



EFFECTS OF TORREFACTION PARAMETERS ON THE PROPERTIES OF BRIMSTONE (*Morinda lucida*) WOOD

A. R. Nasirudeen^{1*}, F. A. Ola³, O. O. Oniya³, A. J. Adesope¹, D. Lasisi¹,
L. A. Balogun¹, W. A. Adesope², I. A. Abdulsalam³ and A. O. Oladiji²

¹*Department of Agricultural and Bio-Environmental Engineering, Oyo State College of Agriculture and Technology, Igboora, Nigeria

²Department of Mechanical Engineering, Oyo State College of Agriculture and Technology, Igboora, Nigeria

³Department of Agricultural Engineering, Ladake Akintola University of Technology, Ogbomosho, Oyo State, Nigeria

*Corresponding author's email address: jibolarufai@gmail.com

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ABSTRACT

Biomass energy is a possible replacement for the conventional energy due to its environmental effects. However, raw biomass has poor combustible properties which can be enhanced by torrefaction. This research studied the torrefaction properties of brimstone wood as potential energy source. Existing torrefaction device was modified by incorporating a thermocouple and temperature regulator and rectangular reactor was used for the torrefaction of brimstone wood. Harvested samples of the materials were manually processed into smaller pieces and sun dried. The sun-dried samples were crushed and sieved into 8, 10 and 12 mm particle sizes. The sieved samples were subjected to ultimate (Carbon (C), Oxygen (O), Hydrogen (H), Nitrogen (N), Sulphur (S) and energy contents) compositions analysed using standard methods. A 3 by 3 Box-Behnken experimental design in the Design Expert 12.0.2 software was used to design the experiment. The influence of heating temperature (220, 260 and 300 °C), heating time (30, 60 and 90 min) and particle size (8, 10 and 12 mm) on the weight of biochar products were studied. Model equations were developed and used to evaluate Biochar Mass Yield (BMY), Energy Yield (BEY) and Energy density (BED) and evaluated based on the heating temperature, heating time and particle size ranges. The data obtained from the experimental design were analysed statistically with Analysis of Variance (ANOVA) at 95 % confidence level. The carbon, oxygen, hydrogen, nitrogen, sulphur and energy contents for brimstone wood were 52.20, 36.88, 2.40, 0.54, 0.89% and 245.89 kcal/100g. R² values of BMY, BEY and BED were 0.9499, 0.9348 and 0.9990 at P≤0.05 for brimstone wood. Torrefied brimstone wood has been identified as potential sources of energy, with heating temperature and heating time as critical parameters that affect properties of torrefied materials.

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1.0 Introduction

The World's energy demand is increasing geometrically as evidenced in the increasing demand for fuels for transportation, industry as well as domestic activities (Melgar et al., 2009). The growth and economic development of the world depends on its energy and other factors such as population growth, infrastructural and technological development and this is putting pressure on conventional energy resources. These factors lead to increase in energy demand despite the environmental pollution and global warming effects resulting from the use of petroleum-based fuels (Adeleye et al., 2018). In the last 39 years, about 75% of human made CO₂ emissions were from the consumption of fossil fuel resources (Ferro et al., 2004). There is increasing emphasis on renewable energies following the global demand for clean energies which are sustainable. One of the renewable energy sources is biomass which is an alternative source of energy derived from biological materials (plant and animals). Existing technologies to convert biomass to energy include thermochemical and biochemical processes. The thermochemical conversion technique which involves combustion, pyrolysis,

gasification and liquefaction processes is more familiar. The thermochemical process of torrefaction which is used for the treatment of biomass at low heating temperatures ranging from 200 to 300 °C under an inert atmosphere is known to be effective for improving the energy density and shelf life of biomass materials (Ferro *et al.*, 2004). It has been widely applied to wood and grass biomass over the past 30 years to maximize the solid yield of biomass materials, thus producing large fraction of biochar ranging from 70 to 85% of the input raw material for use in energy, metallurgical fuel, chemical and fertilization applications. Brimstone tree (*Morinda lucida*) also known as Oruwo in Yoruba language is one of the 80 species in the genus *morinda* of the pyroidea family and it is a tropical rainforest tree. The wood is yellow hence the name brimstone tree. *Morinda lucida* is a medium sized tree at maturity up to 18 - 25 years evergreen shrub with bole and branches often crooked or gnarled; bark smooth scaly, grey to brown, stipules ovate or triangular, 1-7 mm long, falling early. Although the plant is very bitter, yet the whole plants; leaves, stem, bark and roots are known to have medicinal properties. The stem has high energy content to be used in production of charcoal for the local gunpowder. These entirely play important role in satisfying human needs for energy and life processes (Adeleye *et al.*, 2018).

