Mild pyrolytic treatment of *Gmelina arborea* for optimum energetic yields

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Mild pyrolytic treatment of *Gmelina arborea* for optimum energetic yields

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Abstract: One of the most promising routes to produce solid biofuel from biomass is mild pyrolytic treatment (torrefaction). In the present study, mild pyrolytic treatment of *Gmelina arborea* was carried out to obtain optimum energetic yields (mass yield, higher heating value and energy yield). The biomass of 0.5–6 mm particle sizes were torrefied at two different temperatures, 240 and 300°C for residence time of 30 and 60 min. Full-factorial experimental method was used for the optimization of torrefaction conditions in order to produce solid fuel with high energetic yields. The analyses revealed that torrefied biomass was better in terms of heating value, proximate contents and fuel ratio. The results also showed that temperature has the largest effect on the energetic yields compared to residence time and particle size. The optimum torrefaction conditions that produced the highest energetic yields were temperature of 260°C, residence time of 60 min and particle size of 2 mm as predicted using the factorial linear models. The optimum conditions were experimentally validated and the energetic yields obtained were acutely close to

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PUBLIC INTEREST STATEMENT

Biomass is a useful biofuel that can partially or completely replace fossil fuels. However, low energy density, high-moisture content and bio-degradation are some of the setbacks that hinder its adoption on larger scale. One of the major routes to improve on these qualities is mild pyrolytic treatment called torrefaction. Torrefaction is a thermal treatment process that takes place between 200 and 300°C in an inert environment. The impact of this process on biomass could be affected by different factors such as temperature, residence time and particle size among others. Therefore, mild pyrolytic treatment of melina wood was carried out under various conditions to obtain the best factors that yielded the highest energetic yields (mass yield, calorific value and energy yield).

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those predicted using factorial linear models developed in this study. Hence, mild pyrolytic treatment at a temperature of 260℃, residence time of 60 min and particle size of 2 mm is useful to produce solid biofuel with maximum energetic yields.

Subjects: Renewable Energy; Energy & Fuels; Manufacturing Engineering

Keywords: energetic yields; factorial experimental design; *Gmelina arborea*; optimization; weight loss; mild pyrolytic treatment

1. Introduction

Demand for renewable energy has increased in the last few decades due to the greenhouse effects resulting from continuous release of effluents from consumption of fossil fuels. One of the important sources of renewable energy is biomass due to its abundance and low cost (Almeida, Brito, & Perré, 2010). In addition, biomass maintains a closed carbon cycle with no net increase in atmospheric CO_2. This is due to replanting operation that leads to growth of new one taking up CO_2 released to the atmosphere during combustion of previous harvest (Bapat, Kulkarni, & Bhandarkar, 1997). Biomass can serve as clean development mechanism (CDM) for reducing Greenhouse gas emissions and other pollutants as reported by Li and Hu (2003). van der Stelt, Gerhauser, Kiel, and Ptasinski (2011) estimated that the use of biomass for power generation in 2060 will increase to about 200 exajoules (10^{18} J) as against its abysmal level of application in the 1990's.

Biomass can be classified as lignocellulosic and non-lignocellulosic. Lignocellulosic can also be grouped into woody and non-woody biomass. Among these categories is *Gmelina arborea* (Melina). It is one of the highest cultivated exotic species in Nigeria because it can grow on a wide range of soil and climatic conditions. It has a rapid growth rate and low extractives. The plantation of *G. arborea* was estimated to be 112000000 ha in the Southwest part of the country and for its demand in various applications, it yield high waste at the mill which can be employ for other purposes such as hybrid fuel with coal, generation of electricity among others (Onyekwelu, 2004). Raw biomass is known for its hydrophilic nature, low-energy content, high-moisture content and poor grindability among others. However, in order improve the properties of these energy rich materials, a mild pyrolytic treatment method (torrefaction) had been employed by several researchers (Balogun, Lasode, & McDonald, 2014; Chen & Kuo, 2010; Onyekwelu, 2004; Phanphanich & Mani, 2011; Prins, Ptasinski, & Janssen, 2006). Torrefaction is a thermal process/ technology where biomass is heated in an inert environment or nitrogen atmosphere at 200–300 ℃(Lasode, Balogun, & McDonald, 2014). This technology/process is in advanced level of application and development in Europe and other developed countries where biomass has continued to partly or totally replace some fossil fuels applications. However, developing nations such as Nigeria with epileptic and acute energy challenge are yet to take advantage of it (Mohammed, Mustafa, Bashir, & Mokhtar, 2013).

