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Effect of alkaline delignification on physico-chemical and combustion properties of bean chaff briquette

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Abstract. *Abimbola AI, Rapheal IA, Abayomi B, Moki EC, Ayodele OO. 2021. Effect of alkaline delignification on physico-chemical and combustion properties of bean chaff briquette. Asian J Trop Biotechnol 19: 20-27.* Adopting lignocellulose-rich agro-waste materials for briquette production could be regarded as a better alternative energy source and also helps to ameliorate the challenges associated with deforestation and agro-waste disposal. This study examined the effects of alkali pretreatment on briquettes produced from bean chaffs. The proximate analysis, Energy Dispersive X-ray Fluorescence (EDXRF), Fourier-Transform Infrared (FTIR) analysis, Scanning electron microscopy (SEM), physical analysis and combustion properties were determined for the briquettes produced. The mean moisture content of 3.52±0.10% and 5.81±0.01% were recorded for Treated Bean Chaff (TBC) and Untreated Bean Chaff (UBC) briquettes, respectively. A high heating value of 24.18±0.12 MJ/kg was recorded for TBC compared to 21.12±0.01 MJ/kg of UBC briquette samples. Furthermore, it was observed that alkali pretreatment reduced the percentage of Potential Toxic Element (PTE) concentration in the treated sample, as shown by EDXRF. The FTIR results reveal surface modification in the fiber matrix shown by the C-O band shift from 1013 cm^{-1} observed in TBC to 1010 cm^{-1} recorded in UBC. In contrast, SEM shows clear disruption in the biomass matrix due to the alkali pretreatment process. The findings of this study show that the alkaline pretreated bean chaff briquettes have a great potential to be used as biomass fuel.

Keywords: Agro-waste, bean chaff, biomass, briquette, lignocellulose

INTRODUCTION

Bean production occurs in many countries, and Nigeria is one of the world's largest producers and consumers. Common bean also has the economic and environmental benefit of associating with nitrogen-fixing bacteria, which gives an advantage to fixing atmospheric nitrogen and leaving phosphorous (P) for plant growth. It is widely appreciated in developing countries for its affordability and long storage life. It is the most important crop for food security and wealth creation (Hordofa and Etisa 2019).

The utilization of agricultural and forest wastes as biomass is being progressively more studied and could serve as an alternative to fossil fuels and related problems (Fernandes et al. 2013; Amirta et al. 2016). But conversely, it is not easy to handle, transport, store, and use biomass in its original shape due to some factors, including high moisture content, irregular shape and sizes, and low bulk density (Karunanithy et al. 2012). Therefore, it is important to improve energy efficiency and technological innovation for biomass to be a notable impact as fuel.

The application of briquetting technology is one of the promising technology solutions to these problems. According to Wilaipon (2007), briquetting can be defined as a densification process for improving raw materials' handling qualities and enhancing the biomass's volumetric calorific value. In addition, the briquetting technology improves physical and combustion characteristics. The production of briquettes from raw biomass has been extensively studied (Husain et al. 2002; Ndiema et al. 2002).

Modifying the surface of biomass materials utilizing alkaline pretreatment is proficient and is usually done to advance adhesion between the biomass materials and improve the quality properties of the briquette (Mahalingam et al. 2019; Elinge et al. 2020). The briquette production process can be adjusted to improve the quality of the briquette through alkaline pretreatment of the agricultural biomass. Several researchers have generated briquettes from agricultural wastes, but few studies on the alkaline pretreatment of agricultural biomass for briquette production (Elinge et al. 2020; Oyibo et al. 2020; Bamisaye and Rapheal 2021).

To the best of our knowledge, there is no account to study the utilization of beans chaff for the production of biofuels. Therefore, there is a need to investigate the influence of alkaline pretreatment on the proximate, chemical, physical, and combustion properties of the briquette produced from beans chaff.

MATERIAL AND METHODS

Materials

The bean chaff used for this study, as shown in Figure 1, was sourced from Gashua, Yobe State, Nigeria. After collection, it was washed with tap water to remove impurities and dried in the oven at 105ºC for 24 hours. The dried biomass was sieved with a medium size, 2 mm particle and stored in airtight containers until further use.