2. Materials and Methods

2.1 Materials and Equipment

The materials and the equipment used in this study are stated in the following sections:

2.1.1 Materials needed for the Experiment

The materials used for the experiment in the course of this study is Brimstone wood

2.1.2 Equipment and Tools used for the Experiment

The following equipment were used for the experiment in the course of this study:

Electrically heated torrefaction reactor, Laboratory oven drier, electronic weighing scale, Hammer mill, Sieve saker, Bulb calorimeter, Stopwatch, Desiccator, Moisture can, Polythene, Bowl, Sample container, Knife, Cutlass, Thermocouple.

2.2 Methods

2.2.1 Collection and preparation of Brimstone (*Morinda lucida*) Wood

Brimstone (*Morinda lucida*) wood was collected from brimstone tree from a private farm in Iseyin, Iseyin Local Government Area, Oyo State, Nigeria. The leafy parts of the harvested matured wood were cut away from the woody parts. The fresh wood was cut into small sizes manually using a cutlass and then sun dried for some days to reduce the moisture content of the wood to safe storage level of about 12% moisture content wet basis for further studies. The sun-dried samples were crushed into smaller sizes using hammer mill and sieves into different particle sizes using a set of sieves and the sieve shaker. From the sieving operations, three particle sizes; 12 mm which were particles that passed through sieve size 12 mm and retained on 10 mm sieve, 10 mm which were particles that passed through 10mm and retained on 8mm sieve and also 8 mm particles that passed through sieve size 8 mm and retained on 6 mm sieve were collected separately for the experiments. The particles sizes are shown in plate I (a-d).

2.2.2 Determination of the Ultimate Composition of the Solid Biochar Product

Samples of the solid biochar products obtained after torrefaction process were characterized using the methods and procedures of the ASTM D3173-86 standard (2007). The ultimate composition of the biochar determined includes nitrogen content, carbon content, hydrogen content, oxygen content and sulfur contents. The higher and lower heating values of the biochar were also determined (Jaya, 2016).



(a)



(b)



(c)



(d)

Plate 1: Brimstone wood; (a) Raw wood, (b) 12 mm wood particle size, (c) 10 mm wood particle size, (d) 8 mm wood particle size

2.2.3 Determination of Calorific Value

The calorific value of the samples was determined using bomb calorimeter in accordance with ASTM D5865 (2010) standard method. The high and low calorific values which were determined by using the dulong formula as given by Equations 1 and 2

$$HCV = 4.18 * (78 * C + 241.3 * \frac{H*O}{8} + 22.1 * S) \quad (1)$$

$$LCV = 4.18 * (94.19 * C - 0.550 - 52.14 * H) \quad (2)$$

where: *HCV* = Higher calorific value, kcal/100g, *LCV* = Lower calorific value, kcal/100g,

C = Percentage of carbon content, (%), *H* = Percentage of hydrogen content, (%) and

O = Percentage of oxygen content, (%).

2.2.4 Torrefaction Process

Torrefaction of the sample materials were carried out using the modified torrefaction device separately. The prepared samples were processed according to the experimental design layout suggested by the Design Expert software. Equal sample weight of 30 g was used for the two samples in all the experiments. For each experimental run, the desired 30g of the sample was first loaded into the reactor box and then covered up with the lid plate. The heating element connected to electrical source is then switched on and allowed to heat up the heating chamber to the desired preset heating temperature which is read on the temperature monitor. When the preset heating temperature is reached, the loaded reactor is quickly introduced into the heating chamber and allowed to remain in the heating chamber to be heated up to the desired heating temperature for the desired heating time or retention time monitored using a stopwatch. At the end of the retention time, the heating element is switched off and the heated reactor withdrawn from the heating chamber and placed in the desiccator to cool down in the absence of air. After proper cooling, the difference in weight of sample

before and after heat treatment is taken as the weight of biochar material remaining and collected from inside the heating reactor. The collected biochar product obtained is weighed, packed, labeled and characterized for the proximate and ultimate compositions. The experimental procedure was repeated for other runs and sample materials. The data collected from the measurement of the weight of the torrefied solid biochar product was used to evaluate biochar mass yield, energy yield and energy density using Equations 3, 4 and 5, respectively (Bergman et al, 2005).

$$\text{Biochar Mass Yield (B}_{MY}\text{): } B_{MY} = \frac{M_p}{M_r} \times 100 \quad (3)$$

$$\text{Biochar Energy Yield (B}_{EY}\text{): } B_{EY} = B_{MY} \times \frac{HHV_p}{HHV_r} * 100\% \quad (4)$$

$$\text{Biochar Energy Density (B}_{ED}\text{): } B_{ED} = \frac{B_{EY}}{B_{MY}} \quad (5)$$

where: B_{MY} = Biochar Mass Yield, %, B_{EY} = Biochar Energy Yield, %, B_{ED} = Biochar Energy Density, M_p = Mass of torrefied product, (g), M_r = Mass of raw biomass, (g), HHV_r = High Heating Value of raw biomass, (MJ/kg), HHV_p = High Heating Value of torrefied product (MJ/kg).

