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Production of Medium-Grade Fuel from Pyrolysis of Teak Tree Leaves

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***Abstract*: This study investigated the effects of selected process parameters (temperature and time) on bio-fuel yields during pyrolysis of teak tree leaves in a fixed bed reactor. The experiment was designed by applying optimal custom methodology through a two-categoric factor design using Design Expert 12.0.1.0 version software. The batch process was carried out at five levels of heating temperature of 300, 350, 400, 450, and 500 ℃, for five levels of residence times of 10, 15, 20, 25, and 30 minutes. The value of the product yields (bio-char, bio-oil, bio-gas) produced from teak tree leaves was determined using the analysis of variance (ANOVA). The adjusted coefficient of determination (R2) statistic quantifies the proportion of the parameter variability that can be accounted for by the model. Other model equations were developed using the coefficient of determination R2, and an F-value determined their significance. Proximate analysis of teak tree leaves revealed moisture contents of 11.18%, volatile matter of 63.14%, ash content of 17.23%, and fixed carbon of 8.45%. Ultimate analysis showed carbon 41.60%, hydrogen 16.95%, nitrogen 10.58%, sulfur 0.13%, oxygen 30.74%, and higher heating values of 19.34 MJ/kg. Maximum bio-oil yields of 38.41wt% were achieved at 450°C for 25 minutes. Cellulose, hemicellulose, and lignin contents were 14.00, 25.40, and 23.40% respectively.**

***Keywords*: pyrolytic conversion, teak tree leaves, bio-fuel, bio-energy, environmental sustainability, waste management.**

1. Introduction

Biomass, a promising source of renewable and sustainable energy, is an important means of solving energy problems in the world, especially in developing countries (Lucas and Itabiyi, 2012). Over time, fossil fuel has become a popular energy source and it has posed a lot of adverse effects on the environment as a result of greenhouse gas (GHG) emissions (Walsh, 2000; Thomas, 2018; Yaman, 2006; Sangotayo *et al.,* 2017). Biomass has proved to be an alternative source of energy and is environmentally friendly. The availability of biomass makes it of utmost importance to pyrolysis research in terms of clean energy generation (Seo *et al*., 2010; Demiral *et al*., 2008; Balat *et al*., 2009). European Union (EU) and United Nations (UN) regulations consider biomass as a renewable source of energy.

Pyrolysis is a thermo-chemical process that involves breaking down larger molecules into smaller molecules under heat in the absence of oxygen for a short period (Thomas, 2018). Pyrolysis can be described as a thermo-chemical conversion of biomass into pyrolysis products (bio-oil, bio-char, and bio-gas) (Montenegro *et al.,* 2016). This process which involves thermo-chemical decomposition of plant and animal residues in the absence of an oxidizing agent can be regarded as the initial stage of gasification and combustion. Furthermore, bio-oils which are characterized by a high content of volatile acids, moisture, oxygenated compound, viscosity, and aging can be upgraded by pre-treatment of biomass.

Despite the availability and potential, the pyrolytic conversion of teak tree leaves to medium-grade fuels has not been widely explored in Nigeria. This presents an opportunity to utilize these waste materials for energy production while addressing environmental and waste management challenges. Thus, this study investigated the pyrolytic conversion of locally sourced teak tree leaves into medium-grade fuels. The procedure involves determining the ultimate and proximate composition of teak tree leaves to assess their suitability for pyrolysis and evaluating the effects of key parameters, such as pyrolysis time and temperature, on the yield of pyrolysis products. Pyrolysis of teak tree leaves offers the dual benefits of transforming waste into useful fuels with lower harmful emissions and mitigating environmental pollution, resource wastage, and greenhouse gas emissions. Thus, this study contributes to sustainable waste management practices and addresses energy and environmental concerns.

2. Materials and Methods

A. Materials

Teak tree leaves Tectona grandis were sourced locally in Olofin, Iseyin Local Government, Oyo State, South-Western Nigeria. The sample was sundried for two days to reduce its moisture content. After sun drying, it was weighed and sealed in an airtight bag to prevent moisture absorption and kept at room temperature before pyrolysis experiments.