Few literatures available on torrefaction of biomass species in Nigeria are traceable to Balogun (2014) and therefore, further study becomes paramount and important in this area. The influence of torrefaction on biomass is affected by some parameters. These parameters are torrefaction temperature, resident time and particles sizes (Arias et al., 2008). Therefore, in order to avoid critical challenging questions like what optimal conditions is best for torrefaction or access how each condition affects biomass, the present study built linear regression models for torrefaction conditions (temperature, residence time and particle size) of Nigeria biomass (Melina) energetic yields after torrefaction using a full-factorial experimental method. In previous studies, optimization of torrefaction of mixed softwood by response surface method (RSM) for biomass upgrading to high energy density was carried out by Lee, Kim, Lee, Lee, and Lee (2012). Chin et al. (2013) also carried out optimization of torrefaction conditions for high energy density solid biofuel from oil palms biomass and fast growing species available in Malaysia using RSM. They considered effects of two parameters (temperature and resident time) on torrefaction in their model development.

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However, since torrefaction process may also be influence by particle size, the present study considered its effects as well as those of temperature and residence time in optimizing torrefaction conditions of lignocellulosic biomass using full-factorial experimental design method.

2. Materials and method

2.1. Material collection and preparation
The lignocellulosic woody biomass used in this study is Melina wood also known as *G. arborea*. It was obtained from Benin (Nigeria) and converted into chips and fines of ±6.35 mm. Lumber was obtained from Benin, Nigeria (6° 20′ 17.34″ N, 5° 37′ 32.70″ E) and converted into chips and fines below 6.35 mm using a saw wood cutting machine. The wood chips and fines were sun-dried for five days (6 h/day) to remove surface and residual water. The wood chips were further pulverized and screened to four different particle sizes: −0.5 mm (tagged as 0.5 mm), +0.5–2 mm (tagged as 2 mm), +2–4 mm (tagged as 4 mm) and +4–6.35 mm (tagged as 6 mm) sieve sizes.

2.2. Experimental design
A full-factorial regression experimental design using Minitab 17 Software-Trial version was adopted for the present study. The design was blocked with two replicates for each treatment. The dependent variables were mass yield (MY), higher heating value (HHV), and energy yield (EY), while torrefaction temperature (TT), resident time (RT) and particle size (PS) were the independent variables. The low and high value sets for the independent variables are given, respectively, as TT of 240 and 300°C, RT of 30 and 60 min and PS of 2 and 6 mm. Sixteen experimental runs were carried out based on the set conditions as shown in Table 1 and each run was treated as a separate experiment. Severity factor is a pseudo-first-order reaction that combines the torrefaction temperature (TT) and RT in one value and provides an approximate indication of the treatment conditions. Severity factor cumulates the effects of temperature and RT and it was calculated

Table 1. Experimental design interface with three independent and dependent variables in Minitab 17						
	Independent variables			**Dependent variables**		
Run order	**TT (°C)**	**RT (min)**	**PS (mm)**	**MY (%)**	**HHV (MJ/kg)**	**EY (%)**
1	240	30	2	77.17	21.18	88.88
2	240	30	6	79.21	20.75	89.38
3	300	60	6	44.75	25.86	62.93
4	300	30	6	47.96	26.12	68.12
5	300	60	2	42.52	27.04	62.52
6	300	30	2	40.34	28.26	61.99
7	240	60	2	81.18	21.08	93.05
8	240	60	6	66.33	22.37	80.69
9	300	60	6	57.39	22.23	69.37
10	300	60	2	38.07	29.11	60.26
11	240	60	6	77.98	21.15	89.68
12	300	30	6	41.85	27.83	63.33
13	240	30	2	76.85	21.70	90.68
14	240	30	6	78.87	21.32	91.44
15	300	30	2	40.16	27.07	59.12
16	240	60	2	78.54	20.86	89.09

