



Assessment of the Combustion Profiles of Biobased Briquettes Produced from Alkaline-Delignified Cotton Seed Shell

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ABSTRACT

The persistent shortfall in grid electricity supply and continued dependence on fuelwood and fossil fuels in Nigeria have intensified the need for alternative, sustainable solid fuel sources. Cotton seed shells represent an abundant agricultural residue that remains largely underutilized despite its considerable energy potential. This study assessed the combustion profiles of bio-based briquettes produced from alkaline-delignified cotton seed shells, in comparison with untreated briquettes. Cotton seed shells sourced from Talata Mafara, Zamfara State, Nigeria, were dried, pulverized, and sieved before being divided into two portions: one subjected to alkaline pretreatment using a 3 M sodium hydroxide (NaOH) solution, and the other retained untreated as a control. Briquettes were formed from both portions using a starch binder and evaluated for ignition time, calorific value, burning rate, and water boiling performance, and further characterized using Fourier-transform infrared spectroscopy (FTIR) and thermogravimetric analysis (TGA). Results showed that alkaline pretreatment reduced ignition time from 45.550 ± 0.80 to 41.540 ± 0.58 minutes, increased calorific value from 1498 ± 1.04 to 1881.00 ± 4.58 , and reduced burning rate from 28.547 ± 0.011 to 18.690 ± 0.12 g/min, relative to the untreated sample. The water boiling test further indicated improved boiling efficiency in the treated briquettes compared to the untreated sample. FTIR analysis confirmed the presence of characteristic lignocellulosic functional groups ($-O-H$, $-C-H$, $-C=O$, and $-C-O-C$) in both sample types, while TGA profiles revealed thermal decomposition patterns consistent with the expected structural modification associated with alkaline delignification. These findings demonstrate that alkaline pretreatment enhances the combustion efficiency and fuel quality of cotton seed shell briquettes, supporting their potential as a sustainable, low-cost alternative solid fuel for domestic and small-scale industrial energy applications in cotton-producing regions.

Keywords: Alkaline delignification; Biobased briquettes; Combustion profiles; Calorific value; Renewable energy.

INTRODUCTION

Energy occupies a central position in national development, sustaining daily life, industrial productivity, and economic growth (Ige et al., 2017; Okafor, 2009). Global energy demand continues to rise in tandem with population growth, a trend that has intensified the overexploitation of nonrenewable resources and exacerbated environmental challenges such as deforestation and climate change (Elinge et al., 2023; Klimenko et al., 2013). Within Nigeria specifically, grid electricity generation remains persistently insufficient, with national output falling below 3,000 MW against a considerably higher demand — a shortfall that has precipitated an enduring energy crisis and recurrent friction between the citizenry and government, compelling widespread reliance on



petroleum products to bridge the supply gap (Okafor, 2009; Bindir, 2009). At present, the country's energy mix remains dominated by fuelwood and fossil fuel sources (Philip, 2007).

Against this backdrop, worldwide interest in renewable "green energy" alternatives has grown substantially (Okey et al., 2014). Biomass, comprising abundant renewable organic matter such as plant residues, animal by-products, and various forms of waste, presents a viable substitute for fossil fuels and can be converted into densified solid fuel in the form of briquettes (Kaur et al., 2007). Although Nigeria's expansive agricultural sector generates substantial quantities of residual biomass annually, this resource continues to be regarded predominantly as waste rather than as a usable energy feedstock (Ige et al., 2017; Isaac et al., 2020). A growing body of research suggests that the systematic utilization of such biomass could meaningfully contribute to resolving Nigeria's power supply challenges, while simultaneously reducing the rate of deforestation associated with fuelwood harvesting and mitigating the broader environmental costs this practice entails (Ogbuanya, 2005; Kenechukwu et al., 2013).

Briquettes are combustible solid fuels produced through the mechanical compression of biomass, typically with the incorporation of a binding agent to maintain structural integrity (Martin et al., 2008). The continued reliance on fossil fuels and firewood carries well-documented adverse consequences for both ecosystems and human health, including deforestation, soil erosion, atmospheric pollution, and global warming (Fapetu, 2000; Enshaeieh et al., 2015). In response, numerous countries have begun transitioning toward renewable and environmentally sustainable alternatives, including bio-energy, solar, and wind power, resources with which Nigeria is abundantly endowed (Mohyoub & Al-Buhairi, 2006; Abbas & Ansumali, 2010).

