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The effects of alkaline pre-treatment on the combustion profiles of fuel briquettes produced from mango seed shells compared to untreated ones

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ABSTRACT

The need for an alternative energy source to complement the use of fossil fuels and mitigate the environmental and economic challenges arising from the overexploitation of nonrenewable energy sources has led to the use of biomass briquettes as a suitable alternative. This study investigates the performance of alkaline-pretreated biomass briquettes produced from mango seed shells. The pulverized sample was divided into two equal samples. A portion of the sample was soaked in 0.3 M sodium hydroxide for 3 hours, then washed and dried. The second portion remained untreated. Eighty grams of both the treated and untreated samples were thoroughly blended with 20g of starch slurry and fed into the briquetting machine, where they were compressed to produce briquettes. The produced briquettes were sun-dried for 4 weeks and then analyzed. The proximate composition, physical properties, combustibility properties, and SEM of the briquettes were determined. The alkaline-treated sample (BTS) had a value of 1498.67 ± 1.04 kJ/kg, and the untreated sample (UTS) had a value of 1495.44 ± 1.72 kJ/kg. The compressive strength of alkaline pretreated (BTS) was 0.783± 0.250. It took 20 minutes for the treated sample to boil one litre of water, while it took 22 minutes for the untreated sample to boil the same volume. The results showed that both treated and untreated samples could be used for briquette production; however, the treated briquette sample had better performance and hence was a better fuel.

Keywords: Briquette, Proximate Compression, Combustibility.

1. Introduction

It is evident that the world is running short of its energy supply generated from fossil fuels and wood, which is due to the rapid depletion of these non-renewable energy reserves. However, exploring an alternative to this hydrocarbon-based energy has become a matter of economic and environmental importance. Expanding energy services, especially to the rural households, while simultaneously addressing the health and environmental consequences has become a matter of concern (Qi et al., 2023). According to Adeoye & Lawal (2025), energy demand from fuelwood is expected to increase to 91 million tonnes by 2030, resulting in a continued deforestation rate. This highlights the need to explore affordable energy systems for domestic and industrial use in the country, with a focus on renewable, eco-friendly, and cost-effective sources. Fossil fuels, which are non-renewable energy sources, also pose a potential threat to the climate due to the high emission of greenhouse gases (CO_2, CH_4, NO_2, etc.). Considering all these factors has become a central stimulant for research on renewable energy sources.
Recently, there has been a growing global interest in developing technologies that harness renewable energy sources. Also known as green energy, it is utilized for both environmental and economic reasons (Obi et al., 2022; Udume et al., 2022). Biomass is naturally abundant and presents a renewable energy opportunity that could serve as an alternative to fossil fuels. Nigeria has approximately 71.9 million hectares of arable land, and various grasses are cultivated for primary agricultural purposes. The enormous forest resources and landmass in Nigeria, with flourishing vegetation, imply that the regeneration of these resources through appropriate means can be a simple way of contributing to solving a seemingly intractable power supply problem (Ferronato et al., 2022). It is common in most Nigerian rural communities to fell trees. The problem of felling trees for use as a fuelwood source hurts the environment. One way to limit deforestation and protect the environment is through the briquetting of flammable materials. Therefore, using biomass, which is organic waste from this vegetation, will tremendously reduce deforestation and provide an alternative renewable energy source. The effective utilisation of these materials offers many advantages, one of which is their role as a renewable and sustainable energy feedstock for the production of solid fuel briquettes (Zhang et al., 2018). Briquettes are flammable materials formed by the compression or densification of matter into solid form to be used as fuel. Briquetting is a process of binding together pulverized carbonaceous matter, often with the aid of a binder (Obi et al., 2022). The common forms of briquettes are coal briquettes and biomass briquettes. Discovery has shown that binder type and curing conditions exert the greatest influence on briquette quality (Udume et al., 2022). Therefore, it is necessary to carry out studies on different treatment methods and identify the most suitable ones for maximum combustion output. Hence, this research tends to study the effects of alkaline pre-treatment on the combustion profiles of fuel briquettes produced from mango seed shells against the untreated one.

2. MATERIAL and METHOD

2. 1. Materials

The mango shell of *Foetide lour* (Julie), a small-seeded species, was collected within Aliero Local Government because of its low moisture content of 72.04%, compared to other varieties of mangoes in the region. Starch was used as a binder. All necessary glassware was pre-treated by soaking in a dilute nitric acid solution for 24 hours, followed by thorough rinsing with distilled water prior to use. The chemicals, reagents, and solvents employed throughout the study were of analytical grade and general-purpose quality.