2.2.5 Experimental Design and Statistical Analysis

The influence of the experimental conditions of heating temperature from 220 to 300 °C (220, 260 and 300 °C) at 40 °C interval and heating time from 30 to 90 min (30, 60 and 90 min) at 30 min intervals and sample sizes from 8 to 12 mm (8, 10 and 12 mm) at 2 mm intervals for both brimstone wood and ackee fruit pod on the solid biochar product yields and on the properties of the solid biochar products were studied. The experimental range and levels of the process variable codes are presented in Table 1. The data obtained were analysed using 3 factors; heating temperature, heating time and sample size at 3 levels of variation each using 3-factor, 3-level Box-Behnken experimental design of 17 runs at significant level of $p \leq 0.05$ of the Design Expert 12.0. The experiments were carried out according to the design matrix proposed by the software as presented in Table 2.

Table 1: Experimental Range and Levels of Process Variable Codes

Parameter	Unit	-I	0	I
Heating temperature	(°C)	220	260	300
Heating time	(min)	30	60	90
Sample size	(mm)	8	10	12

Table 2: Experimental Design Layout for the Sample Materials

Std	Run	A (°C)	B (Min)	C (mm)
1	1	220	30	10
9	2	260	30	8
11	3	260	30	12
12	4	260	90	12
17	5	260	60	10
15	6	260	60	10
7	7	220	60	12
2	8	300	30	10
8	9	300	60	12
13	10	260	60	10
14	11	260	60	10
16	12	260	60	10
5	13	220	60	8
10	14	260	90	8
3	15	220	90	10
6	16	300	60	8
4	17	300	90	10

A: Heating Temp; B: Heating Time; C: Sample Size.

3. Results and Discussion

3.1 Modified Torrefaction Reactor and Torrefied Biomass Materials

The torrefaction device system consisting of the heating chamber, reactor, the thermocouple for the regulation of the required heating temperatures and the cooling system used for the experiment. Samples of the torrefied biochar materials subjected to various conditions of heating temperature and heating time and obtained after the torrefaction process are shown in plate 2 for the brimstone wood. The biochars obtained from the sample materials as shown in plate 2 were observed to vary in colours from visual image observation with variation in heating temperature and heating time. It was observed that the higher the heating temperature and heating time the darker the biochar from visual image observation. This indicates that the colour of the torrefied products (biochar) changes with increasing torrefaction conditions. Similar morphological changes in colour of paddy straw from yellowish to brown and then dark with torrefaction duration was reported by Swapnaja *et al.* (2020).



Plate 2: Torrefied brimstone wood Samples

Note: T_{i,ii,iii} = 220, 260, 300 °C; t_{i, ii, iii} = 30, 60, 90 min; S_{i, ii, iii} = 8, 10, 12 mm

T = Heating Temperature, °C; t = Heating time, min; S = Sample Size, mm.

3.2 Effects of Torrefaction Processing Parameters on Biochar Mass Yield, Energy Yield and Energy Density

The result of the effects of heating temperature, heating time and sample size on the biochar mass yield, energy yield and energy density of the torrefied samples; brimstone wood are presented in this section.

3.2.1 Effects of processing Parameters on Brimstone Wood Biochar Mass Yield

The design matrix and results of the biochar yields and density obtained for brimstone wood are presented in Table 3. The software presented a model and regression analysis to correlate the biochar mass yield with the independent variables of heating temperature, heating time and sample size considered for the study using a first order polynomial equation for the brimstone wood biochar mass yield as presented in Equation 6.

$$B_{dmy}(\%) = +300.84187 - 0.947062 A - 0.354917 B - 11.26875 C - 0.001333 AB + 0.021875 AC + 0.036250 BC + 0.000861 A^2 + 0.001447 B^2 + 0.150625 C^2 \quad (6)$$

where: B_{bmy} = Biochar mass yield of brimstone wood, %, A = Heating temperature, °C, B = Heating time, min, C = Sample size, mm.

The ANOVA results and statistical parameters of the analysis which describes the relationship between the independent variables and the dependent variable, biochar mass yield is as presented in Table 4. The probability of the model p-value and F-value of 0.0009 and 14.75 of brimstone wood biochar mass yield, indicates that the model suggested by the software for the material was significant at 95% confidence level. The R^2 value evaluated for the biochar yields of brimstone wood is 0.9499 meaning 94.99% of the variability can be accounted for and showing the regression model was satisfactory for the prediction of the biochar mass yield from brimstone wood. The ANOVA results of the model terms presented in Table 4 shows that A, B and A^2 are significant model terms, while it is shown in Equation 6 that AC, BC, A^2 , B^2 and C^2 model terms have synergistic effect on biochar yield and A, B, C and AB have antagonistic effect.

