective energy density [51,52]. Nikkhah Shahmirzadi et al. [50] explored biochar derived from date palm and pistachio residues and reported calorific values of around 29.5 MJ/kg and 30.2 MJ/kg, respectively, attributed to their low ash content and high carbon content. Looking at Table *5*, starch and clay belong to separate statistical groups, indicating that starch is significantly different from clay. Organic binders such as starch contain carbon and hydrogen, which may contribute to the calorific value of biochar briquettes. Starch binders, including cassava starch, are commonly used for their biodegradability and effective binding properties. However, they generally have lower intrinsic calorific values, approximately 15-17 MJ/kg, compared to lignocellulosic biochar [53,54]. Excessive use of starch as a binder can dilute the high-energy carbon matrix of biochar, thereby reducing its overall calorific value. This effect becomes especially significant when the binder content exceeds 10-15 wt%, as demonstrated in studies on rice husk and Gloriosa superba waste briquettes, where higher starch content was associated with notable reductions in heating value [52,50]. Nikkhah Shahmirzadi et al. [50] found that molasses can enhance the calorific value of briquettes after carbonization, whereas bitumen adds high-carbon, high-energy components that increase the net heating value. These findings show that binder type influences calorific value support the principle that binders are active energetic contributors rather than inert fillers, and their chemical composition must be considered in the design of fuel formulations. Table *6* explains that the

90:10 ratio forms a distinct group, while the 70:30 ratio also belongs to a separate group. The 80:20 ratio is grouped with both 90:10 and 70:30, indicating that 90:10 is significantly different from 70:30. If binder percentage has a lower calorific value than biochar, increasing the binder percentage may cause a decrease in the total calorific value. An optimal mixing ratio achieves the highest possible carbon content while maintaining briquette cohesion. Research demonstrates that increasing the proportion of biochar in the mixture increases the calorific value, as the energy content of biochar becomes the dominant factor in the composite [51,53]. However, exceeding practical limits when biochar content is very high can undermine structural integrity. Mohd Fuad et al. [51] reported that EFB-starch briquettes with a 70:30 biochar-to-binder ratio produced significantly lower heating values than those with a 90:10 biochar-to-binder ratio under the same torrefaction conditions. Application of response surface methodology to rice husk briquettes revealed a strong negative correlation between binder concentration and calorific value, highlighting the trade-off between improved processability and reduced energy density [52].

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Char type	3.19	2	1.59	0.20	0.82	
Binder type	323.00	1	323.00	40.15	0.0001	Significant
Ratios	720.03	2	360.01	44.76	0.0001	Significant
Residual	96.53	12	8.04			
Cor total	1142.75	17				

TABLE 7: ANOVA assessing the effects of binder type and mixing ratios on compressive strength

ANOVA, Analysis of Variance

Binder type	N	Mean	Grouping	
Starch	9	26.81	A	
Clay	9	18.33		B

TABLE 8: Grouping of binder types using Tukey method with a 95% confidence level

Mixing ratios	N	Mean	Grouping		
90:10	6	14.67	A		
80:20	6	22.90		B	
70:30	6	30.15			C

