



Characterization of lignocellulose biomass based on proximate, ultimate, structural composition, and thermal analysis

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ABSTRACT

This research deals with the determination of physicochemical and thermal properties of biomass samples to enhance pyrolysis yields. Proximate, ultimate, structural composition, trace elements, and thermal analyses were conducted using fifteen lignocellulose biomass samples obtained in Ajase market, Ajase Ipo, Kwara State, Nigeria, and Omu-Aran, Kwara State, Nigeria respectively. Results obtained from the ultimate analysis showed that Oxygen, carbon, and sulfur show minimum, medium, and peak effects on the HHV and LHV of biomass, while Nitrogen and Hydrogen do not positively influence the HHV and LHV. Also, for the proximate and structural composition analysis, it was observed that fixed carbon, volatile lignin, ether extracts, cellulose, and hemicellulose positively enhanced the desirability of the biomass by increasing its heating value, while moisture, ash, and corrosiveness reduced pyrolysis yield because they reduced the HHV and LHV of lignocellulose biomass.

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1. Introduction

Energy plays a vital role in most human activities; it is the premise of industrial civilization. Without energy, the modernization of our life and cities would not have been actualized [1–3]. Previously, the demand for energy sources was minimal because it was primarily utilized for cooking, heating, and transportation. However, the increase in population growth rate, coupled with technological advancement, necessitated more energy demand [4–6]. This high energy demand warranted the quest for different energy sources, though some have an advanced effect [7]. For instance, fossil fuels constitute the primary energy resource used to generate power for technological development and advancement since the industrial revolution [8].

Nevertheless, there were consequential effects. Studies have shown that the volume of pollutants produced by fossil fuels is on the high side sequel to their usage. This constitutes a health hazard to the public and the environment [9]. Therefore, research-

ers proceeded to generate energy via alternate renewable sources to overcome the 'Energy Crisis.' Among the renewable sources of energy, biomass energy offers a practical solution [2,10] because it offers about a 13% share in the global energy mix in 2016, which is the greatest renewable energy source [11,12].

More attention has been drawn to biomass, among other renewable sources of energy known to mankind due to its ability to produce biochar, bio-oil, and biofuel which reduce the energy crises beclouding mankind and environmental hazards caused by the utilization of fossil fuel for electricity generation [13]. Biomass can be generally considered as the collection of a composite mix of biological materials that include proteins, lignin, fats, and carbohydrates in the form of starch, hemicellulose, and cellulose [14–16].

The degradation of biomass into bio-oil, biochar, and *syn-gas* involve completely thermochemical conversion procedures, such as pyrolysis, liquefaction, gasification, torrefaction, and carbonization [17–19]. These thermochemical conversion yields are mainly affected by a poor determination of elemental contents (C, H, N, O, S) proximate (FC, VM, MC, Ash), and structural composition (cellulose, hemicellulose, lignin, and ether extractive) as reported by [20]. The disposal of Ash obtained from the thermochemical conversion process of lignocellulose biomass contaminates under-

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ground water resources due to heavy metal leaching [21]. Hence, this leads to respiration health problems and reduces the soil's fertility [22,23].

Rajamma et al. [24] emphasized the need to investigate the thermal degradation and decomposition behaviour of biomass before selecting them for thermochemical conversion to enhance their yields. TGA curve shows the weight loss as a function of temperature [25,26] and a distinct characteristic such as the thermal behavior of biomass [27].

Heating value is not only utilized for the design and operation of biomass conversion system but also aid the selection of biomass as fuel [28].

The utilization of an adiabatic bomb calorimeter to determine heating value is easy and accurate, but its availability is not readily accessible to researchers [29–31]. Various researchers proposed different ways of predicting the heating value of biomass based on proximate, ultimate, and Structural composition analysis [32], while [33] observed that fixed carbon, carbon, and hydrogen enhance the HHV, while ash, nitrogen, oxygen, and Sulphur values did not influence the prediction of the HHV.

This study deals with proper characterization (proximate, ultimate, trace element, and thermal analysis) of lignocellulose biomass to aid the selection of biomass as a fuel for thermochemical conversion processes such as torrefaction pyrolysis, gasification, and combustion. Hence, enhancing the quality and quantity of pyrolysis yield from the HHV.