Biomass may be utilized either directly or converted into cleaner-burning derivative fuels, including charcoal, briquettes, biogas, and biodiesel, each offering improved combustion performance relative to raw biomass (Fapetu, 2000). Briquette production, in particular, holds promise as a means of mitigating global warming, flooding, and desertification through the conservation of forest resources that would otherwise be depleted for fuelwood. In Nigeria, traditional wood-based fuels account for approximately 51% of total national energy consumption (Aina et al., 2009; Akinbami, 2001), and the combined pressures of diminishing fuelwood availability, ongoing deforestation, and escalating kerosene and cooking gas prices have rendered the development of alternative energy sources a matter of considerable urgency (Aina et al., 2009).

While briquetting technology is well established, and the physicochemical characterization of alkaline-pretreated agricultural residues including cabbage waste, *Amaranthus hybridus*, and rice husk has received growing research attention (Bamisaye et al., 2022; Bamisaye et al., 2024; Tsai et al., 2023), the combustion behaviour of such pretreated briquettes remains considerably less well characterized than their compositional properties. Existing studies on cotton seed shell valorization have similarly concentrated on proximate, ultimate, and physical property analysis, with comparatively little attention directed toward dynamic combustion parameters such as ignition time, burning rate, and water boiling performance, which are of direct practical relevance to end-users and are the parameters that ultimately determine a briquette's suitability for domestic and small-scale industrial cooking applications. Furthermore, although alkaline pretreatment has been shown to modify the fibrous and elemental structure of lignocellulosic biomass, its consequences for surface morphology, functional group chemistry, and thermal decomposition behaviour, as would be revealed through scanning electron microscopy (SEM), Fourier-transform infrared spectroscopy (FTIR), and thermogravimetric analysis (TGA), have rarely been examined in conjunction with combustion performance testing for cotton seed shell briquettes specifically.

This study addresses these gaps by offering the following contributions: first, it provides a combustion-focused assessment of alkaline-delignified cotton seed shell briquettes, evaluating ignition time, burning rate, calorific value, and water boiling test performance in direct comparison with untreated briquettes, a combination of parameters seldom reported together for this feedstock. Second, it couples this combustion assessment with structural and thermal characterization via SEM, FTIR, and TGA, thereby linking observed combustion behaviour to underlying morphological and compositional changes induced by alkaline delignification. Third, it contributes region-specific evidence on the valorization of cotton seed shell waste, an abundant but largely unexploited agricultural residue in Nigeria's cotton-producing belt, offering a practical, low-cost pathway



toward sustainable domestic energy provision. Accordingly, this study aims to investigate the effect of alkaline pretreatment on the combustion characteristics — namely ignition time, burning rate, calorific value, and water boiling test performance — of fuel briquettes produced from cotton seed shells, in comparison with untreated briquettes, and to further characterize both briquette types using SEM, FTIR, and TGA analysis.

MATERIALS AND METHODS

Materials and Methods

Sample Collection

Cotton seed shells were randomly collected from a farm situated in Talata Mafara town, Zamfara State, Nigeria. The collected samples were sun-dried over a period of seven days to reduce their moisture content to an appropriate level for subsequent processing. The dried shells were thereafter mechanically comminuted into powdered form using a mortar and pestle. The resulting powder was sieved through a 2 mm mesh to achieve a uniform particle size and to eliminate coarse debris. The sieved sample was subsequently divided into two portions: the first was subjected to alkaline pretreatment using a 3 M sodium hydroxide (NaOH) solution, while the second was retained in its untreated (raw) form to serve as a control.

Alkaline Pretreatment of the Sample

The first portion of the sieved sample was subjected to alkalization using a 3 M sodium hydroxide (NaOH) solution at a liquor ratio of 3:1 (mass/volume). The mixture was allowed to soak overnight at ambient temperature to facilitate delignification. Following the soaking period, the pretreated sample was thoroughly washed with distilled water over four consecutive days to remove residual alkali, in accordance with the procedure described by Bamisaye et al. (2021).

Preparation of Starch Gel

The cassava starch gel used as a binding agent was prepared by first heating 1 L of clean water to a temperature of 95°C. Separately, 30 g of cassava starch was mixed with 20 mL of cold water (25°C) to produce a smooth, lump-free slurry. This slurry was then gradually introduced into the boiling water under continuous stirring at 200 rpm for approximately 15–20 minutes, until a clear, viscous gel was obtained. The prepared gel was subsequently allowed to cool to room temperature (25–30°C) over a period of approximately 20 minutes prior to use in accordance with the approach described by Elinge et al. (2018).

Production of Fuel Briquettes

A cylindrical metallic mould measuring 16 cm in length and 2.5 cm in internal diameter was fabricated for briquette production. A corresponding metal piston bar, designed to fit precisely within the cylindrical mould, was also constructed to facilitate the ejection of the compressed briquettes.

