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Physicochemical Analysis of Bambara Groundnut Shell (BGS) for Biofuel Production

Mustapha Danladi Ibrahim[a,c], Suyin Gan[a], Yousif Abdalla Abakr[b], Lai Yee Lee[a], Suchithra Thangalazhy-Gopakumar[a,*]

[a]Chemical & Environmental Engineering, Faculty of Science and Engineering, University of Nottingham, Jalan Broga, 43500 Semenyih, Selangor, Malaysia
[b]Mechanical, Materials & Manufacturing Engineering, Faculty of Science and Engineering, University of Nottingham, Jalan Broga, 43500 Semenyih, Selangor, Malaysia
[c]Department of Chemical Engineering, Faculty of Engineering and Engineering Technology, Abubakar Tafawa Balewa University, Bauchi State, Nigeria
Suchithra.Thangalazhy@nottingham.edu.my

Fossil fuel dependency and global warming drive the need for an alternative and renewable energy source. The Bambara groundnut shell (BGS) is an underutilized biomass for energy applications. The physicochemical analysis of biomass is highly significant in understanding its energy potential, influencing technological processes and environmental concerns. This study focused on the physicochemical and thermogravimetric analysis of three genotypes of Bambara groundnut shells (BGS-G1, BGS-G4 & BGS-G5). Bambara groundnut shells (BGS): (Ex-Sokoto-Cream (BGS-G1), IPB Bam 6-Black (BGS-G4), Songkhla 1-Red (BGS-G5)) were collected from the Crop for The Future Field Research Centre, University of Nottingham Malaysia. The results showed that BGS-G1 had the highest HHV of 18.6 MJ/kg, with the lowest ash content (AC) of 6.8 ± 0.5 wt% and the highest volatile matter (VM) of 74.6 wt% among three genotypes. The ultimate (CHNSO) analysis recorded that the BGS-G1 had 43.9, 6.1, 1.3, 0.1, and 41.7 wt%. The BGS-G1 was the most suitable for pyrolysis or other thermo-chemical conversions among the three genotypes studied.

1. Introduction

Renewable energy sources such as biomass are primarily carbon-neutral and assured a role in solving climate change challenges. Biomass appears as an essential role-playing fuel in several national and international policies. The future of biomass energy conversion is quite optimistic. Agro residues have an enormous scope to produce biofuels, chemicals, and biomaterials and contribute to the global economy.

Although biomass has inherent energy potential, there are some challenges to recovering its energy due to its heterogeneous nature as it varies from industrial wastes to wood, agricultural residues, or energy crops. The physicochemical analysis of biomass is highly significant in understanding its energy potential as each has its peculiar properties, influencing technological processes and environmental concerns. Some concerns are bulkiness, high moisture content, and CO_2 and SO_2 emissions; however, the established thermochemical processes are robust and flexible in co-firing biomass feedstocks for energy.

Fuel characterization is essential for thermal conversion processes, such as combustion, gasification, and pyrolysis. The chemical properties on fuel aspects are ultimate or elemental analysis (C, H, N, S, and O), proximate analysis (moisture, ash, volatile, and fixed carbon), and Higher heating value (HHV). The proximate analysis estimate the feedstock efficiency for the power generation and the yield of products (char, oil and gas) in thermal conversion systems (Torquato et al., 2017). Proximate properties affect the combustion behavior. High moisture content decrease the combustion yield, while low fuel ratio (FC/VM) favours ignition (Pitak et al., 2021), employing significantly elevated temperatures (900 °C), particularly in the case of volatiles determination (Torquato et al., 2017). On the other hand, ash profoundly influences corrosion, and slag formation, in the reactor and accumulates both facilities and management costs.