Alkaline pretreatment

The bean chaff surface was treated with an alkaline solution. Half a cup of beans chaff was treated with 100 ml solution at 3% concentration, and soaking was done for 3 hours at room temperature. After soaking, the sample was filtered and washed with distilled water five times to get pH of 7.0. The washed residues were dried in an oven at 105ºC.

Lignocellulosic composition analysis

Analysis of lignin and cellulose was done according to the method of Mahyati et al. (2013).

The mixture containing 1 g of dry sample (a) and 150 ml of demineralized water was heated in a bathtub at a temperature of 100ºC per hour. The mixture was filtered, and the residue was washed with warm water (300 mL). The residue was dried in the oven until the weight was constant (b). Next, the residue was mixed with 150 mL of 1 N H_2SO_4 and heated in an oil bath at 100ºC for 1 hour. The mixture was dissolved and washed with 300 mL of water, demineralized, and then the residue was dried (c). The remaining residue is soaked in 10 milliliters of 72% H_2SO_4 at room temperature for 4 hours. After that 150 mL of 1 N H_2SO_4 was injected into the mixture and re-injected into the oil bath for an hour. The solid was dissolved with 400 mL of demineralized water, heated in the oven at 105ºC and measured until constant weight (d). Finally, the solid was heated to ashes and measured (e). The proportion of hemicellulose, cellulose and lignin was calculated as follows:

$$\%\ hemicellulose = \frac{(c-b)}{a} \times 100\% \ldots\ldots 1$$

$$\%\ cellulose = \frac{(d-c)}{a} \times 100\% \ldots\ldots 2$$

$$\%\ lignin = \frac{(e-d)}{a} \times 100\% \ldots\ldots 3$$

Briquette production

Briquettes are produced using a briquetting machine that operates on a 10-ton hydraulic jack that attaches to a cylindrical mold chamber that compresses the slurry. The length and width of the briquettes are 94 mm and 61 mm, respectively. Processed cassava starch was purchased from Gashua market, Yobe State, and was used as a binder. The untreated bean chaff was measured, mixed in a ratio of 4: 1, thoroughly mixed with water to form a slurry and fed into the molds to form briquettes and the briquettes were dried in the sun for two weeks.

Determination of physico-chemical and combustion properties of briquettes

Ash content, moisture content, fixed carbon, and volatile matter of the dried matter and briquette sample were determined according to the specifications ASTM D-3172 (ASTMS 2021). The calorific system of briquette photographs is obtained using the LECO AC 350 bomb calorimeter in the system design (ASTMS 2019). Density was determined by DIN 52182 and specifications 51731 published elsewhere (Krizan et al. 2009). The compressive strength capacity of the briquette sample was determined using the ELE tritest 50 compression machine according to specifications D 3173-878 (Wilaipon 2007). The boiling water test was performed by recording the time set for the maximum amount of 500 g of briquette to start boiling 500 ml of water at various meeting conditions.

Elemental composition

Based on the results of the proximate evaluation, the primary sources of common elements such as carbon (C), hydrogen (H), and oxygen (O) for briquettes were estimated using Equations (1), (2), and (3), respectively. These were determined at an estimate of 95% confidence level (Jigisha et al. 2007):

$$C = 0.637\ F c + 0.455\ V m \quad \ldots\ldots (1)$$
$$H = 0.052\ F c + 0.062\ V m \quad \ldots\ldots (2)$$
$$O = 0.304\ F c + 0.476\ V m \quad \ldots\ldots (3)$$

Figure 1. A. Bean chaffs; B. Manual piston briquetting machine, C. Untreated briquette samples, D. Treated briquette samples

Energy dispersive x-ray fluorescence (EDXRF)

EDXRF is a non-Functional tool for the measurable and qualitative determination of main and trace elements in a wide range of sample types (Jyothsna et al. 2020). The EDXRF measurement was performed with the following experimental parameters values: side window tube, high voltage tube: max. 30 kV; emission current: max. 1 mA; power: max. 9 W; air cooled (Ion et al. 2007). The Capacities of X-Ray emitted are detected using Si(Li) and processed by a high Pulse Analysis (Jyothsna et al. 2020).

FTIR (Fourier Transform Infrared Spectroscopy)

Infrared bands of alkali-treated and untreated bean chaffs were measured on AVATAR 330 Fourier Transform Infrared (FT-IR) Spectrophotometer.