Table 3: Design Matrix and Results of Brimstone Wood Biochar Mass Yield, Energy Yield and Energy Density

Std	Run	A (°C)	B (min)	C (mm)	Brimstone Wood		
					Mass (%)	Yield Energy (%)	Yield Energy Density
10	1	260	90	8	38.7	44.07	1.14
14	2	260	60	10	56.1	63.23	1.13
9	3	260	30	8	73.3	76.79	1.05
15	4	260	60	10	54.7	61.86	1.13
17	5	260	60	10	53.9	61.05	1.13
16	6	260	60	10	55	62.09	1.13
13	7	260	60	10	53.6	56.90	1.06
1	8	220	30	10	89.7	93.38	1.04
7	9	220	60	12	82.7	86.63	1.05
6	10	300	60	8	50.7	56.80	1.12
3	11	220	90	10	85.3	91.58	1.07
4	12	300	90	10	39.7	45.77	1.15
12	13	260	90	12	34	38.99	1.15
5	14	220	60	8	86.3	92.65	1.07
11	15	260	30	12	71.7	79.31	1.11
2	16	300	30	10	63.3	70.04	1.11
8	17	300	60	12	40.3	45.72	1.13

A: Heating Temp; B: Heating Time; C: Sample Size

Table 4: ANOVA Result of Mass Yield for Brimstone Wood

Source	Sum of Squares	df	Mean Square	F-value	p-value	Conclusion
Model	4950.78	9	550.09	14.75	0.0009	Significant
A	2812.50	1	2812.50	75.40	< 0.0001	Significant
B	1257.51	1	1257.51	33.71	0.0007	Significant
C	51.51	1	51.51	1.38	0.2784	Not significant
AB	92.16	1	92.16	2.47	0.1600	Not significant
AC	11.56	1	11.56	0.3099	0.5951	Not significant
BC	2.40	1	2.40	0.0644	0.8070	Not significant
A ²	679.92	1	679.92	18.23	0.0037	Significant
B ²	19.15	1	19.15	0.5133	0.4969	Not significant
C ²	23.60	1	23.60	0.6327	0.4525	Not significant
Residual	261.11	7	37.30			
Lack of Fit	257.22	3	85.74	88.12	0.0004	Significant
Pure Error	3.89	4	0.9730			
Cor Total	5211.90	16				
Statistical Parameters			R ² = 0.9499	Adj R ² = 0.8855		
			Pred R ² = 0.2092	Adeq Precision = 13.3585		

The three-dimensional surface graph (3D) and two-dimensional contour (2D) in Figure 1 (a, b and c) showed the effects of heating temperature, heating time and sample size on the biochar mass yield of brimstone wood. The combined effect of heating temperature and heating time on the biochar mass yield of brimstone wood is shown in Figure 3a in three-dimensional surface and contour graphs. Figure 1a shows that biochar mass yield decreased with increase in heating temperature and heating time. The maximum and minimum biochar mass yields were obtained to be 85.3 and 39.7% at 220 °C, 90 mins and 300 °C, 90 mins respectively. Figure 1b for the combined effect of heating temperature and sample size shows that biochar mass yield decreases with an increase in heating temperature. The maximum and minimum biochar mass yields of 82.7 and 40.3% were obtained at 220 °C, 12 mm and 300 °C, 12 mm respectively. While it is shown in Figure 1c for the combined effects of heating time and sample size that increased in heating time results in decreases in biochar mass yield and vice versa. The maximum and minimum biochar mass yields were obtained to be 71.7 and 34.0% at 30 min, 12mm and 90 min, 12 mm, respectively. Similar decreases in biochar mass yield of patula pine wood and wood pellet from 67.75 – 98.46% and 39.2 – 80.3%, respectively at heating temperature range of 200 – 300 °C was reported by Sergio Ramos *et al.* (2017) and Ozge *et al.* (2022), respectively.