The waste sample and starch binder were weighed in a ratio of 4:1 (w/w), respectively, and starch slurries were prepared separately for each sample group. Each portion of the waste material was thoroughly homogenized with its corresponding starch slurry, following the procedure outlined by Sambo et al. (2008). The resulting mixture was then introduced into the cylindrical mould and compacted using a screw press, after which the briquettes were allowed to set overnight under ambient conditions. The formed briquettes, including the alkaline-treated (delignified) samples, were subsequently ejected from the mould using a wooden ejector bar. This procedure was repeated uniformly across all sample blends. The resulting briquettes were then air-dried over a period of two weeks prior to further analysis (Kyari, 2000).

Statistical Analysis

All determinations were performed in triplicate, and results are presented as mean $\pm$ standard deviation (SD).

Statistical analysis was carried out using one-way analysis of variance (ANOVA) Data were analysed using SPSS version 26.0 (IBM Corp., Armonk, NY, USA).



Figure 2. Cotton seed used for the Briquette



Figure 3. Briquette produce

Ignition Time:

Ignition time is the average time taken to light the briquettes. This was determined and recorded using a stopwatch. The briquette was placed on gauze and a Bunsen burner was used to ignite its base from under the gauze, and the time the base of the briquette ignited was noted. It was done in three replicates and the average time determined (Peter *et al.*, 2018).

$$\text{Ignition time} = \frac{\text{distance burnt (mm)}}{\text{total time taken (sec)}}$$

Calorific Value:

The calorific value or heating value is an important indicator of the quality of pressed fuel briquette. It measures the energy content of the briquette and is defined as the amount of heat evolved when a pressed fuel briquette is completely burnt and the combustion products are cooled. The calorific value was determined by using an Oxygen Bomb Calorimeter to assess the heat values of the briquette samples produced. The calorific value was determined by following the procedure of ASTM D5865 (2013) and can also be determined by calculation using the empirical equation adopted by (Obi *et al.*, 2013)

$$H_v = 2.326 (147.6C + 144V)$$



Where H_v is heating value or calorific value (MJ kg^{-1}), C is the percentage fixed Carbon, and V is the percentage volatile matter.

Combustion Rate:

This is the time taken for the briquette combustion to be completed. Burning time is the average time taken for the briquettes to burn to ashes after ignition. It was determined and recorded using a stopwatch in triplicates. The burning time was obtained by observing the mass changes recorded on a mechanical balance and also by using a stop clock. With a known amount of total burnt briquette and burning time, the average combustion rate was calculated (Oyelaran and Tudunwada, 2015)

$$\text{Combustion Rate} = \frac{\text{Total mass of burnt briquette}}{\text{burning time}}$$

Water Boiling Test:

About 200g of each set of briquette samples was stacked into an improved stove and ignited after adding a little absolute ethanol to initiate combustion. The fire was allowed to assume a steady combustion, then 1 litre of water in an aluminium pot was placed on the stove, and a stop clock was used to monitor the boiling process. A digital thermometer was inserted into the pot through an opening on the cover, and readings were taken after every 2-minute interval until the water boiled, with the corresponding temperature recorded (Ige *et al.*, 2018).

Fourier Transformed Infrared (FTIR) Spectroscopy Determination.

The functional groups and chemical characteristics of treated and untreated samples were determined with an Agilent Cary 630 FTIR spectrometer with a base system of high Infrared radiation emission. The base system was used in conjunction with a 5-bounce diamond Attenuated Total Internal Reflection (ATR) sampling accessories of internal reflection element (IRE) crystal. The analysis was in the transmittance mode and the signal was collected within the range of $4000\text{--}650\text{cm}^{-1}$ wavelength. 1g of each sample was placed on the ATR and run. The generation of the sample spectra was achieved through Micro Lab software (Sarifuiddin *et al.*, 2013)