2. Materials and methods

2.1. Source of materials

Seven lignocellulose biomass samples, namely shea butter wood, bamboo, palm kernel shell, rice husk, sugarcane straw, siam weed, and gmeliana wood, were sourced obtained locally from the sawmill, palm oil mill, and local farm respectively in Omu-Aran, Kwara State, Nigeria ($8^{\circ}08'18.85''N$ Latitude and $5^{\circ}06'9.36''E$ Longitude) while Sugarcane bagasse and Sugarcane straw were obtained from Ajasse Ipo, Kwara State, Nigeria ($8^{\circ}13'60''N$ Latitude and $4^{\circ}49'0''E$ Longitude).

2.2. Pretreatment

The biomass samples were pretreated by cleaning, crushed, and sieved to various sizes with 0.1–0.2 mm, 0.2–0.4 mm, 0.4–0.6 mm, 0.6–0.8 mm, and d_p 0.8–1.0 mm to enhance densification [34,35]. The crushed samples were then sun-dried for five days (5 h/day) to remove surface and residual water [36] and later separated from contaminants such as stones and other foreign objects [37–39] before storing them in a zip-locked polyethylene bags at ambient temperature for characterization and pyrolysis experiment.

2.3. Physicochemical analysis

2.3.1. Proximate analysis of sun-dried powered biomass

0.5 g of sun-dried biomass samples were transferred into an empty crucible, and the mass of the crucible plus biomass was weighed and recorded. The samples were heated in an oven (Moisture Analyzer- 105 MW) at 105 °C for 2hrs according to ASTM E 871–80 standard [40]. The Volatile matter (VM) was determined according to ASTM D3175 [41] by weighing an empty crucible and then placing 1 g of each biomass in a muffle furnace, maintained at 900 °C for 8 min all through. Finally, the weight was measured and recorded. Deploring ASTM standard E1755, the biomass ash content (AC) was evaluated using a muffle furnace. 1 g of biomass sample was measured into a silica crucible and later trans-

ferred into an oven (Model No: OF-22G, JESO TECH, Korea) maintained at 105 °C for an hour, after which the crucible was transferred into the desiccator to cool down to room temperature. This practice covers the determination of moisture (MC), volatile matter (VM), and ash (AC) and the calculation of fixed carbon (FC). After that, the crucible was transferred into a muffle furnace charged at 585 °C for 3 h [42,43]. The fixed carbon (FC) was determined by difference, which is $FC = 100 - (MC\% + VM\% + AC\%)$ as suggested by [44,45].

2.3.2. Ultimate analysis of sun-dried powered biomass

Carbon, Hydrogen, and Nitrogen contents were determined using LECO CHN 2000 Elemental Analyzer based on ASTM D5373 standard. Moreover, the Oxygen component was determined by difference ($100 - C\% + H\% + N\% + S\% + Ash\%$) according to ASTM 3174–76 standard [46]. About 0.5 g of the sample was measured into a crucible charged into an oven maintained at 105 °C for 1 h and encapsulated in a thin foil to fit into the LECO CHN 2000 Elemental Analyzer. After that, the sample was transferred into the purge chamber of the furnace maintained at 1300 °C for 7 min [47,48].

2.4. Structural composition analysis of sun-dried powered biomass

The biomass extractive (Ex) process was carried out in a Soxhlet extractor, using Aceton water as the solvent according to ASTM E 1721 standard. 2 g of sun-dried biomass was weighed (A) and then heated in an oven maintained at 105 °C for 2 h. Thereafter, the final sample (B) was weighed and recorded (B). Extractive was calculated by the difference, $Ex = (A - B)$. The hemicellulose content was estimated by measuring 1 g of extractives free biomass (B) into a 250 mL Erlenmeyer flask containing 150 mL of Sodium Hydroxide (NaOH). The sample was placed and heated in a furnace at a controlled temperature of 105 °C for 4 h until a constant weight was achieved (C). Hemicellulose was obtained by the difference between B and C (Adeleke et al., 2019; Akdeniz et al., 2018). The lignin content was determined by drying 1 g of extractives-free biomass (B) in an oven at a temperature of 105 °C for 1 h. until a stable weight (D) was achieved. This final residue weight is the lignin content [49]. The cellulose content (E) was calculated as the difference between the initial weight of the raw biomass sample and the other weights obtained in previous contents during the experiments [50,51].