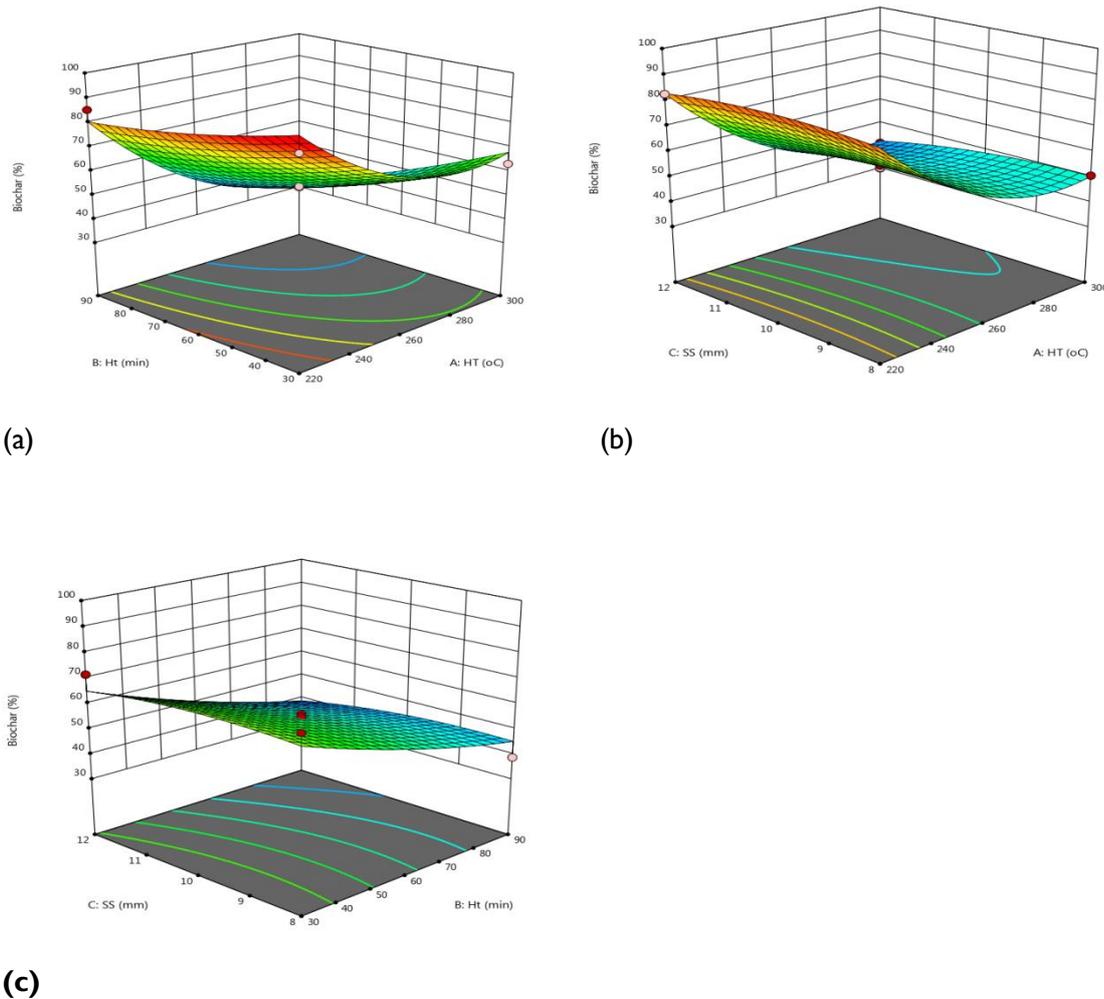


Figure 1: Response surface of biochar mass yield of brimstone wood as influenced by (a) heating temperature and heating time, (b) heating temperature and sample size, (c) heating time and sample size

3.2.2 Effects of Processing Parameters on Brimstone Wood Biochar Energy Yield

The design matrix and results of the biochar energy yield obtained for brimstone wood (B_{bey}) is presented in Table 5. The software presented a model and regression analysis to correlate the biochar energy yield with the independent variables of heating temperature, heating time and sample size considered for the study using polynomial equation for the brimstone wood biochar energy yield as presented in Equation 7.

$$B_{bey}(\%) = +540.17519 - 4.05095 A + 0.88720 B + 19.73375 C - 0.004681 AB - 0.015813 AC - 0.031667 BC + 0.007758 A^2 + 0.001947 B^2 - 0.747562 C^2 \quad (7)$$

where: B_{bey} = Biochar energy yield of brimstone wood, %, A = Heating temperature, °C, B = Heating time, min, C = Sample size, mm

The ANOVA results and statistical parameters of the analysis which describes the relations between the independent variables and the dependent variable, biochar energy yield is as presented in Table 5. The probability of the model p-value and F-value of 0.0022 and 11.15 for brimstone wood biochar energy yield, indicates that the model suggested by the software for the material was significant at 95 % confidence level. The R^2 value evaluated for biochar energy yields of brimstone wood is 0.9348 meaning 93.48 % of the variability can be accounted for, showing the regression models was satisfactory for the prediction of biochar energy yield of brimstone wood. The ANOVA results for the model terms presented in Table 5 shows that A, B and A^2 are significant model terms. While it is shown in Equation 7 that B, C, A^2 and B^2 have synergistic effect on biochar energy yield while A, AB, AC, BC and C^2 have antagonistic effect.

Table 5: ANOVA Result of Energy Yield for Brimstone Wood

Source	Sum of Squares	Df	Mean Square	F-value	p-value	Conclusion
Model	4778.13	9	530.90	11.15	0.0022	Significant
A-HT	2661.22	1	2661.22	55.91	0.0001	Significant
B-Ht	1227.85	1	1227.85	25.80	0.0014	Significant
C-SS	48.31	1	48.31	1.02	0.3472	Not significant
AB	126.23	1	126.23	2.65	0.1475	Not significant
AC	6.40	1	6.40	0.1345	0.7247	Not significant
BC	14.44	1	14.44	0.3034	0.5989	Not significant
A ²	648.69	1	648.69	13.63	0.0077	Significant
B ²	12.93	1	12.93	0.2716	0.6183	Not significant
C ²	37.65	1	37.65	0.7910	0.4033	Not significant
Residual	333.19	7	47.60			
Lack of Fit	309.44	3	103.15	17.37	0.0093	Significant
Pure Error	23.75	4	5.94			
Cor Total	5111.32	16				
Statistical Parameters		R ² =	0.9348;	Adj R ² =	0.8510	
		Pred R ² =	0.0241;	Adeq Precision =	11.5762	