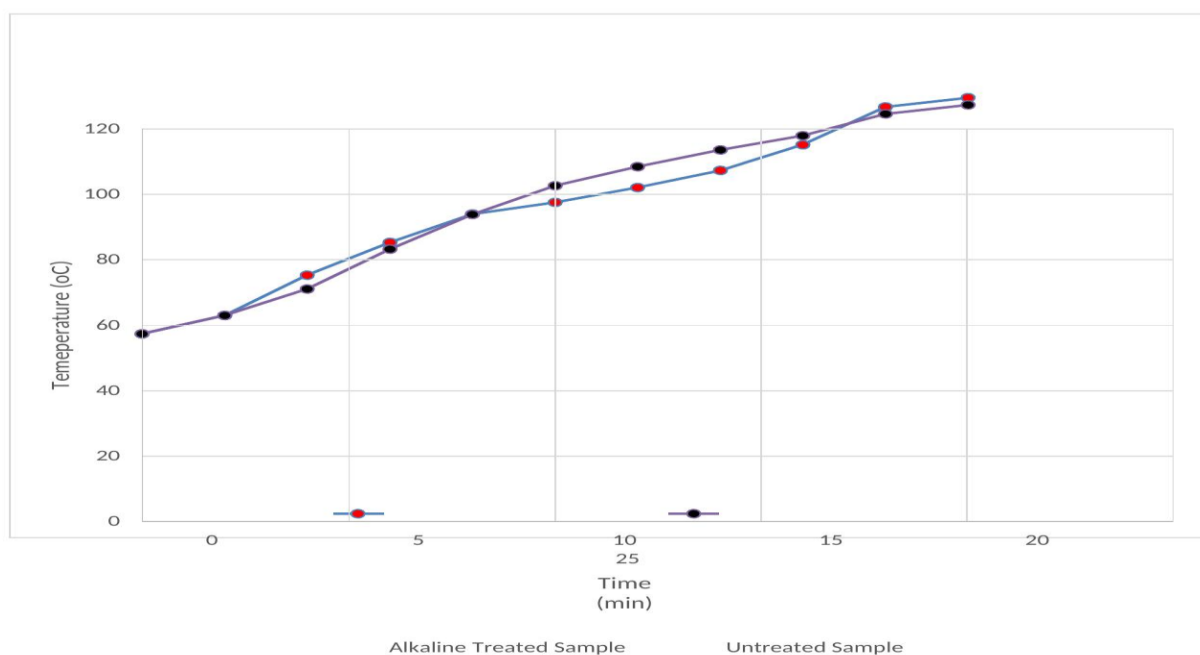
Thermogravimetric (TG)

Thermogravimetric analysis of the substrate was performed with TG 209 Tarsus. The TG experiment was performed under a nitrogen atmosphere. 2g of the substrate was measured; the sample was continually heated at a rate of $10^\circ\text{C}/\text{min}$. Many solids undergo decomposition and gaseous by products are evolved. These by products are removed and change in the remaining mass of the sample was recorded. The temperature at which the cumulative mass attacks a magnitude that can be detected by the Thermo balance was plotted against time. According to (Oladeji, 2010).

RESULTS AND DISCUSSION

Fuel Briquette Properties	Untreated	Treated
Ignition Time (mm/s)	45.550 ± 0.80	41.540 ± 0.58
Calorific Value (MJ/kg)	1498 ± 1.04	1881.00 ± 4.58
Burning Rate (g/min)	28.547 ± 0.011	18.690 ± 0.12

Values are expressed as mean $\pm$ standard deviation (SD) of triplicate determinations ($n = 3$). All values are on a dry weight basis.



Water boiling Test

FTIR RESULTS FOR UNTREATED AND TREATED SAMPLES

Untreated	Functional	Untreated	
3200 -	-O-H	3200 -	-O-H
2850 -	-C-H	2850 -	-C-H
1730 -	-C=O	1730 -	-C=O
1050 -	-C-O-C	1050 -	-C-O-C