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Shrivastava et al., studied physicochemical analysis of seven biomass from oil palm industry. The lignocellulosic biomass had MC, VM, FC, and AC were in the range of 8.1-11.9, 68.3-80, 12.8-26.1 and 1.4-14.8 wt% respectively. Whereas oil palm sewage sludge has different properties as compared to other biomass. C, H, N, S and O content of lignocellulosic biomass varied as 45.6-53.3, 6.2-6.9, 0.2-2.4, 0.01-0.2 and 32.6-44.6 wt% respectively. HHV ranged in between 15.8-18.7 MJ/kg for lignocellulosic biomass (Shrivastava et al., 2021). According to La Villetta et al., the performance analysis of a woodchips biomass had MC, FC, AC, and VM as 13.74. 20.8, 0,51, and 78.69 wt% dry basis, while the CHNSO recorded as 46.6, 5.08, 0.04, 0.015, and 47.76 wt% respectively. It favours a maximum of 22.5% electrical efficiency against the global electrical efficiency of about 13.5%, but the inefficiency in gasification section was affected as the lower heating value of the syngas was 3731 kJ/Nm^3 and recorded a cold gas efficiency was 57–60%.(La Villetta *et al.*, 2018).

The current project studied the bioenergy potential for one of the underutilized crops, Bambara groundnut, that survives in minimum rainfall and generates shells (BGS). Bambara groundnut is a grain legume grown mainly by subsistence farmers in sub-Saharan Africa, such as Nigeria. Its pods are subterranean, hard, and produce justifiable yields in low fertility. Its seed is black, dark brown, red, creamy, white, a mixed colour depending on the varieties; approximately 1.5 cm long, slightly oval, often wrinkled at the matured stage, and containing 1–2 seeds. They are highly cultivated and indigenous to sub-Saharan Africa. Bambara groundnut (*Vigna subterranean* (L) Verdc) is an underutilized legume seed for consumption in various meals. Its composition of protein, carbohydrate, lipid, fibre, and ash are 16–25 %, 42–60 %, 5–6 %, 4.8 %, and 3.4 %. Legumes like Bambara has the capacity for atmospheric N-fixation and climate change mitigation without expensive inorganic Nitrogen-fertilizers, which are also not readily available to local farmers. Bambara groundnut (*V. subterranean*) is an indigenous African crop and is the third most important legume. BG is widely grown in Borno, Anambra, Plateau, Taraba, Sokoto, Bauchi, Benue, Kano, Yobe, Adamawa, and the Gombe State, with the least land area of 17,802 km^2 in Northern Nigeria. In Zambia, Bambara groundnut flour is also for baking bread and producing flavored milk that tastes better than cowpea, pigeon pea, and soybean. They are suitable for food and medicinal purposes (Atiku et al. , 2004). The potential yields of Bambara groundnut at its cultivation can increase significantly without high agronomic inputs. Bambara ground nut is an underutilized leguminous crop for global food security and nutrition.

Agriculturists commonly grow BGS in marginal land. BGS survives better than other tropical legumes, e.g., cowpea and groundnut. Bambara groundnut constitutes pod weight which is about 1000 kg ha^{-1}, 374–896 kg ha^{-1} of the total nut weight and the total pod weight is within 34.2 %. In order to curtail pollution and fossil fuel challenges, it could be harnessed for green energy (Mohammed et al., 2016).

Only BGS-G1 was taken for thermo-chemical conversion studies among all genotypes (Mohammed et al., 2016, Ibrahim, et al., 2022). In this study, other two genotypes of BGS; IPB Bam 6-Black (BGS-G 4), Songkhla 1-Red (BGS-G 5) were taken in addition to Ex-Sokoto-Cream (BGS-G1). In establishing biomass energy potential, this work aimed to investigate and evaluate the physicochemical analysis through HHV, ultimate, and proximate analyses of Bambara groundnut shells (BGS)**.** The thermogravimetric analysis (TGA) was studied to understand the pyrolysis behaviour.

2. Materials and methods

2.1 Biomass sample collection

The Bambara groundnut shells (BGS) (Ex-Sokoto-Cream (BGS-G1), IPB Bam 6-Black (BGS-G4), Songkhla 1-Red (BGS-G5)) were collected from the Crop for The Future (CFF) Field Research Centre (FRC) University of Nottingham, Malaysia. The collected BGS-G1, BGS-G4, and BGS-G5 were ground using an impact rotor mill (model Retsch SM100), sealed in a transparent plastic sample holder and stored in the laboratory for further analysis.