The three-dimensional surface graph (3D) and two-dimensional contour (2D) in Figure 2 (a, b and c) shows the effect of heating temperature, heating time and sample size on the biochar energy yield of brimstone wood. The combined effect of heating temperature and heating time on the biochar energy yield on brimstone wood is as shown in Figure 2a in the three-dimensional surface and contour graphs. Figure 2a shows that energy yield increase as the heating temperature and heating time increase. The maximum and minimum biochar energy yields were obtained to be 93.38 and 45.72 % at 220 °C, 30 min and 300 °C, 90 min, respectively. Figure 2b reveals that energy yield decreased with increase in heating temperature only. The maximum and minimum biochar energy yields of 92.65 and 50.7% were obtained at 220 °C, 8 mm and 300 °C, 12 mm, respectively. While Figure 2c shows that increase in heating time gives decrease in biochar energy yield and vice versa. The maximum and minimum biochar mass yields were obtained to be 76.79 and 38.99% at 30 min, 8 mm and 90 min, 12 mm, respectively. Decrease in biochar energy yield of patula pine wood and wood pellet from 78.51 to 99.46% and from 59.9 to 85.4% at heating temperature range of 200 to 300 °C was reported by Sergio Ramos *et al.* (2017) and Ozge *et al.* (2022), respectively.

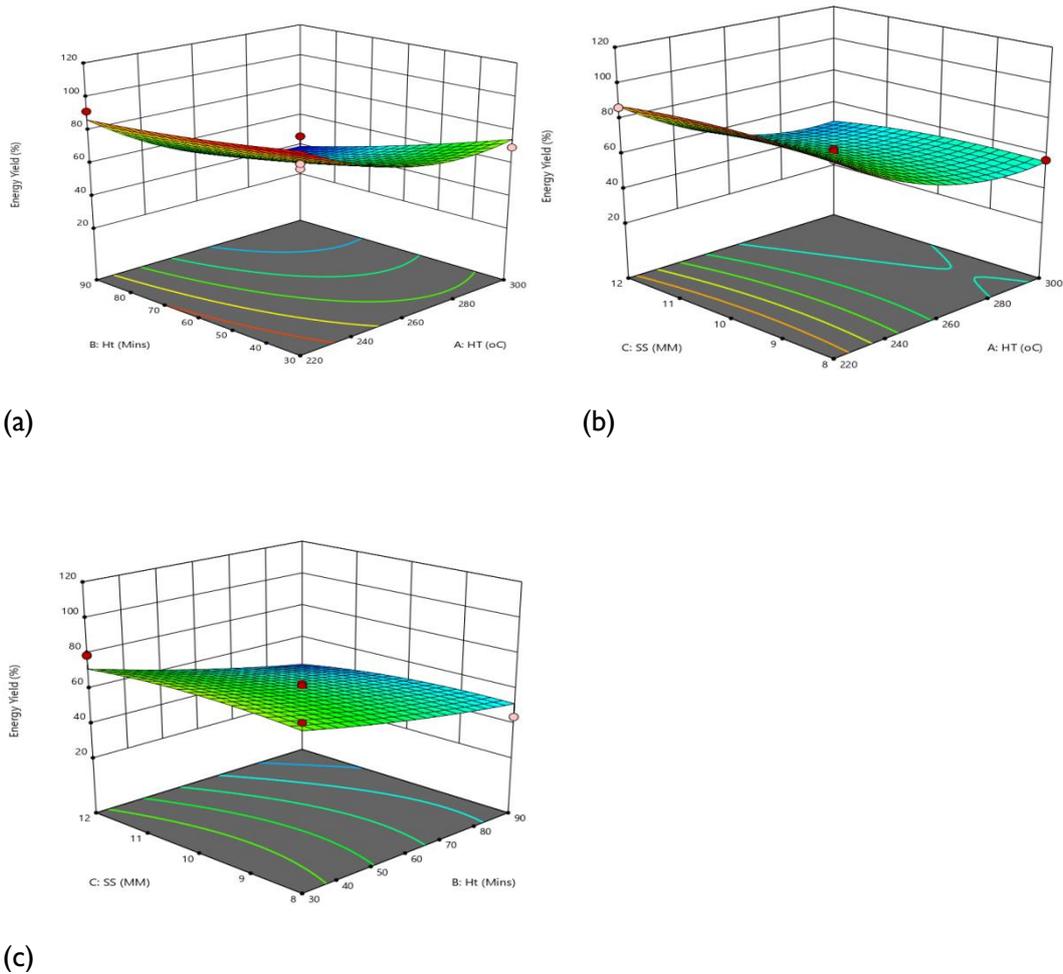


Figure 2: Response surface of biochar energy yield of brimstone wood as influenced by (a) heating temperature and heating time (b) heating temperature and sample size (c) heating time and sample size