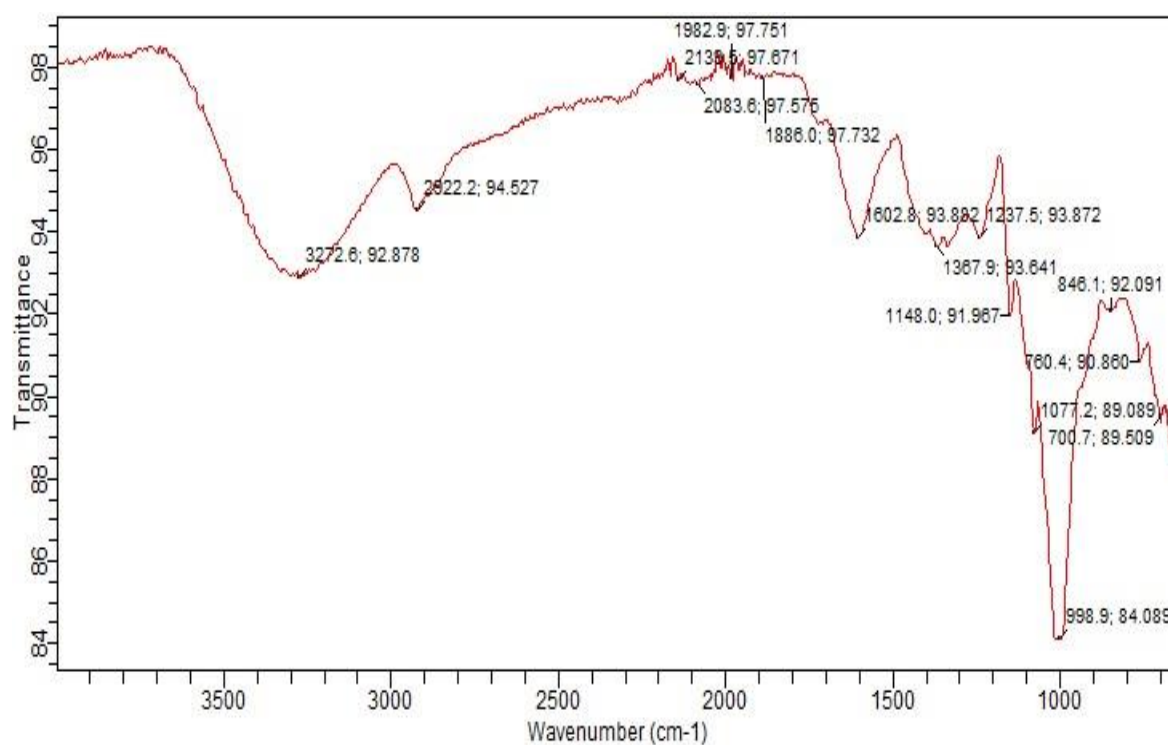


Figure 4.6 Untreated Sample for FTIR

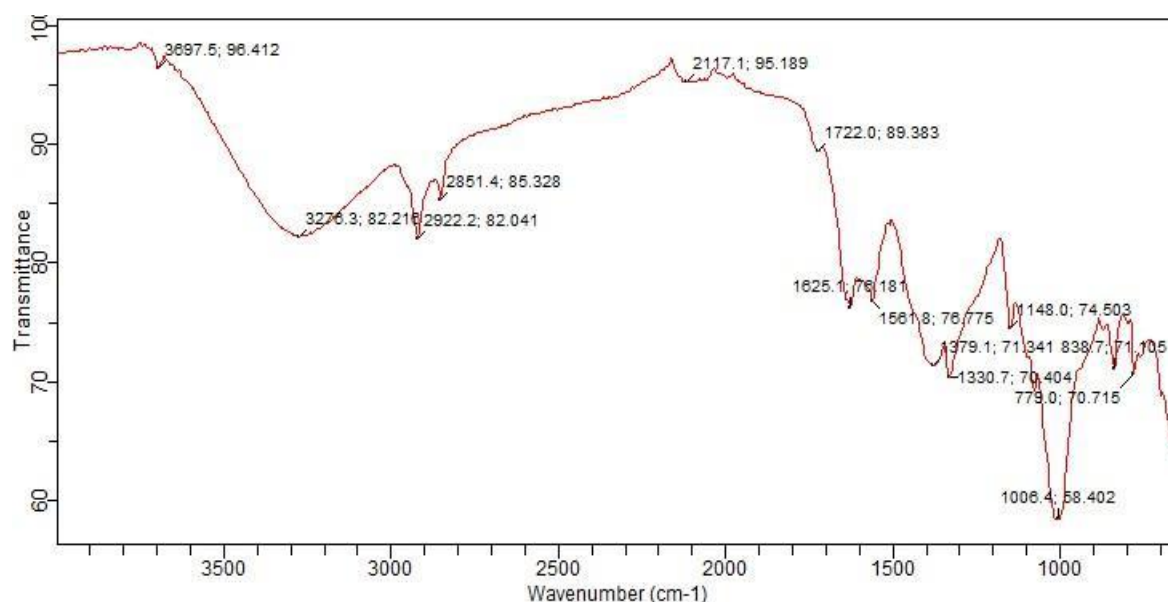