2. 2. Sample Pre-treatment

The collected sample was dried in direct sunlight for 2-3 days to reduce its moisture content before being pulverised into powder form using a mortar and pestle and a mechanical grinding engine. The powder was sieved through a 60-mesh sieve to achieve the required texture size and to remove impurities. The prepared sample was divided into two equal portions.

2. 3. Alkalization of the Sample

The first portion of the sample was soaked in an alkaline solution of NaOH with an optimised concentration of 0.3M. The equivalent in weight of NaOH was soaked with the mango shell powder at a ratio of 3:1 for three hours at room temperature (Bamisaye & Rapheal, 2023). The pre-treatment helps to reduce the lignin content in the mango shell, making it easier to bind its particles together. The other portion was left untreated.

2. 4. Briquettes Production

80g of the sieved alkaline-treated sample and untreated sample were independently mixed with 20g starch binder in the ratio of 4:1 (Obi et al., 2023). The briquettes were produced using a locally made briquetting machine; the mixture was poured into the cylindrical metal container of the briquetting machine and covered with an iron desk with the combined effect of the compression.

Figure 1. Image of homemade fabricated briquetting machine and produced briquette

2. 5. Proximate Analysis

A proximate analysis was conducted on the produced briquettes.

1. Percentage Moisture Content (PMC).
2. Percentage Ash Content (PAC).
3. Percentage Fixed Carbon (PFC).

2. 6. Determination of Briquette Physical Properties

The compressive strength of the briquettes was evaluated following the procedure outlined by Yakubu & Sadiq (2021). The dimensions length and width of each briquette were measured and recorded prior to testing. Each sample was centrally positioned on the lower plate of the testing machine. The plate was then raised electrically until it made contact with the upper plate, and the strain dial gauge was reset to zero. Initial readings for both the strain dial and the load dial at zero compressive force were recorded. A stopwatch was used to time the measurements, with load dial readings taken at every 0.2 mm of displacement, and corresponding strain dial readings noted as well. Additional readings were recorded at 0.4 mm intervals. The loading process continued until a clear indication of failure was observed evidenced by a drop or no change in load across several consecutive readings. Once failure was confirmed, the machine was halted, the motor allowed to come to a complete stop, and the setup reversed before the load was removed. The briquette sample was placed on the movable bed, and the control lever was operated upward to establish contact between the fixed upper plate and the movable lower plate. As soon as a crack appeared in the briquette, indicating compression failure, the dial reading was noted, and the corresponding force value from the strain dial was recorded.

The compressive strength was then calculated using Equation (1), and the result was expressed in kilonewtons per square meter (kN/m²).

$$\text{Compressive strength} = \sigma = RC_R\,(100 - \mathcal{E}\%) / 100A_0 \times 1000 \qquad (1)$$

Where:

R: dial reading, C_R: calibration load dial reading (2.11 Div.) constant, A_0 is cross-sectional area = $\pi D^2/4$, D: diameter of briquette (mm), Ɛ is the strain of sample: σ, Ɛ%: DL/L_0, DL is change in length of sample at compression, and L_0 is initial length of sample before compression.

The density of the briquettes was determined using the formula below:

$$Density = \frac{\text{weight of briquette(g)}}{\text{volume of briquette}(cm^3)} \qquad (2)$$

2. 7. Combustion Analysis

The following combustion analysis was carried out.

1. Calorific Value Determination.
2. Ignition Time Determination.
3. Determination of Burning Rate.
4. Combustibility Test.

2. 8. Lignocellulose Composition Analysis

Analysis of lignin and cellulose was carried out by using the method adopted by Zhang & Zhou (2024), where a mixture of 1 g of dried sample of mango shell and 150 mL of aquadest was heated in a water bath at a temperature of 90 - 100ºC for 1 h. The mixture was filtered, and the residue was washed with hot water (300 mL). The residue was oven-dried until a constant weight was achieved (b). It was then combined with 150 mL of 1 N sulphuric acid (H_2SO_4) and heated in a water bath at 90–100 °C for one hour. After heating, the mixture was filtered, and the remaining solid was rinsed thoroughly with 300 mL of distilled water (aquadest), followed by drying (c). The dried material was then soaked in 10 mL of 72% H_2SO_4 at room temperature for 4 hours. Subsequently, 150 mL of 1 N H_2SO_4 was added, and the mixture was subjected to reflux in a water bath for one hour. The resulting solid was washed with 400 mL of distilled water, then dried in an oven at 105 °C and weighed until a stable mass was reached (d). Finally, the residue was incinerated to ash and weighed again (e). The percentages of cellulose and lignin were then determined using the following calculations:

$$\%\ \text{cellulose} = \frac{c-d}{a}\ X\ 100\% \qquad (3)$$

$$\%\ \text{lignin} = \frac{d-e}{a}\ X\ 100\% \qquad (4)$$

.