Scanning electron microscopy

The analysis of the microstructure of alkali-treated and untreated bean chaffs were evaluated by Scanning Electron Microscopy (SEM). The samples were first transformed into capsules, coated with Palladium (Pd) at 30 mA, and analyzed in a JEOLJFC-5510LV Scanning Electron Microscope.

Statistical analysis

The sum of all the parameters analyzed was calculated by two T-tests (Statistical significance determined using the Holm-Sidak method, with alpha=5.000%.) using Graph Pad Prism® (Version 6.04) and results were presented as mean ± SEM.

RESULTS AND DISCUSSION

Alkaline pretreatment is one major practice that helps improve biofuel quality through bond loosening and fractionation of the biomass matrix, thereby bringing about an effective separation between the lignin and carbohydrates (Zhao et al. 2008). This increases the hemicellulose and cellulosic contents of the biomass. Also, it helps to increase the internal surface area of biomass through adequate disruption of lignin structure and delignification and decreases the degree of crystallinity and polymerization of the potential solid fuel (Zhao et al. 2010; Conde-Mejia 2012). The observed result of the lignocellulosic contents of the untreated (UBC) and alkaline treated bean chaff (TBC) was presented in Figure 2. This shows that alkali pretreatment increases the cellulose and hemicellulose contents of the treated samples. Recorded mean values of 43.20±0.30% (TBC) and 9±0.10% (TBC) were observed compared to the untreated raw bean chaff with a mean value of 40.60±0.02% (UBC) and 8±0.43% (UBC) for the cellulose and hemicellulose contents respectively. Furthermore, the lignin contents of the treated sample were observed to reduce compared to the untreated, as shown in Figure 2. The recorded mean lignin contents of UBC and TBC were 10±0.01 and 8±0.01%, respectively. However, suggested that alkali-pretreatment of TBC results in delignification and increases the cellulose and hemicellulose contents of the biomass sample (Zhao et al. 2010; Bensah and Mensah 2013).

The proximate composition analysis is a determinant of the potentials hidden in any biomass samples and as such, its report helps to know if a supposed biomass sample can be harnessed as an alternative source of energy. Figure 3A shows the proximate composition of the untreated and alkaline treated bean chaff before briquetting, while Figure 3B shows the proximate composition of the untreated and alkaline treated bean chaff briquettes.

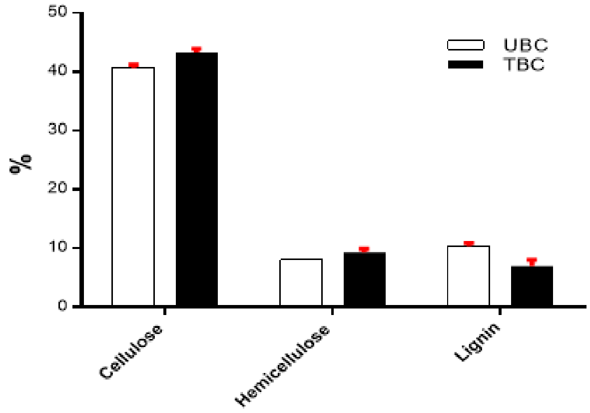
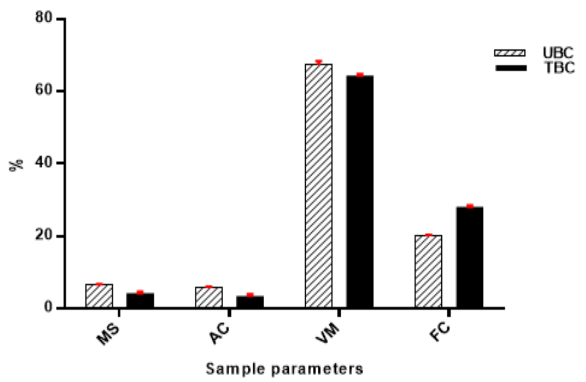


Figure 2. Lignocellulosic contents of the untreated and alkaline treated bean chaff