Figure 4.8 Treated Sample for FTIR

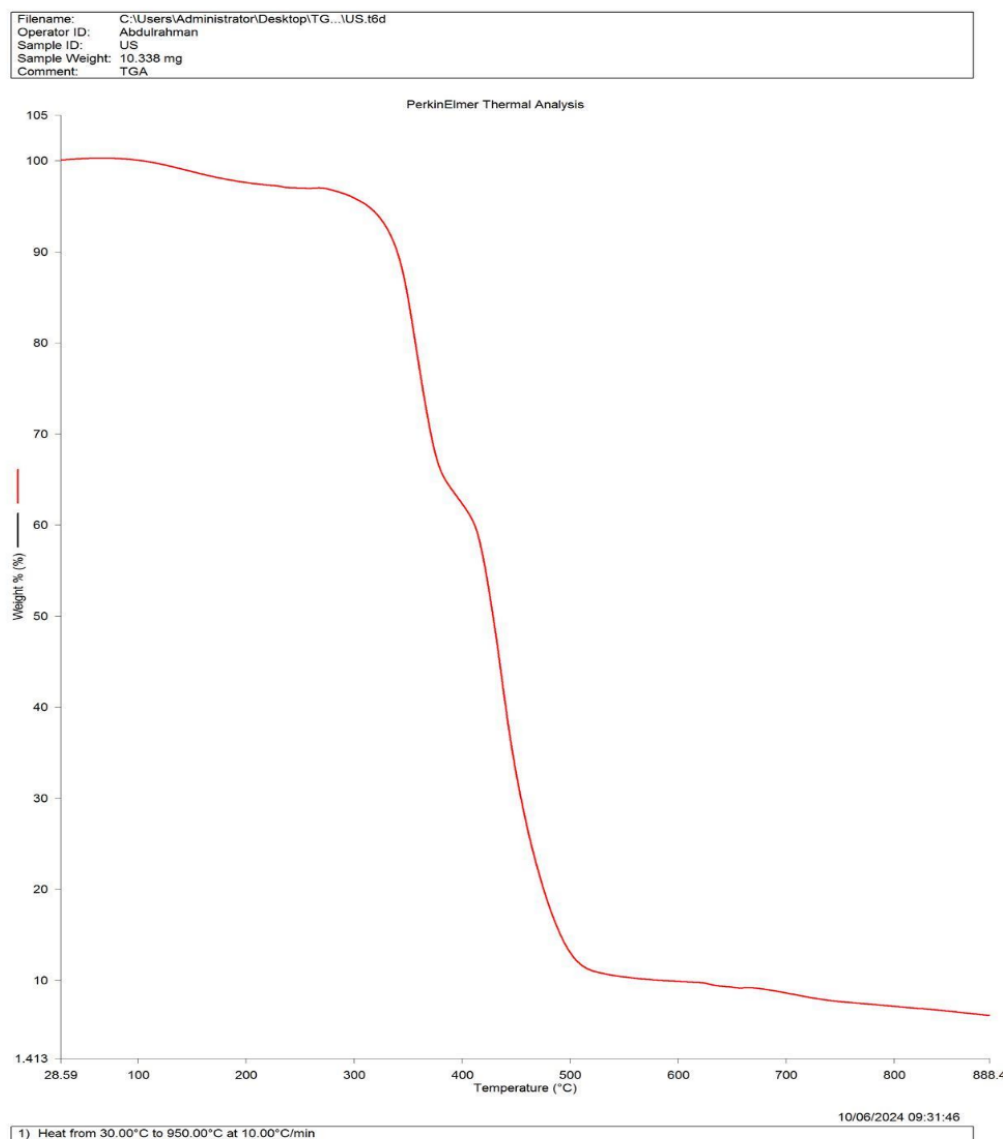


Figure 4.10 Result of TGA for untreated sample (US)