3.2.3 Effects of Processing Parameters on Brimstone Wood Biochar Energy Density

The result of the effects of heating temperature, heating time and sample size on the biochar energy density of the torrefied samples, brimstone wood were presented in this section. The design matrix and results of the biochar energy density obtained for brimstone wood are presented in Table 6. The design matrix and results of the biochar energy density obtained for brimstone wood (B_{bed}) is presented in Table 6. The software presented a model and regression analysis to correlate the biochar energy yield with the independent variables of heating temperature, heating time and sample size considered for the study using polynomial equation for the brimstone wood biochar energy density as presented in Equations 8.

$$B_{bed}(\%) = -0.244531 + 0.011109 A - 0.000354 B - 0.039063 C + 2.08333E - 06 AB + 0.000125 AC + 0.000063 BC - 0.000022 A^2 - 2.08333E-06 B^2 + 0.000156 C^2 \quad (8)$$

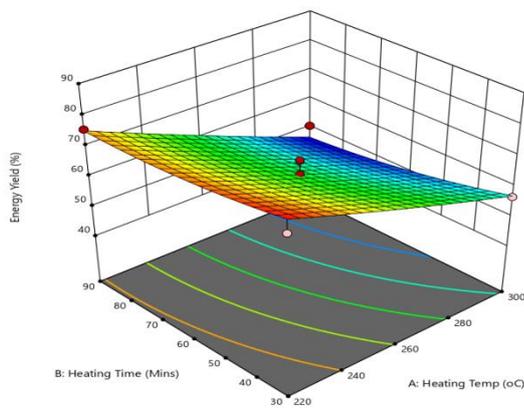
where: B_{bed} = Biochar energy density of brimstone wood, A = Heating temperature, °C, B = Heating time, min, C = Sample size, mm

The ANOVA results and statistical parameters of the analysis which describes the relations between the independent variables and the dependent variable, biochar energy density is as presented in Table 6. The probability of the model P-value and F-value of 0.0001 and 2803.33 of brimstone wood biochar energy density, indicates that the model suggested by the software for the material was significant at 95 % confidence level. The R^2 value evaluated for biochar energy density of brimstone wood is 0.9990 meaning 99.90 % of the variability can be accounted for showing the regression model was satisfactory for the prediction of biochar energy density brimstone wood. The ANOVA results for the model terms presented in Table 8 shows that A , B , AB , AC , BC and A^2 are significant model terms, while it is shown from Equation 8 that A , AB , AC , BC and C^2 have synergistic effect on biochar energy density and B , C , A^2 and B^2 have antagonistic effect.

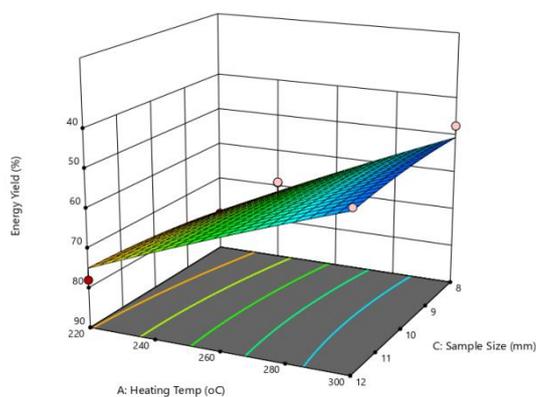
Table 6: ANOVA Result of Energy Density of Brimstone Wood

Source	Sum of Squares	Df	Mean Square	F-value	p-value	Conclusion
Model	0.0184	9	0.0020	543.84	< 0.0001	Significant
A	0.0105	1	0.0105	2803.33	< 0.0001	Significant
B	0.0015	1	0.0015	405.00	< 0.0001	Significant
C	2.083E-06	1	2.083E-06	405.00	0.4896	Not significant
AB	0.0000	1	0.0000	6.67	0.0493	Significant
AC	0.0004	1	0.0004	106.67	0.0001	Significant
BC	0.0000	1	0.0000	7.50	0.0409	Significant
A ²	0.0041	1	0.0041	1083.000	< 0.0001	Significant
B ²	0.0000	1	0.0000	3.00	0.1438	Not Significant
C ²	1.250E-06	1	1.250E-06	0.33333	0.5887	Not Significant
Residual	0.0000	5	3.750E-06			
Lack of Fit	0.0000	2	9.375E-06			
Pure Error	0.0000	3	0.0000			
Cor Total	0.0184	14				
Statistical Parameters			R ² =	0.9990	Adj R ² =	0.9971
			Pred R ² =	0.2959	Adeq Precision =	68.384