2.5. Thermogravimetric analyses (TGA) of sun-dried powered biomass

The thermal degradation and decomposition behaviour of the biomass samples were investigated using a thermogravimetric analyzer (Model: TGA 55). Approximately 3 mg of each biomass was loaded into the crucible, and the samples were heated in the TGA analyzer from 20 °C to 800 °C at a steady heating rate of 10 °C/min under an inert environment of a continuous nitrogen flow rate of 80 mL/min [8,52].

2.6. Heating value analysis of sun-dried powered biomass

The higher heating value (HHV) was determined using a bomb calorimeter according to ASTM D2015-00 standard. About 2 g of biomass sample was measured into a crucible and placed in a high-pressure oxygen atmosphere metallic cylinder (bomb). The results were then displayed on the bomb calorimeter [53,54]. The lower heating value (LHV) was calculated from HHV using equation (1) as proposed by [50].

$$LHV = HHV - (0.218 \times H) \quad (1)$$

where, LHV = lower heating value of fuel in MJ/kg; HHV = higher heating value of fuel in MJ/kg; H = weight % of hydrogen in fuel.

3. Results and discussion

3.1. Proximate analysis

The average values of the proximate analysis are shown in Table 1. The moisture content, which is the number of water molecules contained in the biomass, ranges from $0.18 \leq 2.19$, ash content which is the incombustible inorganic minerals left after the biomass had been burnt ranged $0.94 \leq 17.89$, pH values which is the measurement of the acidity/alkalinity of the biomass ranged $9.98 \leq 12.50$, fixed carbon (FC) which is the solid combustible residue left after the biomass was heated $11.14 \leq 22.40$, while the volatile matter (VM) which is the amount of condensable and non-condensable vapour released when the sample was heated ranged $64.80 \leq 85.20$ (Table 1). The low moisture content recorded by palm kernel shell and shea butter wood reduces the densification of the biomass samples and the heat transfer process. Hence, it reduced the rate of thermochemical decomposition of the biomass samples [28,29,36]. Shea butter wood recorded the highest VM content (85.20%) closely followed by Gmeliana arborea wood (84.61%). It is essential to devolatilize the biomass before using it for the thermochemical conversion process as high VM lead to fast combustion and formation of smoke [53]. The high pH contents recorded in all the biomass samples mostly in siam weed, increased the microbial contents of the biomass, hence, favouring pyrolysis yields. Ash content of the biomasses ranged from 0.94% – 17.89% similar to the research of [28,54]. The increase in ash content was attributed to the biomass absorbing inorganic minerals within the environment. Low ash content is a reasonable requirement for energy conversion (pyrolysis, combustion, and gasification) as recorded in shea butter wood, Gmeliana wood and palm kernel shells, unlike rice husk which possesses high ash content.

Results presented for siam weed are similar to the values reported by [50] whose VM, FC, and Ash contents are 71.2, 18.4, and 4.67% respectively. The values obtained for bamboo, meliana, and shea butter wood (Table 1) are in close agreement with the findings of [5,52] who reported for woody biomass. Similarly, values presented for rice husk and sugarcane straw fall within the same range as the report of [12,38] reported.

The variations in the results when compared with previous studies are due to geographical location, the intrinsic composition of the biomass, soil type where the biomass is sourced and cultivated [13].

3.2. Ultimate analysis

Table 2 depicts the average values of the elements obtained from the ultimate analysis performed on the samples ranged from 40.82 to 50.03%, 5.25 to 6.30%, 0.10 to 0.30%, 43.54 to 53.38%, and 0.02 to 0.27% for Carbon (C), Hydrogen (H), Nitrogen (N), Oxygen (O), and Sulphur contents respectively. The low Sulphur and Nitrogen contents indicate that the biomass is useable for the thermo-

chemical conversion process, e.g., torrefaction, pyrolysis, gasification, etc. due to their low emission of greenhouse gas as reported by [29,36,37]. The Carbon recorded in and shea butter wood, palm kernel shells them favourable for the thermochemical conversion pyrolysis process [8,10,55]. The results present for Gmeliana Arborea and shea butter wood fall within the range of values for hardwood reported by Vassilev (2010) who reported (C:49.6%, H: 6.1%, N: 0.1%, O: 44.1% and S: 0.06%) and (C: 52.3%, H: 6.1%, N: 0.3%, O: 41.2% and S: 0.10%), while the values for bamboo wood closely agreed with what [38] reported.