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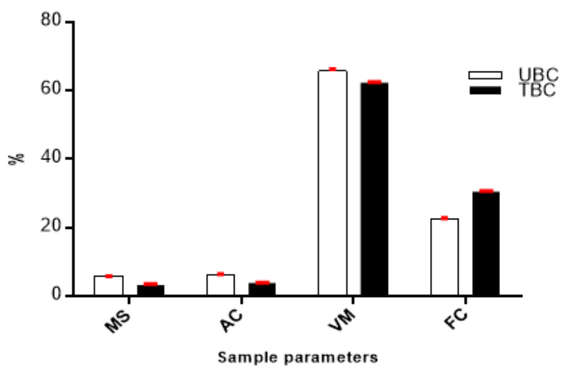


B

Figure 3. A. Proximate composition of the untreated and alkaline treated bean chaff before briquetting, B. Proximate composition of the untreated and alkaline treated bean chaff briquettes

The combustion strength of any biofuel determines the probability of its usability of such for its intended purpose. Such moisture content is the key to this because it measures the degree of dryness or wetness of the briquette (Demirbas 2013; Abayomi et al. 2021) and the shelf life of a briquette sample (Rapheal et al. 2018). Figure 3A shows a mean value of 6.52±0.02% recorded for UBC and 4.41±0.10% for TBC at $p<0.05$ before briquetting. However, a reduction in the moisture content value of the briquette was observed to be smaller at a mean value of 3.52±0.10 % for TBC briquette compared to 5.81±0.01 % observed in the UBC briquette samples. Although, all the samples- both treated and untreated raw and briquette chaff- were observed to be within the acceptable range value of 10-15% moisture content reported by (Maciejewska et al. 2006). This study's result is lower than 9.35±0.17% recorded for treated banana leaves (Bamisaye and Rapheal 2021) and 8.08%, as reported by Deepak et al. (2019). This shows that both the treated and the untreated raw and briquette samples can be considered good biomass and can be used as a solid fuel. The treated briquette sample (TBC briquette) is the best alternative due to its lowest moisture content of 3.52±0.10 %, as shown in Figure 3B, and will have a positive effect on the stored energy value of the briquette.

Determining the ash content of any combustible solid fuel is very important because it gives information about the potential pollution that could result from burning such material. It is a good pointer to environmental pollution management. The ash content and the EDXRF are very important to prevent the potential public health challenges resulting from the usability of any fuel for industrial or domestic purposes. Reports have shown that the ash content value of any biofuel should not exceed 20 % and must be between 5 to 20%. This shows that a low ash content value (that is, <20%) briquette is better quality. Figure 3 shows a mean ash content value of 6.20±0.03% for the UBC of briquette compared to 3.93±0.20% TBC briquette. However, the raw TBC sample recorded a mean value of 3.10±0.01%, as shown in Figure 3B. Upon comparing the percentage ash content of TBC of the raw chaff and briquette, the result shows that briquetting (densification) does not literarily have a significant effect on the ash content value in this sample. However, alkali treatment does, with a good observable difference between the treated and untreated briquette and chaff sample, as shown in Figure 3. This suggests that the treated samples (TBC) raw chaff and briquette with lower ash content value, as shown in Figure 4, are considered to be of better quality than UBC for both the raw chaff and the briquette. The percentage ash content value of this study is lower compared with the observed to be lower when compared with the ash contents of 13.17±0.13% and 15.71±0.29%, which were recorded for both treated starch-bonded and paper-bonded banana leaves, respectively (Bamisaye and Rapheal 2021).

An observable difference in the volatile matter content values of 64.50±0.02% and 67.40±0.30% were recorded for both UBC and TBC at $P<0.05$, respectively, for the raw chaff, as shown in Figure 2. Likewise, for the briquettes sample, a recorded mean values of 62.29±0.01% (TBC) and 66.25±0.05% (UBC). Reports have shown that the rate of combustion and the stored energy released by a solid fuel could be affected by the percentage of the volatile matter content of such biomass (Maninder et al. 2012; Rapheal et al. 2018). This shows that the higher the volatile content values of solid fuel, the faster its ignition rate and the faster it would burn off. This, therefore, shows that the UBC of the raw chaff will burn off faster compared with the TBC briquette. Aside from alkali pretreatment, densification could also be observed to improve the TBC's combustibility property, as shown in Figure 3A, with 64.50±0.02% recorded for TBC chaff and 62.29±0.01 for TBC after briquetting.