Where:

a: sample weight (g), c : the residue weight (g) at the third weighing, d : the residue weight (g) at the fourth weighing, e : the weight of ash (g).

2. 9. Statistical Analysis

The average value of all the parameters analysed would be computed using arithmetic mean and one-way analysis of variance (ANOVA) to determine the probability that two populations are the same and to know whether there is a significant difference.

3. Result and discussion

3. 1. Proximate Compositions of the Briquette Sample

The results of the proximate analyses of briquettes are shown in **Table 1**. It was observed that the moisture content of the untreated sample was about 6.5%, while that of the alkaline-treated sample was 4%. The moisture content of the untreated sample was more than that of the alkaline, which implies that the treatment decreases moisture content. Higher moisture content value is reported to have negative effects on the combustion characteristics of briquettes (Demirbaş & Şahin, 1998). Since higher moisture content indicates a greater degree of wetness in the briquettes (Obi et al., 2023), low moisture content is required to protect against decomposition and disintegration of briquettes during storage and handling.

Ash content for the untreated sample was 1.83%, while that of the alkaline-treated sample was 2.5%, which is not above the maximum required value of not greater than 20% (Abimbola et al., 2022). This revealed that treatment increases ash content. These results compared favourably to the good-quality charcoal range (1.2% to 8.9%) as stated by Abayomi et al (2022) (Bamisaye et al., 2024). The work agreed with the work of Zhang et al., 2023, who recorded 2.5% of ash content for shea nut shell (Qi et al., 2023).

Also, the volatile matter of the two samples is 79.00% to 85.33% for the alkaline-treated and untreated samples, respectively. The fixed carbon content result for the untreated sample was 6.33% and 14.17% for alkaline treatment; hence, treatment increases the fixed carbon content. Generally, the higher the volatile matter, the better the briquettes (Adesola Ajagbe et al., 2025) since high volatile matter briquettes ignite easily. The fixed carbon is used to determine the heating value and acts as a heat generator during combustion (Ferronato et al., 2022). The higher the fixed carbon content of a briquette, the greater the calorific value.

Table 1. Result of the Proximate Analysis of the Briquette

	Untreated Sample	Alkaline-treated sample
Moisture (%)	6.50 ± 0.50	4.33 ± 1.04
Ash (%)	1.83 ± 0.29	2.50 ± 0.50
Volatile matter (%)	85.33 ± 1.15	79.00 ± 0.00
Fixed carbon (%)	6.33 ± 1.04	14.17 ± 1.44