TABLE 9: Mixing ratios grouped using Tukey method at a 95% confidence level

Table *7* shows the ANOVA results for compressive strength, indicating that binder type and mixing ratios had a significant effect ($p < 0.05$) on briquette compressive strength. According to Table *8*, starch and clay fall into distinct statistical groups, indicating that starch differs significantly from clay. Inorganic binders such as clay depend solely on mechanical entanglement, which may result in a brittle structure that fails easily under compression. Starch-based binders, such as cassava starch, are widely used due to their strong initial material compaction and biodegradability [53,55]. However, Ravindiran et al. [54] reported that the effectiveness of these binders is highly sensitive to processing conditions; at lower pyrolysis temperatures, they may volatilize, potentially resulting in a weaker structure if the material is not fully carbonized or cross-linked. Xiao et al. [52] also noted that the complex aromatic structure of lignin provides a more stable binding matrix during high-temperature processing, often resulting in higher final compressive strength. Other binders, such as molasses and calcium hydroxide, function through distinct mechanisms; molasses serve as a viscous adhesive that, during carbonization, forms a hard, glassy phase, while calcium hydroxide can react with constituents in the biochar or surrounding atmosphere to produce cementitious compounds, thereby enhancing the structural strength of the briquettes [52,53]. The selection of a binder is therefore a critical decision when considering briquette strength. Table *9* denotes that all selected mixing ratios (90:10, 80:20, and 70:30) all belong to three different groups A, B, and C, respectively, depicting significant differences among them. Increasing the binder ratio up to an optimal point may maximize strength by completely filling the internal voids of biochar briquette. A high proportion of biochar (>90 wt%) generally results in inadequate binder coverage of particle surfaces, preventing the formation of a continuous binding network and leading to structural weak points and reduced compressive strength [51,52]. However, an overabundance of binder (>15-20 wt%) can be equally detrimental. Excess binder can lead to the formation of thick, carbon-rich domains that may undergo excessive shrinkage or cracking during drying. Alternatively, the binder could act as a soft, non-load-bearing material, hindering the transfer of stress between the rigid biochar particles [50,53]. The best briquettes will be those for which strong bonds between interfaces are formed, which could be through physical entanglements, hydrogen bonds, π-π interactions, especially with aromatics, or co-carbonization during pyrolysis [50,52]. Mixing ratios should be considered carefully in briquette production as emphasized by recent studies.

Relationship among selected properties of briquettes

Correlation analysis was used to examine how briquette properties interact. A correlation matrix, as shown in Figure *7*, displays the correlation coefficients (Pearson's r) for the selected responses (compressive strength, calorific value, burning rate, shatter index, and WBT), with values between -1 and +1 indicating the direction and strength of the linear relationships. It is used to detect multicollinearity, spot patterns in the data, and perform feature selection in machine learning [56].