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Table 2. Effect of temperature, residence time and particle size on proximate contents and fuel ratio of *Gmelina arborea* during torrefaction				
Sample ID	MC (%)	VM (%)	AC (%)	FC (%)
240–60-2	3.01	71.86	2.22	22.89
260–60-2	2.88	54.09	2.18	41.06
280–60-2	2.60	47.92	2.21	47.28
300–60-2	2.53	34.64	2.29	60.55
300–**30**-2	2.47	37.15	2.23	58.17
300–**60**-2	2.53	34.64	2.25	60.55
300–**90**-2	2.49	34.64	2.24	59.06
300–**120**-2	2.37	34.20	2.25	61.19
300–60-**0.5**	2.49	34.26	2.26	61.01
300–60-**2**	2.53	34.64	2.29	60.54
300–60-**4**	2.70	34.45	2.23	59.63
300–60-**6**	2.76	34.87	2.20	51.18
Raw	7.52	81.42	2.15	8.92

using Equation (1) (Lee et al., 2012). The dependent variables obtained for each run are also presented in Table 2:

$$SF = \log\left(RT.\left(\exp^{(TT-T_R)/14.75}\right)\right) \tag{1}$$

where SF is the severity factor, T_R is the reference temperature that has been adopted to be 100°C, TT is the torrefaction temperature in °C and RT is the RT in minutes. Two additional TTs (260 and 280°C) with a constant RT (60 min) for PS of 2 mm, RT (90 and 120 min) at constant temperature (300°C and other PS stated in Section 2.1 at constant RT (60 min) and temperature (300°C were used as torrefaction conditions for comparisons.

2.3. Torrefaction experiment

Tubular furnace (KW-Load-SL-No NEC/65 Model) with capacity to supply N_2 was used for torrefaction process. An Electronic Analytical and Precision Balance (Sartorius BSA Series: BSA 224S-CW) was used to weigh 46 g of Mw samples for each experiment. The sample was poured into a crucible which was then placed in the tubular furnace and N_2 gas flow was continuous at 2 L/min to maintain the inert environment within the tubular chamber. Torrefaction (mild pyrolytic treatment) is usually carried out with low heating rates to avoid uneven heat distribution at higher rate; hence, 12°C/min was employed for the experiment till the desired TT after which it was soaked for each RT. After each RT, the samples were allowed to cool for 4–5 min in the furnace before it was removed and placed in the desiccator for further cooling. This was done to avoid the oxidation that may result in the ignition of the torrefied product. The samples were again weighed in order to study its MY and placed in a zip-locked polythene for further characterizations and analyses. Weight loss and MY were obtained for different set of conditions as follows:

$$W_L = [(I_w - F_w)/I_w] \times 100\% \tag{2}$$

$$MY = 100 - W_L \tag{3}$$

where W_L is the weight loss, I_w is the initial weight of the sample, F_w is the final weight of the sample and MY represents MY.

2.4. Proximate analysis

The proximate analysis for raw Melina wood was carried out in duplicates and the average has been reported. The moisture content (MC) was determined according to ASTM E871-82 (2013) standard in an oven (Model No: OF-22G, JESO TECH, Korea). Volatile matter (VM) contents of raw Mw were determined in accordance with BS EN 15148 (2009) standard. The ash content (AC) of raw Mw was carried out in a muffle furnace (Model No: CBFL518C, USA) in accordance with ASTM E1755-01 (2015) standard. Similarly, the proximate analysis of torrefied sample was carried out according to IS: 1350-1 (1984) standards. Fixed carbon (FC) content was obtained by positive difference between 100 and the sum of moisture, ash and VM contents for both raw and torrefied Melina.

2.5. Heating value determination

The HHV analysis for both raw and torrefied samples were carried out in a Parr 6200 Oxygen Bomb Calorimeter (Model No: A1290DDEE) using ASTM D5865-04 (2004). 0.5 g of oven-dried sample was completely combusted under a pressurized (3.5 MPa) oxygen atmosphere. The analysis was carried out in duplicates and the average has been reported. HHV results were used to obtain the EY after torrefaction as follows (Yan, Acharjee, Coronella, & Squez, 2009):

$$EY = EDR \times MY \tag{4}$$

$$EDR = HHV_{Product}/HHV_{raw\ Mw} \tag{5}$$

where EDR is energy densification ratio and MY is the MY (%).