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B. Experimental Process Design Using Response Surface Methodology (RSM)

The experimental design for the effect of temperature and time on the product yields for teak tree leaves was carried out using Design Expert version 12.0.1.0. The experiment was based on the fact that the factors chosen were functionally related to the pyrolysis product yields. The optimal (custom) design was selected with two categoric factors (Temperature and Time), and a five-level design approach was also adopted, with the feedstocks' pyrolysis temperatures (300,350,400,450 and 500℃,) and pyrolysis time (10, 15,20, 25, and 30 mins) serving as the independent or input variables, and the pyrolysis product yields, which are bio-char, bio-oil, and bio-gas, are the dependent variables.

The statistical analysis and generation of response surface plots were performed on the pyrolysis process variables using Design Expert 12.0.1.0. The value of the bio-char, bio-oil, and bio-gas produced from teak tree leaves was determined using the analysis of variance (ANOVA). The adjusted coefficient of determination (R^2) statistic quantifies the proportion of the parameter variability that can be accounted for by the model. Other model equations were developed and appropriately expressed by the coefficient of determination R^2, and their significance was determined by an F-value.

C. Experimental Setup and Procedure

The teak tree leaves pyrolysis was conducted using a fixed bed pyrolysis system, as shown in Figure 1. The pyrolysis system consists of a retort, condensate receiver unit, and gas collection unit, all made of mild steel. The teak tree leaves were pyrolyzed in the absence of oxygen. 100 g of the teak tree leaves each was fed into the retort according to the number of runs determined by Design Expert Version 12.0.0. Then, bolts and nuts were covered and tightened with a gasket incorporated to prevent gas leakage. The retort was placed inside the clay brick-lined electric furnace and pyrolyzed by varying the temperature from 300 to 500℃, at 50 ℃, intervals with a varying residence time of 10 to 30 minutes at an interval of 5 minutes. The retort was connected through a lagged galvanized pipe to the condensate receiver, and the valve on the condensate receiver was closed for a few minutes before gas release. This was done to condense an appreciable amount of gas to liquid before allowing uncondensed gases to flow to the gas collection unit. After condensation, the valve was opened to allow uncondensed gases to escape into the gas collection unit for scrubbing. After the prescribed holding time, the process was terminated, and the bio-char was removed from the retort and allowed to cool and then measured using the Ohaus top loading weighing balance as well as bio-oil yields and expressed in percentages of the weight of the initial raw sample of the teak tree leaves according to equation 1,2 and 3.

$$\%Bio-Charyield = \frac{Massofthecharobtained}{Massoftherawsamples} \times 100 \quad (1)$$

$$\%Bio-oilyield = \frac{Massoftheliquidobtained}{Massoftherawsamples} \times 100 \quad (2)$$

$$\%Bio-Gas\ yield = 100 - (\%Bio-Charyield + \%Biooilyield) \quad (3)$$

on the experimental design, this procedure was repeated for all other samples at different pyrolysis temperatures and residence times. The optimum temperature and holding time for the bio-oil yield were then recorded for further analysis and assessment.

Fig. 1. Pyrolysis equipment setup (Renewable energy lab. LAUTECH)

D. Bio-oil Characterization and Ultimate Yield Analysis

The elemental composition of the bio-oils was carried out using the CHONS Elemental Analyzer. The Sulphur content of the sample was determined using a spectrophotometer, and the amount of oxygen in the sample was obtained by adding the percentage total of carbon, Sulphur, nitrogen, and hydrogen and subtracting from 100. Mathematically,

$$\%oxygen = 100 - (\%\ of C + N + S) \quad (3)$$

This study analyzes the yield performance of the pyrolytic process using the statistical performance measure known as the Signal-to-Noise Ratio (SNR). SNR measures the desired signal level against the background noise level, and for pyrolysis product efficiency, SNR should be as high as possible. The SNR was calculated using equation (4) (Başar et al., 2022).

$$SNR = -10 \log\left[\frac{1}{n}\sum_{i=1}^{n}\frac{1}{y_i^2}\right] \times 100 \quad (4)$$

y_i is the % yield of each pyrolytic product as obtained from equations (1), (2), and (3), and n is the number of experimental runs.