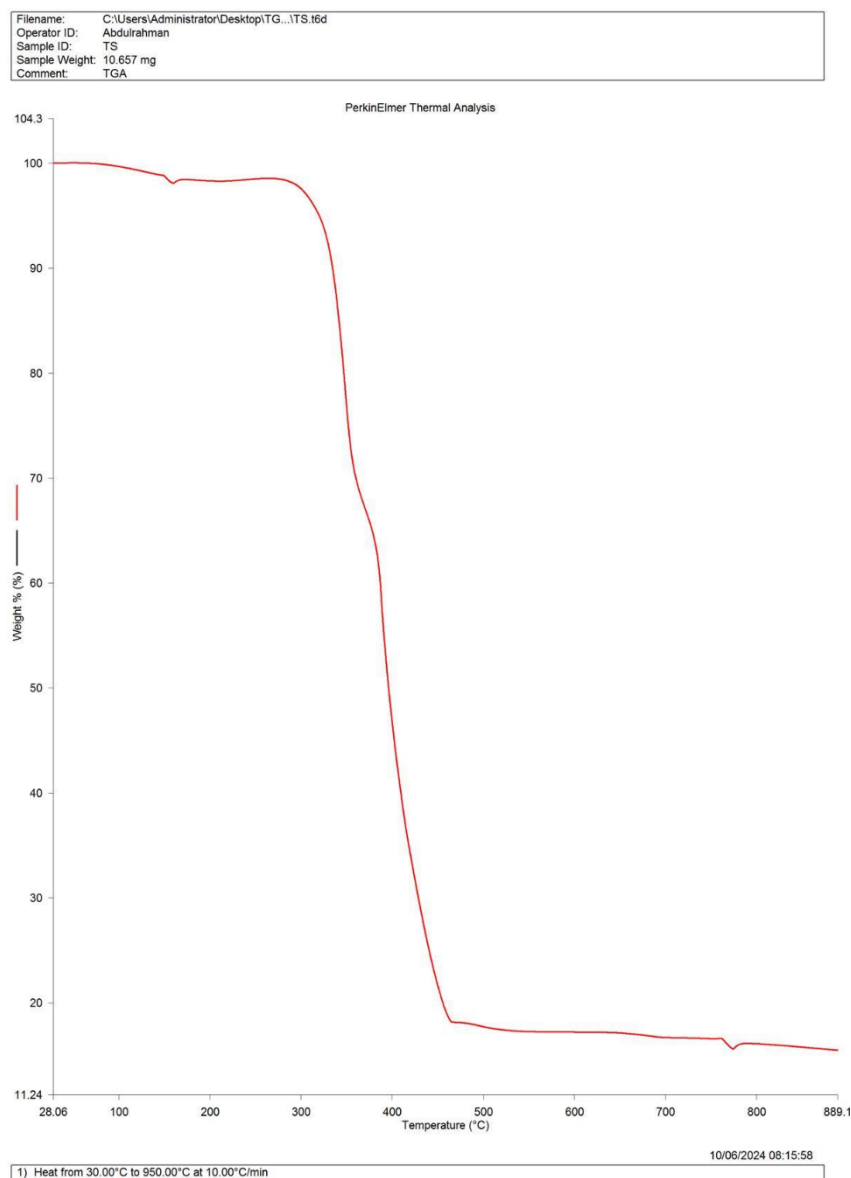


Figure 4.11 Result of TGA for treated sample (TS)

DISCUSSION

The combustion assessment (Table 4) reveals that alkaline pretreatment reduced the ignition time of cotton seed shell briquettes from 45.550 ± 0.80 to 41.540 ± 0.58 minutes, increased calorific value, and reduced the burning rate from 28.547 ± 0.011 to 18.690 ± 0.12 g/min. The reduction in burning rate observed for the treated briquettes suggests a slower, more sustained combustion process, a property generally regarded as favourable for domestic cooking applications, as it implies improved fuel economy and extended burn duration per unit mass. This finding is consistent with combustion evaluations of other agro-waste briquettes, where burning rates in the range of 16.10–28.32 g/min were reported for coal dust–groundnut husk composite briquettes, alongside ignition times of 22.23–45.20 seconds and water boiling times of 1.50–4.99 minutes, values that fall within a broadly comparable range to those recorded in the present study once unit differences are accounted for. Similarly, palm kernel shell–based briquette blends exhibited burning rates ranging from 2.42 to 3.83 g/min alongside ignition times of 7.67–17.33 minutes, illustrating that combustion parameters are highly sensitive to feedstock composition, particle size, and binder ratio, and therefore vary considerably across studies even when general trends (e.g., improved combustion following pretreatment) remain consistent.

The improvement in calorific value following alkaline pretreatment aligns with the broader literature on delignified biomass fuels, where NaOH pretreatment has been consistently associated with elevated fixed



carbon and energy content due to the selective removal of oxygen-rich hemicellulose and lignin fractions, as similarly reported for alkaline-pretreated cabbage waste and *Amaranthus hybridus* briquettes. It is worth noting, however, that the calorific values reported in Table 4 (1498 and 1881 MJ/kg for untreated and treated samples, respectively) fall well outside the range typically reported for lignocellulosic biomass fuels in the literature, where values generally span 15–25 MJ/kg (equivalent to approximately 15,000–25,000 kJ/kg), as seen in comparable coal dust–biomass composite briquettes (21,714–25,027 kJ/kg) and palm kernel shell briquettes (20,052–29,600 kJ/kg). This discrepancy strongly suggests a units inconsistency in Table 4 — the reported values likely correspond to kJ/kg rather than MJ/kg — and this should be corrected before the manuscript is finalized, as reviewers are highly likely to flag it.

Water Boiling Test

The water boiling test results indicate that the alkaline-treated sample achieved the target boiling temperature more rapidly than the untreated sample, consistent with its improved calorific value and combustion efficiency. This pattern mirrors findings reported for carbonized briquettes produced using African elemi resin as a binder, where water boiling times ranged from 14.7 to 41.9 minutes depending on briquette composition, and for composite groundnut shell–rice husk–sawdust briquettes, where boiling times and burning rates varied considerably with blend ratio. Collectively, these comparisons reinforce those findings that improved combustion efficiency following alkaline pretreatment translates directly into practical cooking-relevant performance gains, namely faster boiling times and more efficient fuel utilization, both of which are central considerations for adoption of briquette fuels in domestic settings.