2.2 The ultimate (CHNSO) and proximate analysis

The elemental composition of carbon, hydrogen, nitrogen and sulphur (C, H, N, and S) was performed in an elemental analyzer (Vario MACRO Cube, Elementar). A measurement of about 0.1 mg was placed into a tin capsule and dropped into the elemental analyzer. In an elemental analyzer, the catalytic combustion would happen in excess O_2. The combustion should take place as a complete conversion of the C, H and S to CO_2, H_2O, and SO_2 , while all of the N content should reduce to gaseous N_2. These products can be quantitatively analyzed using a suitable system, such as thermal conductivity detectors or flame ionization chromatography (Madanayake et al., 2017). Usually, oxygen measurement is indirect, as the weight estimation is by the subtraction of the concentrations of all other elements (C, H, N, S) and ash content from 100 %**.**

The higher Heating Value (HHV) was determined in accordance with British Standard No. BS4379 in a bomb calorimeter (Parr 6100). The moisture content (MC) was determined in the oven. 1 g of shredded biomass wasplaced in a convection oven at 105 ± 3 °C for 16 hours. The dish (s) that contained the sample were taken out of the oven, placed in a desiccator, cooled to room temperature, and reweighed. The volatile matter was ascertained by heating approximately 10 to 15 mg in a TGA at a constant temperature of 10 °C/minutes. The study was carried out in N2 at a flow rate of 20 mL/minutes flow rate from 30-110 °C and held at 110 °C for 10 minutes (for moisture removal). The samples were further heated between 110-900 °C and at 900 °C and held for another 10 minutes. The calculated volatile matter was the per cent weight loss from 110 to 900 °C. The ash content (AC) of biomass determination was in a muffle furnace, with a 1 g sample placed in a crucible, heated at 575 ± 10 °C for 4 h, at a ramp of 10 °C /minutes. As the ash's crucible was weighed to the nearest 0.01 g, it was carefully placed in a desiccator and cooled to room temperature. The fixed Carbon (FC) content was calculated using the Equation (1):

$$FC = 100 - (MC + VM + AC) \tag{1}$$

2.3 Thermogravimetric Analysis (TGA)

Thermogravimetric analysis was conducted in STA6000 TGA-DSC (Perkin Elmer) to study the kinetic parameters such as activation energy and pre-exponential factor of the pyrolysis of samples. The samples were heated at 10 °C/ minutes between 30 to 950 °C temperature under a nitrogen atmosphere at a 20 mL/minutes flow rate. The kinetic parameters for the thermal degradation of the samples can be determined by the Doyle's method as in Equation (2) for simplicity. The pre-exponential factor (A) and the activation energy (E_a) can be evaluated from the slope and intercept of the regression line of ln[-ln(1-α)] against 1/T, respectively.

$$\ln[-\ln(1-\alpha)] = \ln\left(\frac{AE_a}{\beta R}\right) - 5.33 - 1.052\frac{E}{RT}\ \cdot \tag{2}$$

where A is the pre-exponential factor, min^{-1}; E_a is the activation energy, J.mol^{-1}; β is the heating rate, $°\text{C min}^{-1}$; T is the absolute temperature, K; R is the universal gas constant, $\text{J.mol}^{-1}.\text{K}^{-1}$; α is the degree of conversion of the sample at any time.

3. Result and Discussion

3.1 The proximate analysis

Biomass with high moisture content suits biochemical methods, including fermentation, anaerobic digestion, wet torrefaction, or hydrothermal carbonization. Woody biomass has lower moisture than herbaceous biomass. Most industrial biomass applications have thermochemical processes that utilize woody biomass and low-moisture varieties of herbaceous biomass. The results in Table 1 are within the operation conditions for thermochemical processes.