The HHV and LHV varied from biomass to biomass because their fuel characteristics differed. The highest HHV and LHV recorded were 23.10 and 20.47 MJ/kg for shea butter wood, while rice husk has the lowest HHV and LHV at about 16.63 and 14.89 MJ/kg. The increase in the HHV and LHV was due to their high fixed carbon (FC) and carbon contents which are the main source of heat [28].

3.3. Structural composition analysis

The average structural composition analysis (Table 3) shows that the cellulose, hemicellulose, lignin, ether extract, and corrosive values of the biomass species are $25.32 \leq 45.80\%$, $20.12 \leq 30.94\%$, $19.44 \leq 45.41\%$, $0.20 \leq 0.46\%$, and $0.0001 \leq 0.002\%$ respectively. The values of cellulose, hemicellulose and lignin contents obtained for woody/forestry biomass (Table 3) falls within the range of values for hardwood reported by Saini et al. (2015) and Rowell et al. (2021) whose values are (Ce: $45 \pm 2\%$, He: $30 \pm 5\%$, Li: $20 \pm 4\%$) and (Ce: $45.4 \pm 3.5\%$, He: $26.0 \pm 3.0\%$, Li: $23 \pm 3.0\%$) respectively. The high lignin, ether extract, and cellulose content recorded in shea butter wood and sugarcane bagasse respectively would enhance pyrolysis yields [42]. Low hemicellulose content in corn cobs reduced the degradation pathway of biochar production and facilitated charring and incomplete combustion of the biomass via the pyrolysis process [56,57]. High lignin contents of the biomass would decrease the pyrolysis rate, hence an increase in the bio-char yield, while the high cellulose and hemicellulose contents increase pyrolysis rate, leading to an increase in the yield of bio-oil, NCG [58].

Fig. 1 depicts the TGA profile from the thermal reaction of Bamboo (B), Rice husk (C), Gmelina Arborea wood (G), Sugarcane straw (M), Palm kernel shell (P), Shea butter wood (S), and siam weed (W) under an inert environment (nitrogen flow rate, 1 mL/min) at a steady heating rate of 10 °C/min in the temperature range of 1 to 900 °C.

The TGA curve undergoes three phases of decomposition: the pre-heating phase (moisture evaporation), volatile devolatilization, and carbonization [59,60][60,61]. The pre-heating stage (first zone of pyrolysis) recorded approximately 6.2% weight loss up to 280 °C. During this stage, drying and decomposition of a low quantity of volatile components occurred due to the release of both free water and chemically bonded water [8,50]. The fluctuations of the TG curve were due to the biomass utilized to carry out the experiment runs which had been sun-dried and possessed low moisture con-

Table 1
Proximate analysis.

SAMPLE	MC	VM	FC	Ash	pH
Siam weed	1.12 ± 0.04	78.11 ± 0.7	15.87 ± 0.04	4.9 ± 0.02	12.50
Gmeliana arborea	2.19 ± 0.02	84.61 ± 0.8	11.14 ± 0.05	2.06 ± 0.01	10.67
Sugarcane straw	0.90 ± 0.04	77.25 ± 0.4	13.31 ± 0.06	9.60 ± 0.01	10.46
Rice husk	0.89 ± 0.05	61.80 ± 0.6	16.44 ± 0.03	20.89 ± 0.02	11.14
Shea butter wood	0.18 ± 0.03	85.20 ± 0.7	13.68 ± 0.07	0.94 ± 0.01	9.98
Palm kernel shell	0.40 ± 0.02	73.70 ± 0.6	22.40 ± 0.05	3.50 ± 0.01	10.39
Bamboo wood	1.03 ± 0.05	80.20 ± 0.1	14.13 ± 0.08	4.64 ± 0.02	11.45

**MC-Moisture contents; VM- Volatile Matter; FC- Fixed carbon.

Table 2
Ultimate analysis.