2.6. Statistical analyses and model development

Full-factorial linear regression models were developed between the dependent variables (MY, HHV and EY) and independent variables (TT, RT and PS) both in coded and uncoded formats. Optimum torrefaction conditions obtained were used for torrefaction of the sample in order to confirm and validate the models developed.

3. Results and discussion

3.1. Weight loss, and proximate of torrefied biomass

The weight loss and HHV_{rise} of torrefied lignocellulosic biomass are shown in Figure 1. HHV_{rise} was obtained by subtracting the HHV of raw biomass (Mw) from that of torrefied samples ($HHV_{rise} = HHV_{torrefied\ sample} - HHV_{raw\ Mw}$). The result presented in Figure 1 shows that weight loss increased with an increase in severity factor. The weight loss ranged from 20.89 to 60.02% depending on the set parameters for the thermal treatment and this is directly relate to the HHV_{rise} of torrefied biomass. The HHV_{rise} ranged from 2.5 to 9.6 MJ/kg for the torrefied *G. arborea*. The mild pyrolytic treatment (torrefaction) at every set parameter improved the quality of biomass based on the increment in the HHV. The weight loss as well as HHV_{rise} for the torrefied biomass were relatively closed in values after the severity factor of 7.37. At this point, temperature was kept constant at 300°C while the RT was varied in Equation 1. This implied that irrespective of RT, the influenced of temperature was more pronounced on the *G. arborea* during torrefaction. The trend of the increment in HHV is similar to previous studies for woody biomass (Lasode et al., 2014; Tchapda & Pisupati, 2014).

The moisture, ash, VM and FC contents of raw biomass presented in Table 2 are within the range obtained in previous studies for various woody biomass (Chen & Kuo, 2010; Jones et al., 2012). The proximate contents were influenced by the different torrefaction conditions. The MC of torrefied biomass are in the range of 2.4–3% and are lower compared to 7.52% of raw biomass. The AC of the torrefied sample is similar to that of raw sample and it further affirms that torrefaction process does not affect AC of biomass (Adeleke, Odusote, Paswan,

Figure 1. Weight loss and *HHV*$_{rise}$ based on severity factor (2 mm).
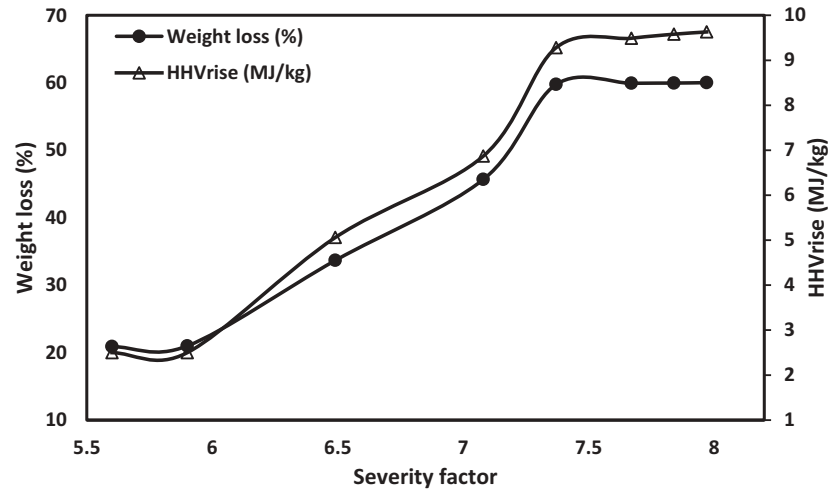
Lasode, & Malathi, 2019; Sarvaramini & Larachi, 2014). The FC increased drastically at all torrefaction conditions compared to 8.93% of raw biomass. However, the increase was majorly influenced by increment in TT against the minor impact of RT and particles size as shown in Table 2. This was because of higher carbonation and higher loss of volatile compounds as the temperature increased. The highest FC obtained was 61.09% and it corresponds to the lowest VM (34.20%) as shown in Table 2. This is an agreement with the assertion of Basu (2013) that at high TT, loss in VM is very high. The VM (71.86%) obtained for samples that underwent torrefaction at 240°C were close to 81.42% of raw biomass. Reduction of hemicellulose begins at temperature higher than 180–240°C and operating within this temperature zone, cellulose is thermally stable which may account for high VM in the samples torrefied at 240°C (Basu, 2013). The fuel ratio (VM/FC) which is also an important response that reveals improvement in fuel property of biomass are presented in Figure 2 based on various parametric settings. Figure 2 shows that the fuel ratio increased with temperature increment. The RT does not have a pronounced pattern of influence on the fuel ratio. However, to a certain degree, the fuel ratio reduced as the PS increased (Figure 2). This implied that the rate of degradation of hemicellulose, cellulose and lignin in the biomass as the PS became bigger was low. The rate of heat transfer within the biomass polymer contents could possibly be limited due to large PS (Basu, 2013; Lee et al.,