3. Results and Discussion

A. Physical and Chemical Composition of Teak Tree Leaves

Table 1 shows the results of the physical and chemical properties of teak tree leaves. The moisture content for teak tree leaves is 11.18%. High water content can affect the quality of the bio-oil produced because the high-water content in bio-oil can cause a low heating valve (Sakulkit *et al*., 2020). However, excess moisture content can also help reduce the viscosity of bio-oil and reduce NOx emissions (Pimenidou *et al*., 2012). The ash content for teak tree leaves is 17.23%. The amount and chemical composition of ash have effects on the fuel properties of biomass, and therefore important for the selection of the type of conversion technology (Itabiyi, 2014: Demirbas, 2009). Low ash content means low slagging and fouling effects. Biomass with less ash content is reported to have higher heating values as compared with those containing higher ash content. Although ash does not contribute to the overall heating value, certain elements and alkali metals in the ash are catalytic to thermal decomposition, low ash content also has been reported to maximize bio-oil yield (Boateng *et al.,* 2006). The volatile content obtained for teak tree leaves is 63.14%. Combustible volatile content aids the onset of pyrolysis by yielding at higher temperatures as it burns. Ultimate analysis results showed that teak tree leaves have 41.60% C, 16.95% H and 30.74% O2. These elements constitute the major composition of teak tree leaves as this is evident from the results of elemental analysis in Table 1. These values are comparable to some biomass such as tanjong leaves. Tanjong leaves have 38.50% C,5.23% H, and 40.28% O_2 (Kalita and Saikia, 2004).

Table 1
Results of physical and chemical composition of teak tree leaves

Composition	Teak Tree Leaves
Moisture content (%)	11.18
Ash content (%)	17.23
Volatile matter (%)	63.14
Fixed carbon content (%)	8.45
Carbon (%)	41.60
Hydrogen (%)	16.95
Oxygen (%)	30.74
Sulphur (%)	0.13
Nitrogen (%)	10.58
Cellulose (%)	14.00
Hemicellulose (%)	25.40
Lignin (%)	23.40
Lower Heating Valve (MJ/kg)	17.09
Higher Heating Value (MJ/kg)	19.34

B. Statistical Analysis Models

1) Teak tree leaves

The analysis and prediction of the model for biochar yield from teak tree leaves were determined by applying the Design Expert (12.0.1.0) version. The significance of the statistical models for these equations was assessed using the F-test analysis of variance (ANOVA), as shown in equations (5), (6), and (7).

$$C_Y=39.74 + 10.41(A1) + 2.65(A2) + 0.0364(A3) - 5.69(A4) + 4.99(B1) +2.57 (B2) + 0.3604(B3) – 3.43(B4) \quad (5)$$

$$B_Y=29.025 – 6.16(A1) – 0.8328(A2) + 0.652(A3) + 4.31(A4) – 0.02(B1) – 0.02(B1) – 0.7088(B2) – 0.1812(B3) + 1.66(B4) \quad (6)$$

$$G_Y= 30.91 – 3.96(A1) – 1.53(A2) – 0.4056(A3) + 0.7924(A4) – 2.67(B1) – 1.56(B2) – 0.2456(B3) + 0.9824(B4) \quad (7)$$

Where,
C_Y= Bio-char yield (wt%),
B_Y = Bio-oil yield (wt%),
G_Y= Bio-gas yield (wt%),
A = Temperature (degree), B = Time (min).

The probability values for prob > F being less than 0.05 in Table 2 indicated that the quadratic models for the response variables were statistically significant at a 95% confidence level. The F-statistic values for the bio-char, bio-oil, and bio-gas confirmed that the models were significant. The model terms A, B, A2, B2, and AB also significantly affected the biochar, bio-oil, and bio-gas yields.

The statistical parameters obtained from the ANOVA for the developed models are presented in Table 3. These parameters were used to establish the accuracy of the developed models. The bio-char yields model showed a relatively high determination coefficient, R^2 value of 0.9878, and a low coefficient of variation, C.V value of 2.56. The corresponding values obtained for bio-oil yields were an R^2 of 0.9714 and a C.V. value of 2.80; for bio-gas yield models, an R^2 value of 0.9824 and a C.V. value of 1.99 were obtained. The closer the determined coefficient to unity, the better the model suits the experimental data, indicating less difference between the calculated and measured values.