SAMPLE	C (%)	H (%)	N (%)	O (%)	S (%)	HHV	LHV
Siam weed	45.82 ± 0.3	5.79 ± 0.03	1.20 ± 0.01	46.95 ± 0.2	0.24 ± 0.03	20.40	19.14
Gmeliana arborea	47.50 ± 0.1	5.65 ± 0.03	0.27 ± 0.02	46.42 ± 0.1	0.16 ± 0.01	19.15	17.92
Sugarcane straw	44.80 ± 0.1	5.94 ± 0.03	0.10 ± 0.02	48.89 ± 0.1	0.27 ± 0.01	17.01	15.71
Rice husk	40.82 ± 0.3	5.25 ± 0.03	0.38 ± 0.01	53.38 ± 0.2	0.17 ± 0.03	16.63	14.89
Shea butter wood	50.03 ± 0.1	6.30 ± 0.03	0.11 ± 0.02	43.54 ± 0.1	0.02 ± 0.01	23.10	21.73
Palm kernel shell	48.28 ± 0.1	5.39 ± 0.03	0.12 ± 0.02	46.18 ± 0.1	0.03 ± 0.01	21.65	20.47
Bamboo wood	45.02 ± 0.3	5.91 ± 0.03	0.30 ± 0.01	48.58 ± 0.2	0.19 ± 0.001	18.86	17.57

**C-Carbon; H-Hydrogen; N-Nitrogen; O-Oxygen; S-Sulphur; HHV-Higher heating value; LHV-Lower heating value.

Table 3
Structural composition analysis.

SAMPLE	Ce %	He %	Li %	Ee %	Co
Siam weed	25.32 ± 1.06	26.60 ± 0.2	29.60 ± 0.62	0.49 ± 0.02	0.001
Gmeliana arborea	45.30 ± 1.08	28.15 ± 0.2	25.7 ± 0.59	0.25 ± 0.01	0.00113
Sugarcane straw	40.99 ± 0.93	27.49 ± 0.1	20.75 ± 0.4	0.73 ± 0.02	0.001
Rice husk	36.56 ± 0.82	20.12 ± 0.2	19.44 ± 0.2	0.20 ± 0.02	0.00113
Shea butter wood	44.80 ± 0.11	30.94 ± 0.2	23.82 ± 0.3	0.44 ± 0.02	0.001
Palm kernel shell	28.92 ± 0.14	25.01 ± 0.1	45.41 ± 0.4	0.65 ± 0.02	0.001
Bamboo wood	40.96 ± 0.92	25.83 ± 0.2	21.96 ± 0.2	0.25 ± 0.02	0.00303

**Ce-Cellulose; He-Hemicellulose; Li-Lignin; Ee-Extractive, Co-Corrosiveness.

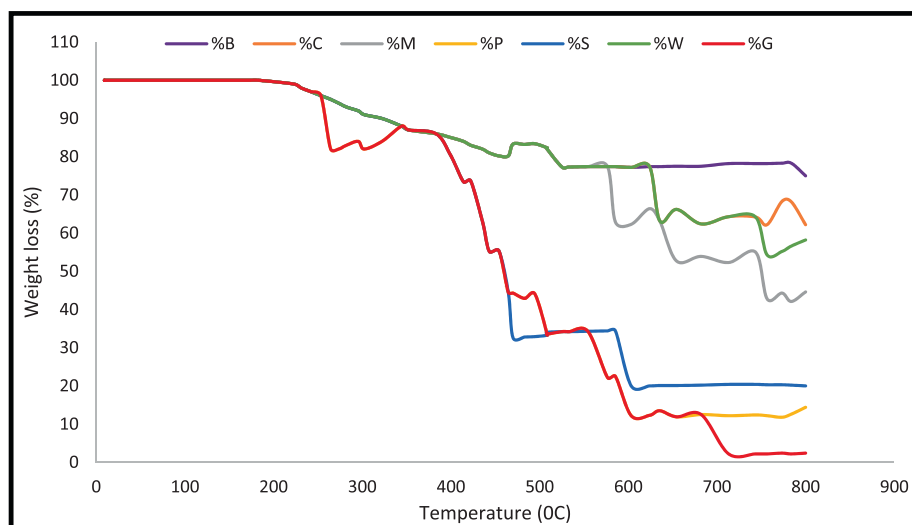


Fig. 1. TGA of Weight loss of 10 °C/min for Bamboo(B), Rice husk (C), Gmelina Arborea wood (G), Sugarcane straw(M), Palm kernel shell (P), Shea butter wood (S), and Siam weed (W) at the heating rate of 10 °C/min.