There is a highly significant difference in the observed fixed carbon content of the treated and untreated biomass samples; for both the raw chaff and its briquette counterpart. The fixed carbon content of TBC after briquetting was observed to be the highest at a mean value of 30.26±0.01% compared to 21.74±0.12% for UBC for the briquette sample, as shown in Figure 3B. Densification and alkali pretreatment was observed to have improved the quality of the FC of the biofuel. The study, however, shows that the briquette with the highest FC value will have a correspondingly high rate of energy release and a lower time of cooking compared to the ones with low FC value, which are the UBC of both untreated biomass of the raw chaffs and the briquettes samples as shown in Figure 3.

The physical characteristics and combustion properties of untreated and alkaline-treated bean chaff briquettes, including the density, compressive strength, combustibility test and the time taken to burn to ashes, were presented in Table 1. The density and compressive strength are determinant parameters for the compactness of solid biofuel. In addition, they determine the shelf life of briquettes. This means that a solid fuel with a considerably high density or compact value will last longer in storage which can aid its transportability without any observable compromise in the integrity of the stored energy value of the biofuel. The recorded high density and compressive strength values of 0.71±0.10 N/mm^2 and 1.11±0.20 g/cm^3 in the alkali-treated (TBC) sample compared to the UBC value shown in Table 1 can be attributed to the reduction in diameter of the fiber matrices of the biomass samples. This corroborates the result obtained in Figure 2. Thereby confirming that delignification, which is the disruption and restructuring of the biomass matrices, has occurred, and the impact of these could be observed in the time taken to burn to ashes in which TBC recorded a value of 14.12±0.02 min compared to 12.40±0.14 min to boil 500 cm3 recorded for UBC. This, therefore, shows that alkaline treatment improved the quality of the solid biofuel produced from raw bean chaff through an observable increase in fixed carbon content, density, and calorific value, which are essential to the heat energy of a biofuel.

Table 1. Physical characteristic and combustion properties of untreated and alkaline treated bean chaff briquettes

Sample/parameters	UBC	TBC
Density (g/cm^3)	0.52±0.05	0.71±0.10
Compressive strength (N/mm^2)	0.72±0.03	1.11±0.20
Water quantity (cm^3)	500	500
Combustibility test (min)	6.21±0.40	4.57±0.40
Time taken to burn to ashes (min)	12.40±0.14	14.12±0.02

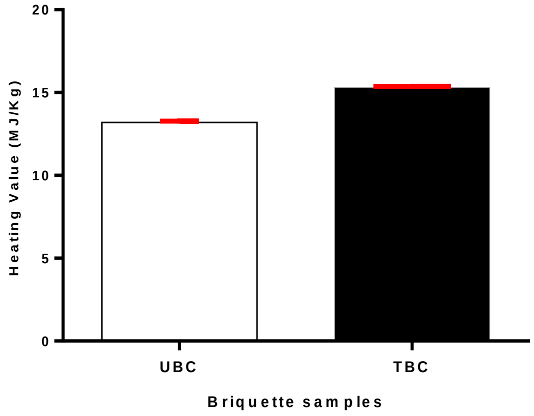
The amount of stored heat energy in the briquette sample is shown by the measure of its heat value, otherwise known as the calorific value (Santhebennur and Jogttappa 2012). The raw and briquette samples' heating values are presented in Figures 4A and 4B, respectively. Mean high heating values of 24.18±0.12 and 21.12±0.01 MJ/kg were recorded for both the TBC and UBC of the briquette samples shown in Figure 4B compared to the raw chaff with a low mean value of 15.41±0.10 (TBC) and 13.17±0.01 MJ/kg (UBC) shown in Figure 4B. The results are compared well with Brand and Jacintho's (2017) report, in which a calorific value of 17.98 MJ/kg was recorded, and Lubwama and Yiga (2017) recorded a value of 23 MJ/kg were recorded. This study showed an observable high significant difference between the heating value of the raw chaff and the briquette produced from bean chaff which is suggestive of being as a result of densification improved the heating capacity of a solid biofuel due to the compactness of the fiber matrices resulting to low porosity as a result of delignification.