Model adequacy was further assessed through the adjusted R^2, Predicted R^2, and adequate precision. Adequate precision measures the Signal Noise Ratio (SNR), and an SNR value greater than 4 is desirable. Therefore, the SNR of 44.8212 for bio-char yields, 28.9487 for bio-oil yields, and 41.2887 for biogas yields in the analysis indicates that the model can be used to navigate design space.

C. Effect of Pyrolysis Parameters on the Product Yields

1) Teak Tree Leaves

Figure 2 shows the three-dimensional response surfaces and contour plots of teak tree leaves pyrolysis product yields. These figures showed the combined effect of pyrolysis temperature and residence time on biochar, bio-oil, and bio-gas product yields. The product yields from the pyrolysis experiments at different temperatures (300, 350, 400, 450, and 500°C), with residence time (10,15, 20, 25, and 30mins) show that the bio-char decreased as the pyrolysis temperature increased from 300°C to 500°C as shown in Figure 2(a). The maximum biochar for the teak tree leaves was obtained as 55.64% at a pyrolysis temperature of 300°C and residence time of 10 mins.

The biochar agreed with the pyrolysis convention that, as the pyrolysis temperature increases, the amount of char generated decreases (Ola *et al*., 2014). Bio-oil increased as the pyrolysis temperature increased from 300 to 450°C and then decreased at 500°C with residence time, as shown in Figure 2(b). The highest bio-oil yields were obtained at 38.41% at a pyrolysis temperature of 450°C, with a residence time of 25 minutes. The results showed that the bio-gas increased as the pyrolysis temperature increased with residence time, and the highest bio-gas was obtained as 40.27% at a pyrolysis temperature of 500°Cand at a residence time of 30 mins, as shown in Figure 2(c); this indicates that bio-gas increased as the temperature and time increased, respectively. This result is consistent with

Table 2
Analysis of variance (ANOVA) for product yields (Bio-char, Bio-oil, and Bio-gas) from teak tree leaves

Response 1: Bio-Char (%)

Source	**Sum of Squares**	**Df**	**Mean Square**	**F-value**	**p-value**	
Model	1331.83	8	166.48	161.43	< 0.0001	Significant
A-Temperature	1014.42	4	253.60	245.91	< 0.0001	
B-Time	317.41	4	79.35	76.95	< 0.0001	
Residual	16.50	16	1.03			
Cor Total	1348.33	24				

Response 2: Bio Oil (%)

Source	**Sum of Squares**	**Df**	**Mean Square**	**F-value**	**p-value**	
Model	361.40	8	45.18	68.05	< 0.0001	Significant
A-Temperature	320.59	4	80.15	120.73	< 0.0001	
B-Time	40.81	4	10.20	15.37	< 0.0001	
Residual	10.62	16	0.6639			
Cor Total	372.02	24				

Response 3: Bio Gas (%)

Source	**Sum of Squares**	**Df**	**Mean Square**	**F-value**	**p-value**	
Model	337.71	8	42.21	111.90	< 0.0001	Significant
A-Temperature	223.60	4	55.90	148.17	< 0.0001	
B-Time	114.11	4	28.53	75.62	< 0.0001	
Residual	6.04	16	0.3773			
Cor Total	343.74	24				

Table 3
Model comparison statistics of product yield (Bio-char, Bio-oil, and Bio-gas) from teak tree leaves

Bio-Char yield			
Standard Deviation	1.02	R^2	0.9878
Mean	39.74	Adjusted R^2	0.9816
C.V. %	2.56	Predicted R^2	0.9701
		Adequate Precision	44.8212
Bio-oil yield			
Standard deviation.	0.8148	R^2	0.9714
Mean	29.05	Adjusted R^2	0.9572
C.V. %	2.80	Predicted R^2	0.9303
		Adequate Precision	28.9487
Bio-Gas Yield			
Standard deviation	0.6142	R^2	0.9824
Mean	30.91	Adjusted R^2	0.9737
C.V. %	1.99	Predicted R^2	0.9571
		Adequate Precision	41.2887

recent research that found that bio-oil yields and bio-gas increased as temperature increased (Lazzari *et al*. 2016 and Rivas *et al*. 2020).