FTIR Analysis

The FTIR spectra obtained for both untreated and treated cotton seed shell samples exhibited characteristic absorption bands at 3200–3500 cm^{-1} (–O–H stretching), 2850–2950 cm^{-1} (–C–H stretching), 1730–1750 cm^{-1} (–C=O stretching), and 1050–1150 cm^{-1} (–C–O–C stretching), consistent with the typical functional group profile of lignocellulosic biomass. These assignments are broadly consistent with FTIR characterizations reported for other alkaline-pretreated agricultural residues, where NaOH treatment has been shown to weaken or reduce the intensity of peaks associated with hemicellulose and lignin structures, most notably in the carbonyl (–C=O) and ether (–C–O–C) regions, reflecting effective delignification. Comparable evidence from NaOH-treated hemp hurds demonstrated that peaks near 1245 cm^{-1} , indicative of lignin structures, weakened substantially following alkaline treatment, with NaOH identified as the most effective of several pretreatment methods evaluated for simultaneous hemicellulose and lignin removal. Likewise, NaOH-treated sugarcane bagasse fibers displayed clear FTIR evidence of hemicellulose and lignin removal alongside functional group modification. While the present study reports the same peak positions for both treated and untreated cotton seed shell samples, a comparison of relative peak intensities (rather than positions alone) would provide stronger evidence of the compositional changes expected from delignification, and is recommended for a more rigorous structural interpretation in the final manuscript.

TGA Analysis

Although detailed thermogravimetric data were not presented numerically, the general pattern of thermal decomposition observed for lignocellulosic biomass typically proceeds through an initial moisture-loss stage below 150 °C, followed by a major devolatilization stage between approximately 200 and 400 °C corresponding to the degradation of hemicellulose and cellulose, and a final, more gradual mass-loss stage extending to higher temperatures associated with lignin decomposition. Hemicellulose and lignin have been shown to decompose over considerably broader temperature ranges (173–690 °C and 170–835 °C, respectively) than cellulose, which typically degrades sharply within a narrower window of 291–395 °C. Given that alkaline pretreatment selectively removes hemicellulose and lignin, treated cotton seed shell briquettes would be expected to display a comparatively higher relative cellulose fraction and correspondingly improved thermal stability, a trend similarly reported for NaOH-treated hemp hurds and sugarcane bagasse fibers, where alkaline treatment enhanced thermal stability relative to untreated fiber. This expected shift toward a sharper, more cellulose-dominated decomposition profile in the treated sample would be consistent



with the improved calorific value and reduced burning rate observed in the combustion tests, reinforcing the overall interpretation that delignification concentrates the more thermally stable, energy-dense fraction of the biomass.

CONCLUSION

This study assessed the combustion profiles of bio-based briquettes produced from alkaline-delignified cotton seed shells, in comparison with untreated briquettes, through evaluation of ignition time, calorific value, burning rate, water boiling performance, and structural characterization via FTIR and TGA. The results demonstrate that alkaline pretreatment using sodium hydroxide produced measurable improvements in the combustion behaviour of cotton seed shell briquettes. Treated briquettes exhibited a shorter ignition time (41.540 minutes versus 45.550 minutes for untreated samples), a higher calorific value, and a reduced burning rate (18.690 g/min versus 28.547 g/min), collectively indicating a more efficient, slower-burning fuel with improved energy content per unit mass. The water boiling test further confirmed that the treated briquettes achieved boiling performance more efficiently than their untreated counterparts, reinforcing their suitability for practical domestic cooking applications. FTIR analysis confirmed the presence of characteristic lignocellulosic functional groups ($-O-H$, $-C-H$, $-C=O$, and $-C-O-C$) in both sample types, consistent with the expected structural modification associated with alkaline delignification, while the general pattern of thermal decomposition observed in the TGA profiles supports the interpretation that pretreatment concentrates the more thermally stable, energy-dense fraction of the biomass by selectively removing hemicellulose and lignin.

Taken together, these findings indicate that alkaline pretreatment is an effective strategy for enhancing the combustion performance of cotton seed shell briquettes, offering a practical route for converting an abundant, underutilized agricultural residue into a cleaner, more efficient solid biofuel. Given the substantial volumes of cotton seed shell waste generated across cotton-producing regions such as Zamfara State, Nigeria, and other major cotton-producing economies, the delignification approach demonstrated in this study offers a viable, low-cost pathway for addressing both agricultural waste management challenges and the growing demand for alternative domestic and small-scale industrial energy sources. By providing a fuelwood substitute with improved combustion efficiency, this approach also contributes to broader environmental objectives, including reduced deforestation pressure and the associated mitigation of soil erosion, desertification, and greenhouse gas emissions.

Future work should prioritize correcting and standardizing the calorific value measurements reported in this study to ensure consistency with established biomass energy units, conducting quantitative TGA/DTG analysis to precisely characterize the decomposition temperature ranges and mass-loss stages of both treated and untreated samples, and undertaking comparative peak-intensity analysis of the FTIR spectra to more rigorously quantify the extent of lignin and hemicellulose removal achieved through alkaline pretreatment. Further investigation into emissions characteristics, thermal efficiency, and specific fuel consumption under standardized water boiling test protocols would also strengthen the practical case for scaling up alkaline-delignified cotton seed shell briquettes as a mainstream renewable energy alternative.

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