Table 1: The results of the BGS-G1, BGS-G4, and BGS-G5 proximate and ultimate analysis

		Proximate analysis (wt%)				Ultimate analysis (wt.%)				
Sample	HHV (MJ/kg)	MC	AC	VM	FC	C	H	S	N	O
BGS-G1	18.6	8.6 ± 3.0	6.8 ± 0.3	70.3	18.7	43.9	6.1	0.1	1.3	41.8
BGS-G4	16	11.7 ± 0.5	9.1 ± 0.1	62	17.2	40.8	5.6	0.2	1.5	42.8
BGS-G5	15.7	10.2 ± 0.5	10.1 ±0.3	64.2	15.5	39	6.1	0.2	2.1	42.5

At a relatively low heating rate, cellulose degrades to rather stable anhydrous cellulose resulting in charring of high char, but at a high heating rate, rapid volatilization of cellulose produces volatile products. On heating biomass, volatile biomolecules of biomass are cleaved, producing the bio-oil after condensation (Tripathi et al., 2016) . Table 1 indicates the BGS-G1 recorded the highest VM of 70.3 wt%. The HHV is related to high fixed carbon (FC) and carbon contents which are the primary heat source (Onokwai et al., 2022). In Table 1, the maximum HHV biomass of BGS-G1 (18.6 ± 0.5 MJ/kg) with the highest FC. The HHV values of the BGS were high and similar to those found in coals (16–34 MJ/kg) (Pacioni et al., 2016). The HHV of the herbaceous biomass competed with the most agro-residues and woody plants. For example, a palm kernel shell recorded 18.82 MJ/kg HHV with a carbon content of 48.78 wt% (Shahbaz et al., 2017), whereas *Silphium perfoliatum* L. and *Helianthus salicifolius* A. Dietr. (perennial herbaceous crops) had HHV of 17.72 and 18.44 MJ/kg (Peni et al., 2022). The HHV of samples can be determined using the modified Dulongs formula, as mentioned below in Equation (3). C, H, and O represent the samples' carbon, hydrogen, and oxygen contents, on a dry basis expressed in mass percentages.

$$HHV\ (MJkg^{-1}) = [33.86 * \mathrm{C} + 144.4 * \left(\mathrm{H} - \frac{\mathrm{O}}{8}\right) + 9.428 * \mathrm{S}]/100 \quad (3)$$

The validation of the HHV using Equation (3) shows a confirmation of the experimental results BGS-G1, BGS-G4, and BGS-G5 as 16.1, 14.2, and 14.4 MJ/kg with a minimum error of about 15 %. Forest waste materials have higher carbon content (44–53 %) with lower ash content (0.3–8 wt%) among lignocellulosic biomass (Raza et al., 2021). High AC of biomass affects its potential fuel desirability, while high extractive content contributes to fuel desirability. Ashes and inorganic elements are residues after biomass combustion and produce immense challenges in power plants, such as slagging, corrosion, and fouling et al., 2008). The BGS-G1 had the lowest ash content among the three genotypes.

3.2 The ultimate analysis and Van Krevelen plot

High carbon contents positively contribute to the fuels' HHV. The biomass hydrogen and oxygen contents sharply decrease due to dehydration reaction during pyrolysis. However, since the carbon content increases, higher heating values of products increase. The carbon conversion efficiency is about 60 % for the biomass during pyrolysis. Forest waste materials have the highest in carbon (44–53 %) among lignocellulosic materials (Raza et al., 2021). Table 1 shows the hydrogen of BGS-G1, BGS-G4, and BGS-G5 as 6.1, 5.6, and 6.1 wt%. The values recorded are similar to several biomass analyses or studies. BGS-G1, BGS-G4, and BGS-G5 samples had oxygen ranging between 41.8–42.5 wt%, with only BGS-G1 lower than their carbon contents. The sulphur and nitrogen are relatively small as the minimum BGS-G1 (0.1 wt%), and the maximum BGS-G5 (2.1 wt%) require a more sophisticated process to mitigate the emissions for a scale-up plant. Low sulphur and nitrogen contents indicate their potential for thermochemical conversion due to low greenhouse gas emission (Onokwai et al., 2022).