tent. Active pyrolysis of the biomass occurred during this process [59]. During the second phase, thermal degradation of hemicellulosic and cellulosic polymers simultaneously leads to the formation of an excessive number of small-molecule gas-phase and liquid-phase components with a relatively large molecular weight and a greater percentage of tar was precipitated [60] [61]. Optimum weight loss was recorded for G at about 60% (280–470 °C), while B possessed the lowest weight loss of 27% (280–480 °C). These losses in weight were attributed to the degradation of light volatile compounds below 100 °C [8]. The later phase had no significant weight loss due to the slow decomposition of the lignin contents during the final phase (carbonization) at a temperature of about 470–900 °C (Yu et al., 2020). The overall weight losses are B (25%), C (38%), G (98%), M (60%), P (95%), S (90%) and W (80%). G possessed the highest weight loss after undergoing exothermic thermal decomposition at different pyrolysis temperatures closely followed by P. Hence, ANG is more suitable for the pyrolysis pro-

cess among the samples reviewed next to P, S, and W respectively as the rate of weight loss is proportional to the energy released [25].

Table 4 shows the influence of heating rates on devolatilization parameters. The heating rate positively influenced the total weight loss temperature (T_{peak}) and the temperature at degradation termination ($T_{off-set}$) but did not affect the initial degradation temperature (T_{on-set}). The region of primary decomposition of the biomass samples was compared to the works of Adeleke et al. (2019) and Balogun et al. (2014).

4. Conclusion

This study investigates seven lignocellulose biomass samples based on proximate, ultimate, structural composition, and thermal properties to aid biomass selection for the thermochemical conver-

Table 4

Shows the influence of heating rates on devolatilization parameters basically for the region of primary decomposition of the biomass samples compared to the previous study.

S/N	Biomass	H (°C/min)	T _{ONSET} (°C)	T _{PEAK} (°C)	T _{OFFSET} (°C)	T _{BURN-OUT} (°C)	Weight loss %	Ref
1	B	10	200	400	500	550	25	PW
2	C	10	198	450	500	550	38	PW
3	G	10	196	500	600	650	98	PW
4	M	10	197	420	800	–	60	PW
5	P	10	180	400	600	650	95	PW
6	S	10	197	500	550	600	90	PW
7	W	10	200	400	600	650	80	PW
8	M	10 °C/min	197	326	502	607	58	##
9	Teak	5 k/min	295	330	347	–	–	

sion process. Results obtained showed that moisture, ash, and nitrogen contents negatively influenced pyrolysis yields because to lead to low HHV and LHV, while fixed carbon, carbon, cellulose, lignin, hemicellulose, and ether extract will have enhanced thermochemical conversion process as a result of their high HHV and LHV. The volatile matter (VM) must be kept within the normal range because an increase in VM leads to fast combustion and the formation of smoke. The low hemicellulose content in siam weed reduced the degradation pathway of biochar production and facilitated charring and incomplete combustion of the biomass via the pyrolysis process. High lignin contents of the biomass would decrease the pyrolysis rate, increasing the biochar yield. While the high cellulose and hemicellulose contents increase pyrolysis rate, hence, leading to an increase in the yield of bio-oil, NCG. The thermal analysis showed that thermal degradation of hemicellulosic and cellulosic polymers simultaneously leads to the formation of an excessive number of small-molecule gas-phase and liquid-phase components with a relatively large molecular weight and a greater percentage of tar was precipitated. Also, the heating rate positively influenced the total weight loss temperature (T_{peak}) and the temperature at degradation termination (T_{off-set}) but did not affect the initial degradation temperature (T_{on-set}). This study has been able to determine the energy potential of seven lignocellulose biomass for use as a fuel, which would aid researchers in selecting the best biomass for thermochemical conversion process. Hence, Shea butter wood is more recommended for the pyrolysis process, closely followed by Palm kernel shells next to wood due to their high energy potential. Much comprehensive studies are still required in order to make lignocellulose biomass viable for industrial scale in terms of efficient energy production and cost.

CRedit authorship contribution statement

A.O. Onokwai: Methodology, Software, Validation, Formal analysis, Writing – original draft. **E.S.A. Ajisegiri:** Resources, Writing – review & editing, Visualization. **I.P. Okokpujie:** Conceptualization, Supervision, Project administration, Funding acquisition. **R.A. Ibi-kunle:** Resources, Supervision. **M. Oki:** Resources, Investigation. **J.O. Dirisu:** .

Data Availability

The authors confirm that the data supporting the findings of this study are available within the article. Raw data that support this

study can be provided by the corresponding author upon reasonable request.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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