The ultimate analysis of both UBC and TBC is presented in Table 2. The result indicated that all the analyzed parameters, including carbon, oxygen, and hydrogen contents, increased in the treated samples compared to the untreated, as shown in Table 2. However, the differences in the results of the obtained values of oxygen and hydrogen contents of UBC and TBC were minimal compared to the large difference in the mean value observed in the percentage carbon content of UBC and TBC. The recorded mean values of 43.60±0.02% were observed for carbon content in UBC compared to TBC with a mean value of 47.35±0.10%, which was higher than the untreated sample (Matali et al. 2016). Also, the ultimate analysis results were corroborated by the EDXRF results shown in Figure 4B compared to Figure 4A, in which the observed counts of carbon were observed to be slightly below 1000 counts in the UBC spectrum compared to the TBC spectrum in which carbon counts was observed to be above higher than 1000 counts (Figure 4B) This have an impact on the heating capacity and fixed contents of the biomass samples of the raw bean chaff prior briquetting and after briquette production as shown in Figure 3A and 3B, respectively.

Table 2. Ultimate analysis of untreated and alkaline treated bean chaff

Samples	C%	O%	H%
UBC	43.60±0.02	38.25±0.01	5.24±0.30
TBC	47.35±0.10	39.29±0.01	5.47±0.02

Note: Data are means of three replicates (n= 3) ± SEM using Graph Pad. Prism, t-test. UBC: Untreated Bean Chaff, TBC: Treated Bean Chaff



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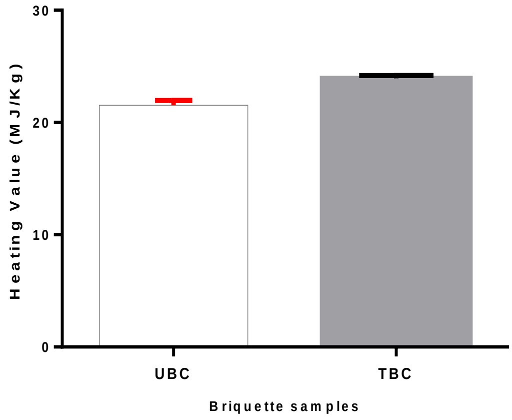


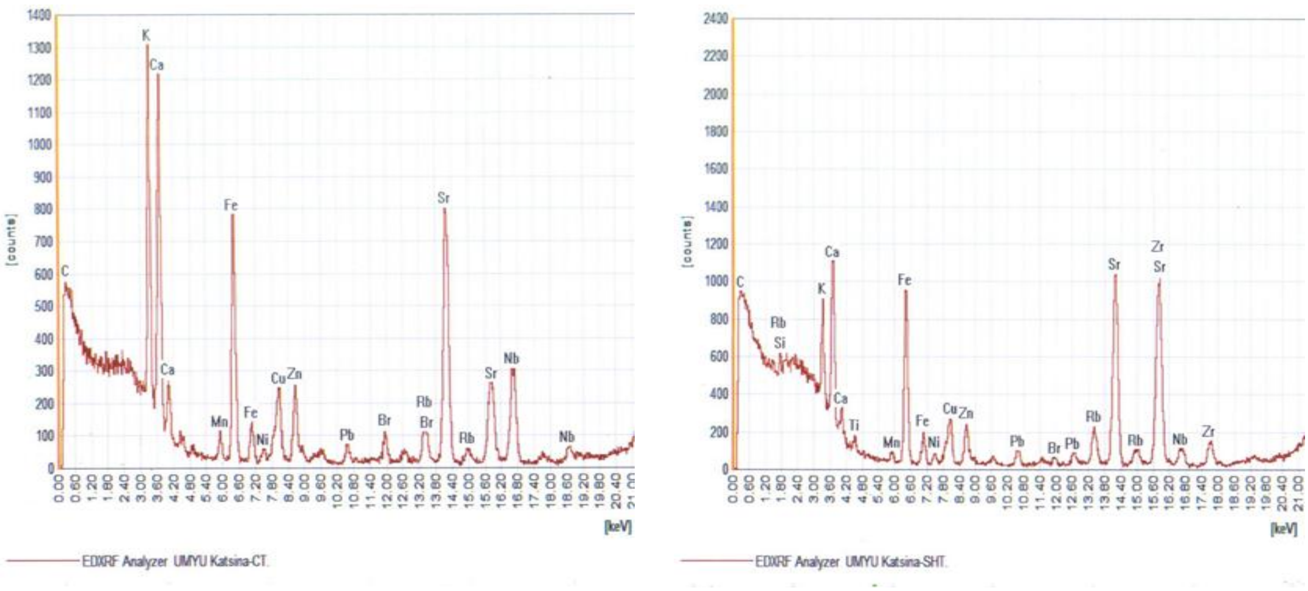
B

Figure 4. A. Heating value untreated and alkaline treated bean chaff, B. Heating value untreated and alkaline treated bean chaff briquettes