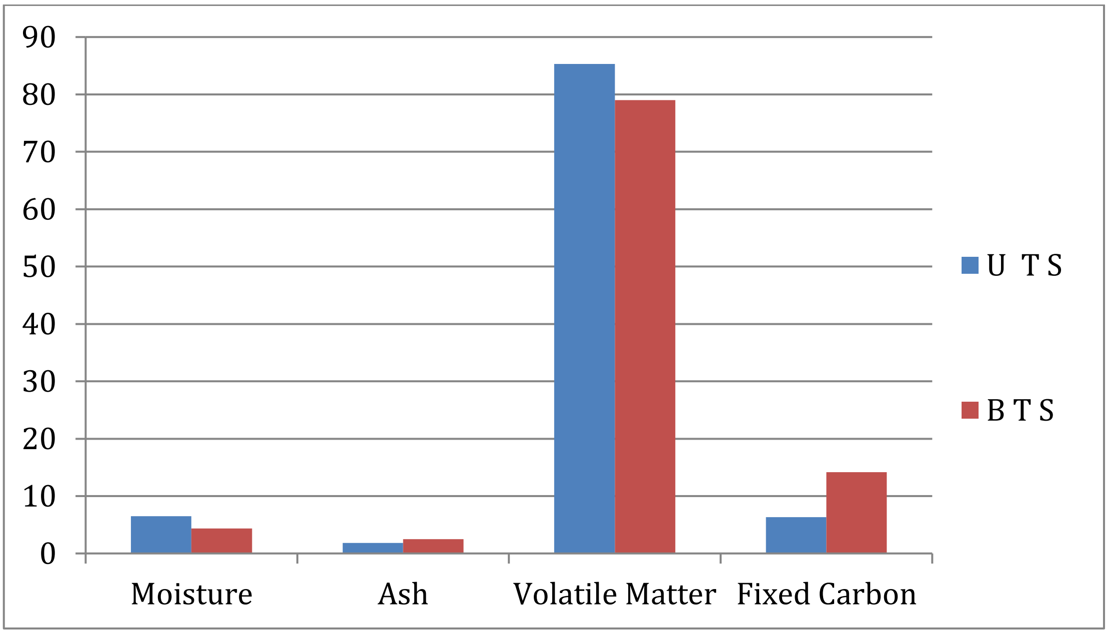

Figure 2. Proximate Analysis

3. 2. Physical Properties of the Briquette

Table 2 shows the result of the physical properties of the respective briquette samples, results of the physical properties of the briquettes are shown in Table 2, which shows that the alkaline sample has the highest density and compressive strength. The compressive strength from the untreated sample was 0.717 ± 0.012 N/mm^2 while that of the alkaline-treated sample was 0.783 ± 0.250 N/mm^2. It was observed that there is no significant difference in the compressive strength of the respective samples with ($p<0.05$) on this property. This is probably because they were compressed at a constant force upon the same cross-sectional area of the briquettes. The compressive strength values indicate that the briquettes are suitable for transportation, packing, and storage. The high compressive strength of the alkaline-treated sample, as shown in Table 2, could be attributed to the loss of hemicellulose in the samples. According to Oyibo et al 2020, NaOH-treated samples usually have a decreased fibre diameter of the samples' particles because of the loss of hemicellulose in the samples.

The density of the briquettes decides how long they would burn and the amount of fly ash they would release while burning High-quality briquettes should have high-energy content and burn for a longer time. The density of the briquettes for the untreated sample was 0.359 ± 0.005 g/cm^3, while that of the alkaline sample was 0.357 ± 0.00 g/cm^3. Thus, there was a significant difference in the density of the briquettes at ($p<0.05$). The briquette densities were found to be lower than that of the mahogany tree, which was 0.682 g/cm^3 as stated by Zhang & Zhou (2024).

Table 2. Physical Properties

	Compressive Strength (N/mm^2)	**Density (g/cm^3)**
Untreated sample	0.717 ± 0.012	0.359 ± 0.005
Alkaline-treated sample	0.783 ± 0.250	0.359 ± 0.005

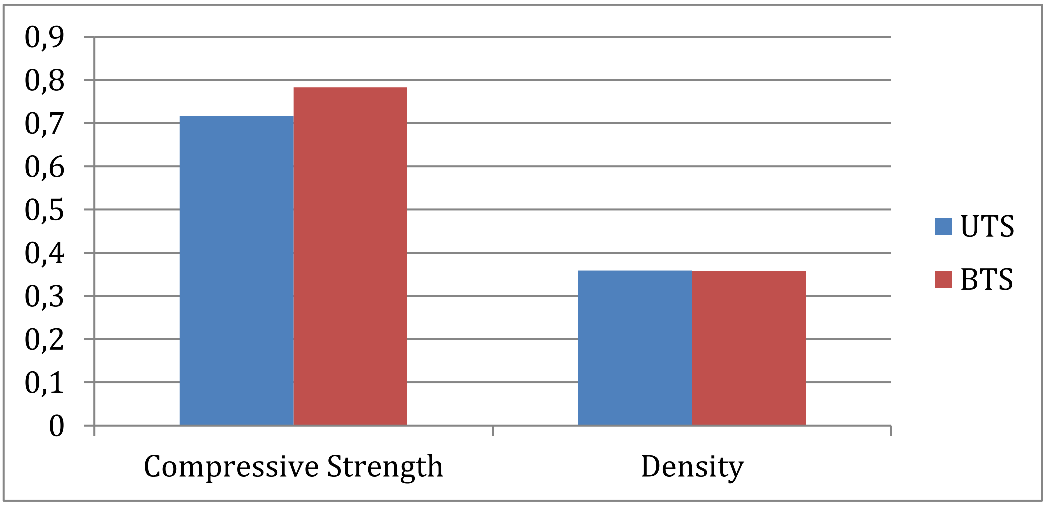

Figure 3. Physical Properties of Briquette Sample

3. 3. Combustibility Test of the Briquettes

Table 3. Combustion Characteristics

	Calorific value (KJ/Kg)	**Burning Rate (g/min)**	**Ignition Time (mm/s)**
Untreated Sample	1495.44 ± 1.72	28.547 ± 0.011	45.550 ± 0.580
Alkaline-treated sample	I498.67 ± 1.04	18.690 ± 0.012	41.540 ± 0.528