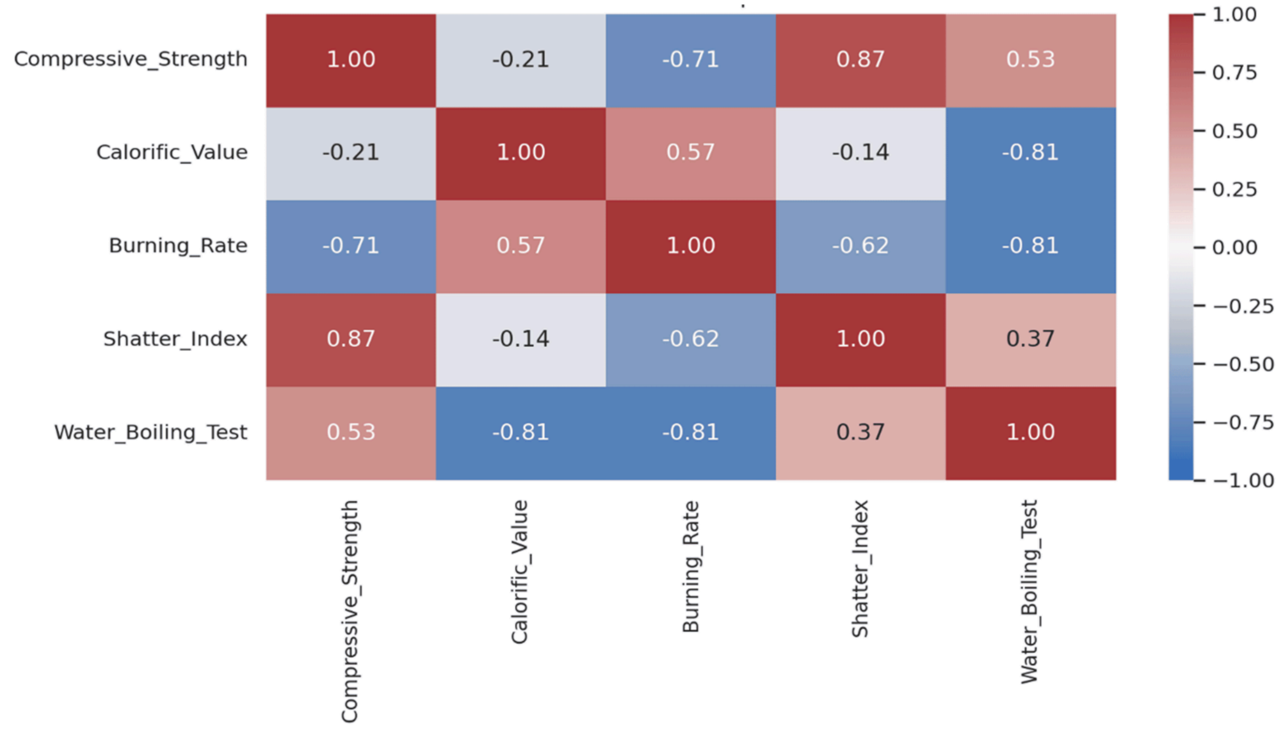

FIGURE 7: Correlation matrix for responses

The maximum value of positive correlation coefficient, r = 0.87, was noted for compressive strength and shatter index. Briquettes having high crush resistance ability in compressive strength tests had high durability during multiple shatter index tests. As a result, cohesion ability of briquette matrix increases and its resistance towards both static and dynamic loads is improved. The findings are consistent with the earlier researches on biomass briquettes, which have shown strong positive interdependence of compressive strength and impact resistance due to compaction and binder content ratios [57,58]. Another important positive correlation was identified between calorific value and burning rate, r = 0.57. The positive correlation indicates that briquettes with high energy will be highly flammable and combustible.

The WBT yielded a highly significant negative correlation with calorific value of r = -0.81 and burning rate of r = -0.81. The briquettes with high calorific value and initial burning rates finish the boiling process in a shorter period. These findings are consistent with previous research on charcoal briquettes derived from agricultural residues. Calorific value and burning rates were correlated with improved cooking efficiencies as reported by Silas et al. [59]. One significant negative correlation found was between compressive strength and burning rate, r = -0.71. The burning rate decreases with increased compressive strength. Compression reduces the pores within the briquette; hence, the oxygen absorbed is minimal. It takes time to burn completely, which is suitable for burning for an extended period.

Conclusions

Corncob char exhibited the highest fuel potential, with its fixed carbon (71%) and elemental carbon (78%) after pyrolysis, surpassing both cowpea shell char with its fixed carbon (62%) and elemental carbon (72%) and coconut husk char with its fixed carbon (55.7%) and elemental carbon (68.2%). Nitrogen and sulfur content in all samples were found to be substantially low, which ensures efficient burning. Corncob char briquettes recorded the highest mean compressive strength (22.84 ± 5.96) N/mm^2 and coconut husk char briquettes recorded the highest mean calorific value of (20.04 ± 0.74) MJ/kg. The study also found out that binder type and mixing ratio significantly influence the compressive strength of briquettes, while binder type, biochar type, and mixing ratios significantly affect the calorific value of briquettes. Compressive strength and shatter index had a positive correlation coefficient (r = 0.87), and calorific value and burning rate also had a positive correlation coefficient (r = 0.57). However, WBT and calorific value had a negative correlation coefficient (r = -0.81), and compressive strength and burning rate also had a negative correlation coefficient (r = -0.71). Future research should investigate the potential of alternative organic and inorganic binders beyond starch and clay, such as molasses, gum Arabic, or natural resins. Researchers should also explore a broader range of agricultural residues, specifically readily available waste residues such as rice straw, sugarcane bagasse, hay, and sawdust. Subsequent studies should expand the characterization of produced briquettes by examining properties such as ash fusion temperature, smoke emission density, ignitability, and microstructural analysis (Scanning Electron Microscopy/Energy Dispersive X-ray Spectroscopy).

Additional Information

Author Contributions

All authors have reviewed the final version to be published and agreed to be accountable for all aspects of the work.

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Disclosures

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Data Availability Statements

The datasets (and/or code) supporting this study are available from the corresponding author upon reasonable request.

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