Figure 2. Effect of temperature, residence time and particle size on fuel ratio of *Gmelina arborea* during torrefaction.

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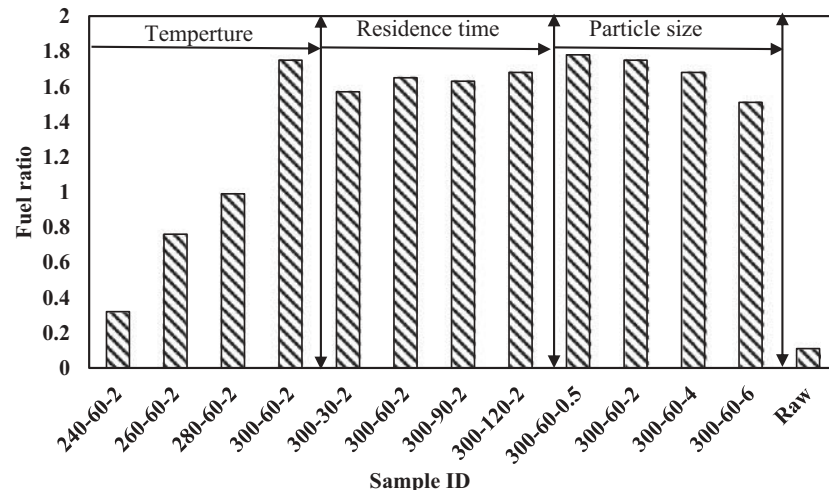
Table 3. Analysis of variance (ANOVA) of the models for EY, HHV and EY of *Gmelina arborea* during torrefaction					
Terms	**Degree of freedom**	**Sequential sum of square**	**Adj. sum of square**	**F-Value**	**P-Value**
Mass yield (MY)					
Model	7	4554.70	650.67	28.93	0.000*
Linear	3	4350.99	1450.33	64.49	0.000*
TT	1	4326.02	4326.02	192.35	0.000*
RT	1	1.18	1.18	0.05	0.824
PS	1	23.79	23.79	1.06	0.334
TT*RT	1	26.24	26.24	1.17	0.312
TT*PS	1	111.36	111.36	4.95	0.057
RT*PS	1	3.27	3.27	0.15	0.713
TT*RT*PS	1	62.85	62.85	2.79	0.133
Error	8	179.92	22.49		
Total	15	4734.63			
Higher heating value (HHV)					
Model	7	136.871	19.553	12.95	0.001*
Linear	3	122.247	40.749	26.99	0.000*
TT	1	116.316	116.316	77.05	0.000*
RT	1	1.266	1.266	0.84	0.387
PS	1	4.666	4.666	3.09	0.117
TT*RT	1	1.904	1.904	1.26	0.294
TT*PS	1	6.477	6.477	4.29	0.072
RT*PS	1	1.134	1.134	0.75	0.411
TT*RT*PS	1	5.108	5.108	3.38	0.103
Error	8	12.076	1.510		
Total	15	148.947			
Energy yield (EY)					
Model	7	2640.50	377.21	29.54	0.000*
Linear	3	2559.43	853.14	66.81	0.000*
TT	1	2556.57	2556.57	200.21	0.000*
RT	1	0.35	0.35	0.03	0.873
PS	1	2.52	2.52	0.20	0.669
TT*RT	1	11.21	11.21	0.88	0.376
TT*PS	1	46.82	46.82	3.67	0.092
RT*PS	1	17.75	17.75	1.39	0.272
TT*RT*PS	1	5.30	5.30	0.42	0.537
Error	8	102.15	12.77		
Total	15	2742.66			

*Significant factor.