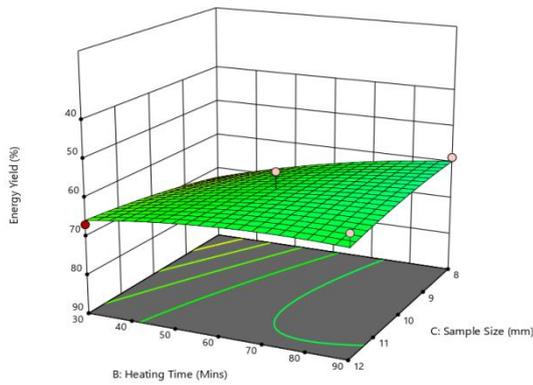
The three-dimensional surface graph (3D) and two-dimensional contour (2D) in Figures 3 (a, b and c) shows the effect of heating temperature, heating time and sample size on the biochar energy density of brimstone wood. The combined effect of heating temperature and heating time on the biochar energy density on brimstone wood is shown in Figure 3a in form of three-dimensional surface graph. Figure 3a shows that energy density increases as the values of heating temperature and heating time increases. The maximum and minimum biochar energy density was obtained to be 1.15 and 1.04 at 300 °C, 90 mins and 220 °C, 30 mins respectively. Figure 3b shows that the energy density increased with increase in heating temperature and sample size. The maximum and minimum biochar energy density of 1.14 and 1.05 obtained at 300 °C, 12mm and 220 °C, 8mm respectively. Also, Figure 3c shows that increase in heating time gives increase in biochar energy density and vice versa. The maximum and minimum biochar energy density were obtained to be 1.15 and 1.11% at 90 mins, 8 mm and 30 mins, 8 mm respectively. Similar increase in biochar energy density of poplar chip from 0.99 to 1.15 at temperature range of 200 to 300 °C was reported by Xun Luo (2011).



(a)



(b)



(c)

Figure 3: Response surface of biochar energy density of brimstone wood as influenced by (a) heating temperature and heating time (b) sample size and heating temperature (c) heating time and sample size

3.3 Ultimate Composition of Torrefied Brimstone Wood Solid Products

The results obtained for the ultimate analysis of the solid torrefied brimstone wood products are as presented in Table 7. In the Table, the torrefaction holding time for the experiments was 60 min. From Table 7, it can be inferred that the weight percentage of carbon in the torrefied solid products increased from 55.21 - 67.40% for the brimstone wood as the torrefaction temperature is increased from 220 to 300 °C. Similar increase in carbon content of Colombian wood species from 52.73 to 55.01 as torrefaction temperature is increased from 200 to 300 °C have been reported by Margareta *et al.* (2021). The increase in the carbon content is only an apparent increase due to the decrease in the oxygen content from 43.74 to 38.05% for patula pine at torrefaction temperature range of 200 to 300 °C. These results indicate that the torrefaction process increases the energy density of the products by removing oxygen (Sergio Ramos *et al.*, 2017 and Jinxia Fu *et al.*, 2021). From Table 7, it can be deduced that the nitrogen content increased slightly from 0.57 to 0.83% for the brimstone wood. This result is similar with increase in nitrogen from 0.13 to 0.15% for the patula pine torrefied from 200 to 300 °C reported by Sergio Ramos *et al.* (2017). The increase in nitrogen is a relative increase due to decrease level of oxygen (Jaya, 2018). While sulfur content did not vary much with an increase from 0.87 to 0.64% and from 1.70 to 1.55% for the brimstone wood at torrefaction temperature between 200 to 300 °C. Similar observations were made by Jaya (2016) that sulfur content decreased from 0.25 to 0.11 % for 160 and 270 °C torrefaction temperature for lodge pine grind.

From Table 7, the energy content obtained for the brimstone wood products as presented shows that the energy content increased from 26.20 to 28.55 MJ/Kg for brimstone wood with increased in torrefaction temperature from 220 to 300 °C. Similar increases in energy content were reported for wood waste from 20.18 to 24.00 MJ/kg by Margareta *et al.* (2021) at torrefied temperature range of 225 to 300 °C. The energy content of the products indicates the ability to do work. The higher the energy contents of a sample the higher the work that will be done (Unroto *et al.*, 2020).

Table 7: Ultimate Composition of Raw and Torrefied Brimstone Wood

Sample	Properties	Unit	Raw	Heating Temperature, °C		
				220	260	300
Brimstone Wood	Carbon Content	%	52.20	55.21	57.96	67.40
	Oxygen Content	%	36.88	35.55	33.40	29.50
	Hydrogen Content	%	2.40	2.32	2.21	2.13
	Nitrogen Content	%	0.54	0.57	0.67	0.83
	Sulfur Content	%	0.89	0.87	0.71	0.64
	Energy Content	MJ/kg	24.59	26.20	27.85	28.55

4. Conclusions

- i. The surface colour of the torrefied products (biochar) changes with increase in torrefaction conditions. Biochar mass yield decreases with increases in torrefaction conditions for the biomass materials. Biochar energy yield increases as the value of torrefaction conditions decreased for the two samples. While Biochar energy density increases as the torrefaction condition increases. Similar results were reported by (Sergio Ramos *et al.*, 2017 who worked on patula pine wood.
- ii. The values of ultimate composition of carbon, oxygen, hydrogen, nitrogen, sulfur and energy contents of biochar products for the brimstone wood have been obtained. The energy content increased from 26.20 to 28.55 MJ/kg for brimstone wood with increased in torrefaction temperature from 220 to 300 °C.

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