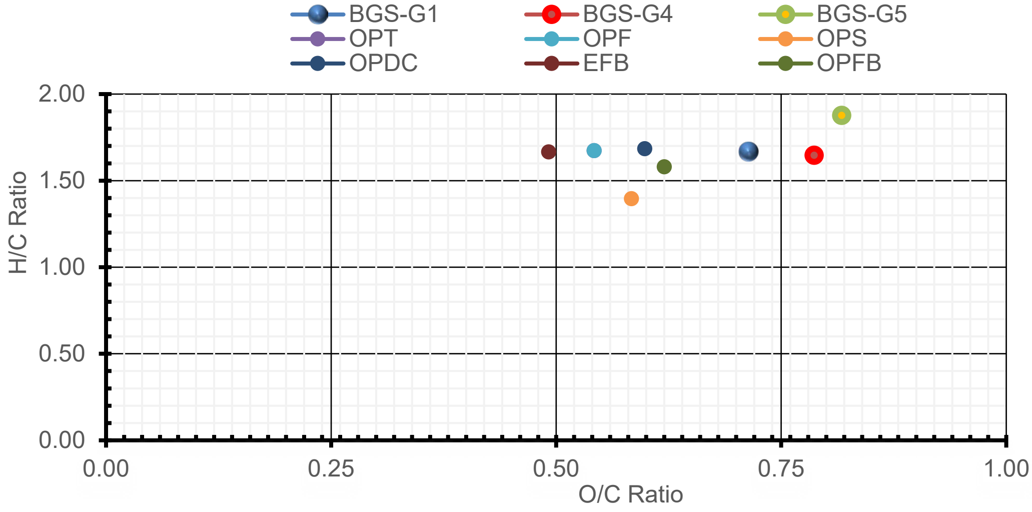

Figure 1: The Van Krevelen Diagram of BGS-G1, BGS-G4 and BGS-G5 as comparison with oil palm trunk (OPT), oil palm fronds (OPF), oil palm shell (OPS), oil palm roots (OPR), oil palm decanter cake (OPDC), empty fruit bunches (EFB), and oil palm fiber (OPFB) from (Shrivastava et al., 2021).

Van Krevelen Diagram shows that BGS-G1 is better biomass with the lowest O/C ratio, where BGS-G5 has the highest H/C and O/C. The high atomic ratio for BGS-G5 was due to its low amount of C among the three genotypes tested. The results were compared with oil palm lignocellulosic biomass from Shrivastava et al. The atomic ratios were recalculated using their ultimate analysis results (Shrivastava et al., 2021). BGS atomic ratios were within the range of lignocellulosic biomass (H/C: 1.4-1.8 and O/C: 0.6-0.8) reported by Cai et al. (Cai et al., 2017).

3.3 TGA Analysis and Kinetics

The thermal degradation profile of the selected BGS biomass samples is at the temperature ranges of 30–900 °C in Figure 2. The TGA shows that stage 1 had a temperature range between 30–190 °C, showing the moisture removal by evaporation, with a weight loss for samples BGS-G1, BGS-G4, and BGS-G5 are at an average of 10 wt%. As reported by Singh et al., the temperature of 30–175 °C, and 50–160 °C, are attributed to moisture removal and light volatiles. Stage 2 ranges between 190–620 °C, with the weight loss for hemicellulose and cellulose devolatilization. This high volatility in might result from the effect of smaller particle sizes. The temperature range (175–620 °C) at the second stage witnessed a weight loss of about 73 % which was related to the thermal degradation of hemicellulose and cellulose contents (Singh et al., 2021). The prominent peak in DTG plot attained at 320 °C was mainly due to the decomposition of cellulose (Singh et al., 2021). An EFBF and POME sludge at 20 °C/min of nitrogen lost 63.57 % of the cellulose and hemicellulose present in EFBF between 160−420 °C (Chong et al., 2017). Stage 3 had a temperature range between 620–900 °C for recording a weight loss of about 10 wt%. Lignin decomposition is slower due to a complex natural polymer of aromatic compounds to degrade with higher temperatures than cellulose and hemicellulose beyond 420 °C (Chong et al., 2017). The third region had some mineral decomposition, and it could be advisable to undergo gasification of the samples studied above 700 °C for complete biomass utilization.