2012). Also, reduction in PS is always accompanied by an increase in reactive surface which could facilitate heat and mass transfer. This can possibly affect the responses no matter how small, as shown in Table 3.

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3.2. Main and interactions effects between torrefaction parameters on mass yield, HHV and energy yield

The MY, HHV and EY of the torrefied biomass samples were compared with the raw in Figure 3. The MY which is the quantity of solid residue that remains after torrefaction decreased with an increase in TT, as shown Figure 3. Similarly, the EY also deceased with increasing the temperature. Notably, in the course of torrefaction, energy-lean volatiles, moisture and gases are released from biomass, taking up certain quantity of resident energy in biomass (Shinya, 2008); however, the rate increased with increasing the temperature. This significantly implied that temperature has a large effect on both the mass and EYs negatively. On the other hand, the HHV of torrefied biomass increased with increase in temperature. This is a positive influence. Thus, the influence of temperature was more pronounced than RT and PS on MY, HHV and EY during torrefaction. Low TT setting (240°C) gave high MY but progressing towards 300°C, the MY drastically decreased and this implied that for maximum MY, torrefaction must be carried out at low-temperature settings. However, biomass torrefaction at low temperature will give low HHV due to minimum degradation of hemicellulose and cellulose contents of biomass. Similar observation was made for EY as RT and PS had abysmal effects on these responses. Figure 3 shows that navigating from low to high setting of RT, the yield relatively remains constant. However, as the PS increased from 0.5 to 6 mm, the HHV of the torrefied biomass reduced. This is an indication that it has significant effect on HHV and should be a major factor to be considered during the torrefaction biomass. Advancing from low- to high-temperature setting, HHV yield drastically increased which is as a result of severe devolatization, thereby allowing more C–C bonds to be formed rather than O–H bonds that dominate hemicellulose in biomass (Yan et al., 2009). The observation of the present study is in agreement with the study by Chin et al. (2013) on optimization of torrefaction conditions for high energy density solid biofuel from biomass and fast-growing species in Malaysia. Similar to the present study, it was concluded that temperature had the largest influence than reaction time during torrefaction of the fast growing species.

3.3. Factorial regression models and response optimizer

The analysis of variance (ANOVA) of the models for the MY, HHV and EY of *G. arborea* during torrefaction is presented in Table 3. For α-level of 0.05, Table 3 shows that only the effect of TT was significant for torrefaction of lignocellulosic compared with RT and PS because the *p*-value was 0 and is lower than the threshold significance level (0.05). The two-way interaction effect between temperature and residence (TT*RT) was not significant for MY, HHV and EY due to large *p*-value greater than 0.05 which served as the threshold value. However, the *p*-value