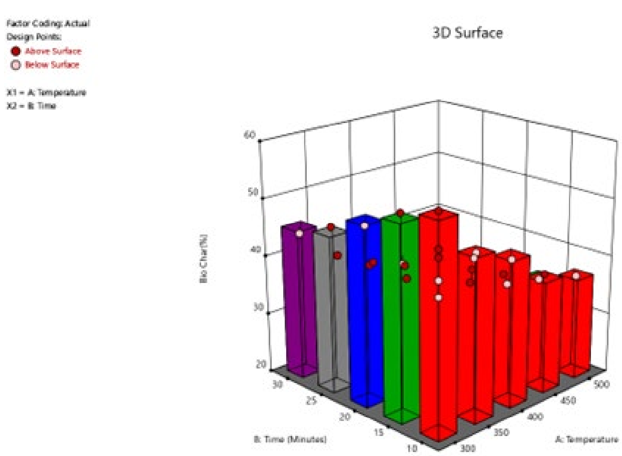

Fig. 2(a). Response surface 3D plot showing the interaction and effects of temperature and time on char yield from pyrolysis of teak tree leaves

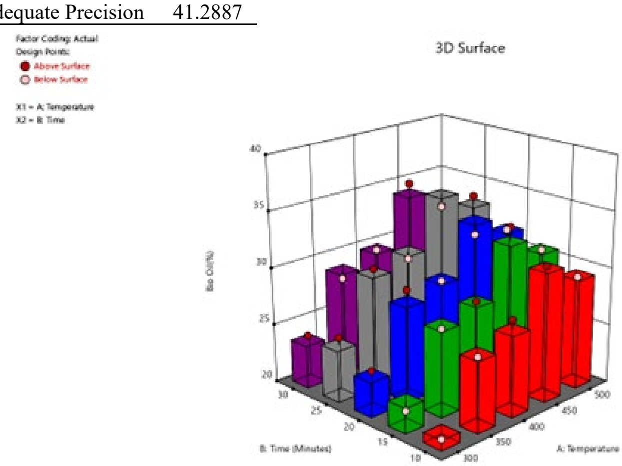

Fig. 2(b). Response surface 3D plot showing the interaction and effects of temperature and time on bio-oil yield from pyrolysis of teak tree leaves

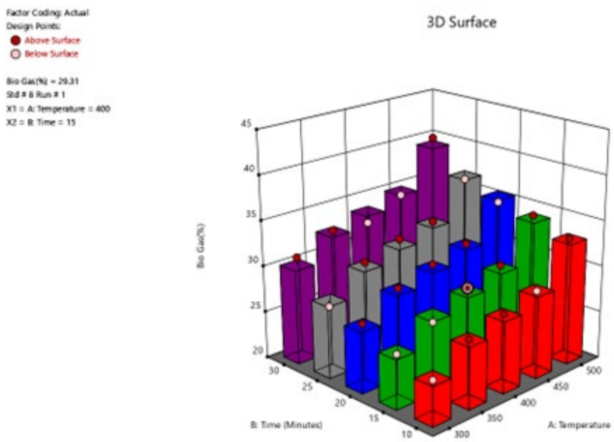

Fig. 2(c). Response surface 3D plot showing the interaction and effects of temperature and time on bio-gas yield from pyrolysis of teak tree leaves

4. Conclusion

The effect of temperature and time on the bio-fuel yield from pyrolysis of teak tree leaves at optimum temperature and time have been investigated and analyzed. The feedstock analysis revealed that teak tree leaves have a high potential for biofuel production due to their high carbon and hydrogen content and low sulfur content. The moisture content, ash content, volatile matters, and fixed carbon results of the leaves were 11.18, 17.23, 63.14, and 8.45 %, respectively. Maximum bio-char (55.64%), bio-oil (38.41%), and bio-gas (40.27%) were obtained at temperatures of 300, 450, and 500℃, respectively, and residence time of 10, 25, and 30 minutes in the same order. The bio-oil produced can be used as an alternative fuel for domestic and industrial applications.

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