Table 3. Elemental concentrations of untreated and alkaline treated bean chaff

Elements	K	Ca	Mg	Fe	Na	P	S	Pb	Sn	Si	Nb	Ba
UBC (%)	1.45	1.23	0.04	0.05	0.00	0.20	0.09	0.18	0.00	0.36	0.17	0.17
TBC (%)	0.63	1.10	0.05	0.05	0.29	0.18	0.07	0.40	0.07	0.65	0.05	0.27

Note: UBC: Untreated Bean Chaff, TBC: Treated Bean Chaff



A B

Figure 5. A. The EDXRF Spectrum of Untreated Bean Chaff (UBC), B. The EDXRF Spectrum of alkaline Treated Bean Chaff (TBC)

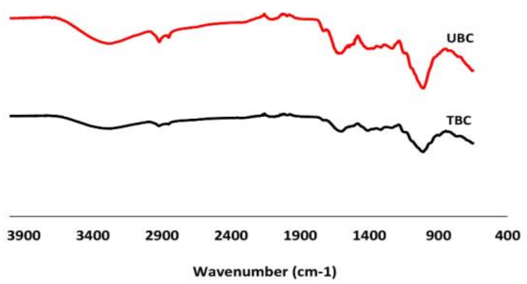


Figure 6. Showing the FTIR spectrum of untreated (UBC) and alkaline-treated bean chaff (TBC)

Table 4. Showing the FTIR vibrational stretch of untreated (UBC) and alkaline-treated bean chaff (TBC)

S/n	UBC (cm^{-1})	TBC (cm^{-1})	Assignment
1	3280	3268	O-H
2	2918	2922	Methylene Ass/Sym
3	2851	2851	Sp^3 stretch
5	1733	-	Aldehyde, C-O-H
5	1606	1601	NH_3 deformation
6	1010	1013	C-O
7	-	764	C-H, bending vibration

The EDXRF is one of the important analytical non-destructive multi-element analyses for determining trace or potential toxic elements (PTEs) in biomass samples. The major application of this technique in this work is to determine the amount of PTE at their trace level in a potential solid fuel. Thus, monitoring the rate of potential environmental pollution and health hazards that could arise from the continual usage of such fuel. This study shows that alkaline pretreatment improves the biomass's combustion properties and fuel quality, as shown in Table 3. On the other hand, the PTEs that are sulfur (s), lead (Pb), tin (Sn), and niobioun (Nb) tend to cause public health havoc; eye, skin, brain, and respiratory tract diseases (Ali et al. 2019), were observed to have been reduced drastically as shown in Table 3. Also, the carbon content, which determines the efficiency of combustibility and one of the potential parameters that determine the usage of any biomass for fuel purposes, was observed to have increased due to alkali pretreatment, as shown in Figure 5 for UBC and TBC, respectively.

The FTIR analysis was used to assess the surface modification and restructuring in both treated and untreated samples. The results of this analysis are presented in Table 4. The absorption band in the range of 3570-3200 cm^{-1}, which is attributed to the broad hydrogen-bonded (O-H) group, was observed in both UBC and TBC samples. However, a value of 3268 cm^{-1} was recorded in the OH absorption band of TBC compared to 3280 cm^{-1} in UBC, as shown in Table 4. This suggests that NaOH treatment results in bond fractionation or breaking in the fiber matrix. Furthermore, an absorption band in the range of 1026-1000 cm^{-1} is majorly a characteristic absorption band resulting from C-O stretch in cellulose, hemicellulose and lignin (Akhtar et al. 2016; Bamisaye and Rapheal 2021). A corresponding shift in the C-O stretching with a wave number value of 1013 cm^{-1} observed in TBC compared to 1010 cm^{-1} recorded in UBC could be attributed to delignification due to alkali treatment, as shown in Figure 6.