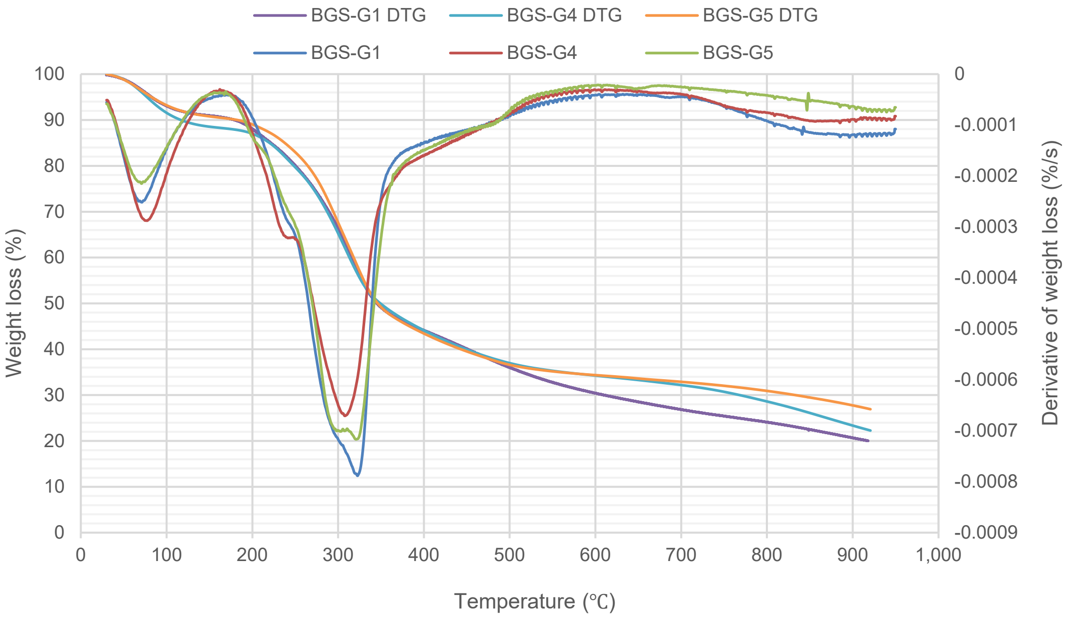

Figure 2: The TG and DTG plots of BGS-G1, BGS-G4, and BGS-G5

Table 2: Kinetic analysis for BGS-G1, BGS-G4, and BGS-G5

Sample	E_a (kJ.mol^{-1})	A	R^2
BGS-G1	24.1	50.06	0.97
BGS-G4	18.8	28.39	0.94
BGS-G5	18.9	25.47	0.94

Activation energy (E_a) and pre-exponential factor (A) were determined via linear regression from the data obtained from the thermogravimetric analysis. Activation energy is the minimum energy required before molecules can initiate the reaction. In this study, all three samples in Table 2 had almost similar activation energy, however, the activation energy of BGS-G1 had relatively higher activation energy. However, they are all lower compared to most biomass studied (Chong et al., 2017). This implies that the studied samples are more active in the degradation reaction.

4. Conclusions

Three genotypes of Bambara Groundnut Shell: Ex-Sokoto-Cream (BGS-G1), IPB Bam 6-Black (BGS-G 4), Songkhla 1-Red (BGS-G 5) were studied for their potential for thermos-chemical conversions through proximate, ultimate analyses, HHV and thermal degradation. BGS-G1 had higher FC and VM and lower AC as compared to other two genotypes. HHV was higher for BGS-G1 as compared to BGS-G4 and BGS-G5, whereas they both have almost similar HHV. Thermogravimetric analysis of the DTG/TGA described a suitable pyrolysis temperature within 190–650 °C, while the Van Krevelen diagram shows a lower O/C for BGS-G1 than the BGS

(G4 and G5). BGS-G1 was more recommendable for bioenergy than the other two genotypes BGS (G4 and G5) samples, however, the energy potential of BGS-G4 and BGS-G5 were comparable to BGS-G1.

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