Table 3 shows the result of the combustion properties of the respective briquette samples. The results show that the calorific value of the treated sample, as shown in **Table 3**, was higher than the calorific value of the untreated sample. This shows that there is a significant difference in the calorific value when a biomass is treated. This is probably due to the fact that treatment of a sample affects its moisture content. The calorific values obtained range between 1495 KJ/Kg and 1498 KJ/Kg. The higher the calorific value, the better the combustion characteristics for energy generation.

The untreated sample had the highest burning rate, even though it had the lowest calorific value. However, this deviation was explained by the SEM result in **Figure 4**. The untreated sample has a very porous interface; the higher the porosity, the higher the circulation of oxygen, which increases the combustion rate (Ngene et al., 2024). A slow burning rate is desirable because less heat is required for cooking (Yunusa et al., 2023). The result of the burning rate, as presented in **Figure 4**, agrees with the results of Zhang et al., 2023**,** who treated carbonised rice husk with different binders.

The time taken for the untreated sample to attain a steady glow was higher than the time taken for the treated sample to attain a steady glow, as shown in **Figure 4**. The ignition time of both treated and untreated samples is lower than that of Abayomi et al, 2022 (Bamisaye et al., 2024).

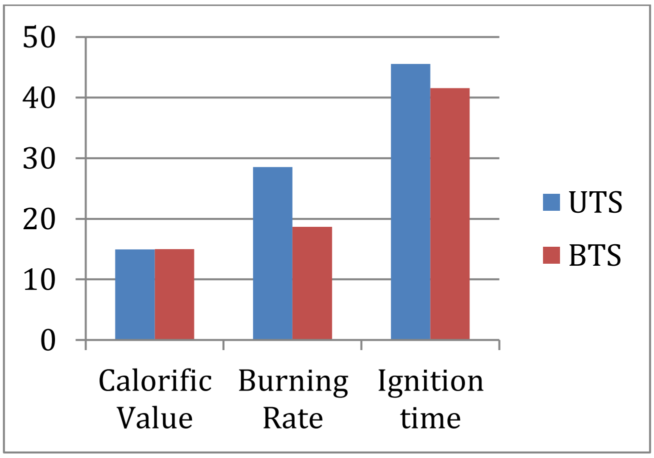

Figure 4. Combustion Properties of Briquettes Sample

3. 4. Combustibility Test

Figure 5 below shows the result for the water boiling test on the alkaline-treated sample and the untreated sample.

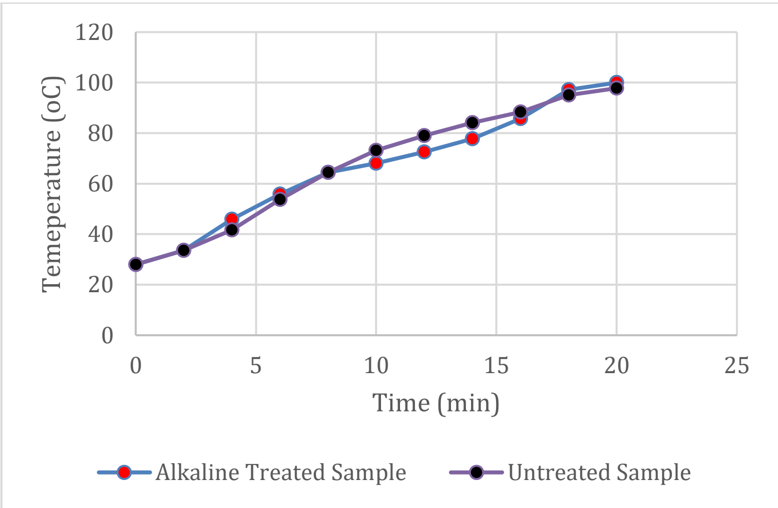

Figure 5. Water Boiling Test

The results from the combustibility test show that the alkaline-treated sample has a higher boiling rate at a lower temperature range between 0^0C to 5^0C, and a lower rate between 5^0C to 10^0C. Generally, the alkaline-treated briquette has a shorter combustibility time of 20 minutes than the untreated sample, which has a combustibility time of 22 minutes. These results show that the treated sample boils water faster than the untreated sample.