Figure 3. Effect of temperature, residence time and particle size on energetic yields of *Gmelina arborea* during torrefaction.
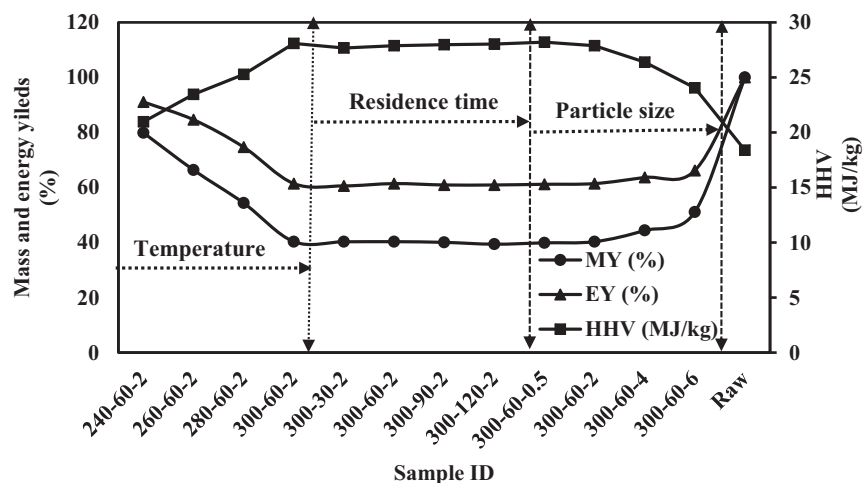
for the two-way interaction between TT and PS (TT*PS) was a little higher than the threshold (0.05), as shown in Table 3. This corresponds to the results of the experimental data (Figure 3), where it was observed that PS has a significant contribution during the torrefaction of biomass. The three-way interactions (such as TT*RT*PS) was not significant at all. To fit the response function and experimental data, factorial linear regression was performed and the relatively high correlation factor (R^2) values for MY (Equation (6)), HHV (Equation (7)) and EY (Equation (8)) show that the factorial regression models are well correlated with the actual data. The values of R^2 obtained from regression analysis for these linear models at 95% confidence level were 0.962, 0.9189 and 0.9628 for MY, HHV and EY, respectively. The R^2 relates well with quadratic models developed using surface response method for torrefaction of biomass by (Lee et al. 2012; Chin et al. 2013)

$$MY(\%) = 177.6 - 0.456TT + 16.179PS - 0.0551TT^*PS \tag{6}$$

$$HHV(MJ/kg) = 9.8 + 0.054TT - 4.63PS + 0.018TT^*PS \tag{7}$$

$$EY(\%) = 205.9 - 0.5TT + 1.9PS - 0.0007TT^*PS \tag{8}$$

The response optimizer in Minitab 17 was used to obtain the optimum torrefaction conditions that will yield the maximum MY, HHV and EY. The optimum conditions obtained were TT of 260°C, RT of 60 min and PS of 2 mm. Similarly, from the response optimizer, the predicted responses were MY of 66.67%, HHV of 23.33 MJ/kg and EY of 81.17% at high desirability close to the weight value set (unity/1). The optimum conditions (260°C, 60 min and 2 mm) were validated in a torrefaction experiment. The results were 66.33%, 23.45 MJ/kg and 84.58% for MY, HHV and EY, respectively. These outcomes were in close agreement with predicted values by Minitab 17 response optimizer. The result suggests that the models can correctly predict MY, HHV and EY of lignocellulosic biomass in Nigeria and tropical regions without the need for higher order term inform of quadratic or polynomial functions.

Generally, the present study significantly showed that mild pyrolysis process enhances the energy properties of *G. aborea* of Nigeria origin. The study also formulated linear models for the prediction of torrefaction output in terms of MY, HHVs and EY based on parametric settings for woody biomass of tropical region.

4. Conclusion
The mild pyrolytic treatment of *G. arborea* for optimum energetic yields was carried out with it parametric settings investigated using full-factorial experimental design. The study therefore concluded the following:

(i) the mild pyrolytic treatment (torrefaction) improved the HHV of *G. arborea* from 18.39 to a range of 20–28%;

(ii) the fuel ratios of *G. arborea* increased with an increase in temperature;

(iii) temperature has the largest influence on energetic yields (MY, HHV and EY). However, the two-way interaction between it and PS has also a significant impact;

(iv) the linear models developed can be used for prediction of the energetic yields of biomass from Nigeria due to the high correlation factor (R^2); and

(v) the optimum torrefaction conditions that gave the highest energetic yields are TT of 260°C, RT of 60 min and PS of 2 mm.

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Nomenclature table

S/N	Abbreviation/symbols	Meaning
1	AC	Ash content
2	ANOVA	Analysis of variance
3	*EDR*	Energy densification ratio
4	EY	Energy yield
5	FC	Fixed carbon
6	F_w	Final weight of sample after torrefaction
7	HHV	Higher heating value
8	I_w	Initial weight of samples
9	MC	Moisture content
10	MY	Mass yield
11	PS	Particle size
12	RT	Residence time
13	R^2	Correlation factor
14	TT	Torrefaction temperature
15	SF	Severity factor
16	T_R	Reference temperature
17	VM	Volatile matter
18	W_L	Weight loss
19	α	Threshold significant level

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