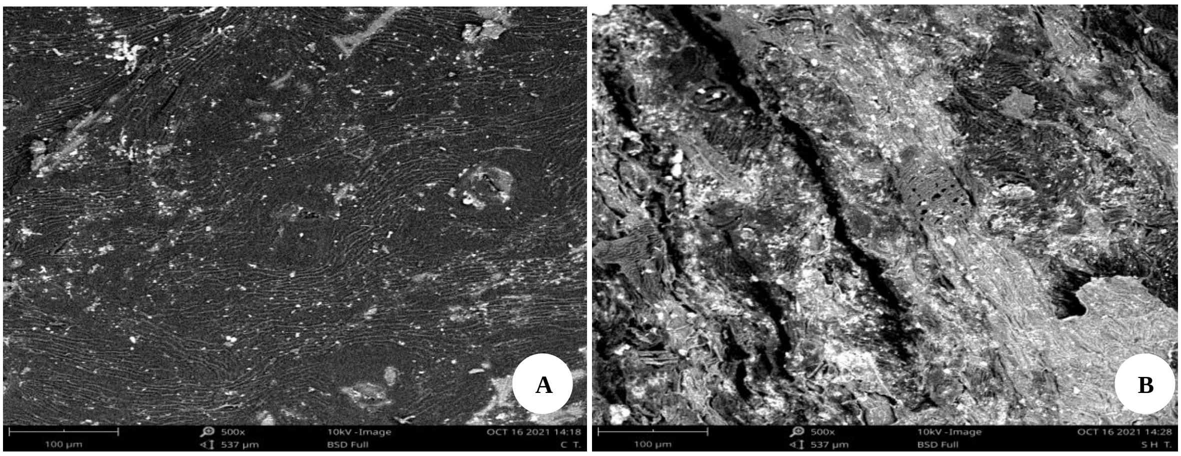


Figure 7. The SEM micrographs of: A. Untreated bean chaff, B. Alkaline Treated bean chaff

The SEM micrograph of untreated bean chaff (UBC) and treated bean chaff (TBC) were presented in Figure 7. The result showed a high degree and clear disruption in the biomass matrix, which could be attributed to the penetration of NaOH in the hemicellulose complex (OH group) in the treated sample, thereby providing a better tackiness and reduction in the lignin content of the biomass compared to 7A in which minimal or no disruption was observed (Bamisaye and Rapheal 2021). However, this corroborates the result obtained in Figure 7B, in which delignification was observed due to the low percentage of lignin contents of the biomass sample of TBC, and also the FTIR result that established that modification, that is, a disruption in the biomass matrix has occurred due to alkali pretreatment compared to the untreated counterpart of the biomass samples.

In conclusion, this study compared the combustion properties of the treated and untreated raw bean chaff and the briquette samples produced with this biomass. The proximate analysis of both the raw chaff and briquettes and the heating value of the bean chaff were determined before and after alkali treatment which improved the quality of the biomass sample. The study's finding reveals that all the essential parameters showed that alkali pretreatment reduced the percentage of Potential Toxic Element (PTE) concentration in the treated sample, as shown by the EDXRF result, thus producing an environmentally friendly briquettes sample. Also, the observed high mean heating value, fixed carbon content, compressive strength, and density of 24.18±0.12 MJ/kg, 30.26±0.01%, 1.11±0.20 N/mm^2 and 0.71±0.10 g/cm^3, respectively of the treated biomass samples compared to the untreated sample was due to the alkali pretreatment process. Furthermore, FTIR analysis was used to confirm bond disruption in the fiber matrices by alkali treatment resulting in delignification at a value of 8±0.01% in TBC with an observed vibrational C-O stretch of 1013 cm^{-1} in the alkali-treated sample (TBC) to 1010 cm^{-1} in UBC. Removing lignin components from the feedstock was necessary because a manually fabricated briquetting machine was adopted. This improved the compactness and porosity, thus showing improved combustion properties of the briquettes sample. In conclusion, alkali-treated briquettes possess better characteristics of a solid biofuel than their untreated counterpart.

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