3. 5. Morphology of the Produced Briquette

The surface morphology of the alkaline-treated sample and the untreated sample are shown by scanning electron microscopy in **Figures 6 and 7**, respectively.

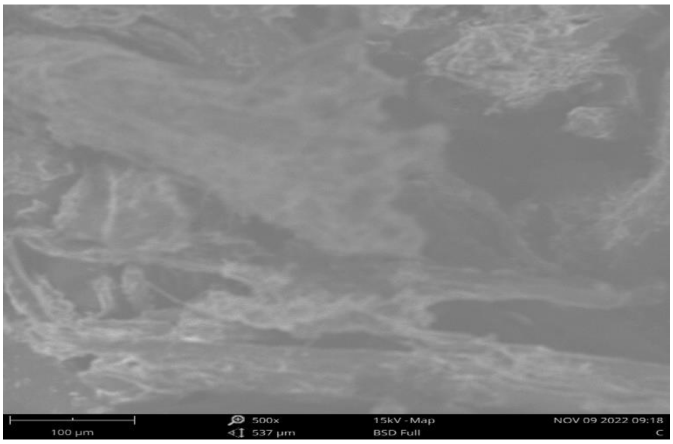

Figure 6. Morphology of Alkaline-Treated Sample at 1500x Magnification

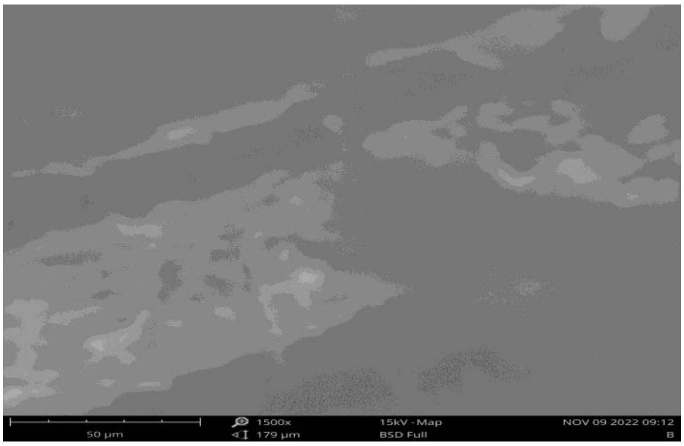

Figure 7. Morphology of Untreated Sample at 1500x Magnification

From the results, it can be observed that the outer surface texture of the untreated sample was porous, and there was an absence of destructive features on the surface. The alkaline pre-treatment caused more compaction of the briquette material, as shown in **Figure 6**. This compaction was due to the pressure of hemicellulose, lignin, and other impurities such as wax, pectin, and grease covering the components and protecting the cellulose inside (Zhang et al., 2018). The destructive degree of roughness of the NaOH-treated samples was attributed to the penetration of the NaOH solution into the samples. NaOH breaks down and removes hydroxyl groups and surface impurities in the amorphous non-cellulose constituents, which increases the number of crystalline sites exposed on the fibre surface (Zhang et al., 2018).

4. Conclusion

This research investigated and compared the combustion characteristics of mango seed shell briquettes in both treated and untreated forms. Experimental results indicated that briquettes derived from both sample types are suitable as biomass fuel sources. Nonetheless, the treated briquettes exhibited superior biofuel properties compared to their untreated counterparts. Specifically, the treated samples demonstrated an enhanced calorific value, higher density and compressive strength, greater fixed carbon content, reduced moisture levels, and required less time to boil an equivalent volume of water under identical conditions. Scanning Electron Microscopy (SEM) revealed that treatment with sodium hydroxide (NaOH) altered the surface structure, making it rougher due to the removal of amorphous, non-cellulosic components as the solution infiltrated the fibre surfaces. This increased surface roughness improved fibre-matrix interfacial bonding, thereby enhancing resistance to moisture, as reflected in the lower moisture content of the treated briquettes.

5. Recommendations

Based on the findings of this study and the conclusion made, it is recommended that:

1. More research should be focused on briquettes using varied feedstocks (biomass).
2. Reagents other than NaOH should be explored as alkalis in the treatment of biomass for briquette production.
3. Acid-treated mango seed briquettes should be researched.

Conflict of Interest

On behalf of all authors, the corresponding author states that there is no conflict of interest.

Data Availability

No data was used for the research described in the article.

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