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INFLUENCE OF TORREFACTION TEMPERATURE ON THE CHEMICAL COMPOSITION OF *Leucaena leucocephala* GROWN IN NIGERIA

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ABSTRACT

Wood is polymeric with varying quantities of extractives and inorganics. The quantity of the polymeric substances (Cellulose, Hemicellulose and Lignin) can be changed by torrefaction. Hence, this study investigated the influence of torrefaction on the chemical composition of Leucaena leucocephala within the temperature range of 225 to 300°C. The compositional analysis of the lignocellulosic biomass involved the utilisation of both wet chemistry and gravimetric methods. These methods determined the quantities of hemicellulose, cellulose, Klason lignin, extractives, and ash content. The findings revealed notable variations in extractive, hemicellulose, and cellulose content across different temperatures and parts of the biomass. The hemicellulose content of Leucaena ranged from 24% to 33%. There was no significant difference in the ash content, although an increase in value from 1.57 at 225°C to 2.71 at 300°C was observed. Furthermore, the cellulose content decreased with rising temperature, whereas the lignin content increased. These results provide insights into the chemical transformations occurring in wood during torrefaction.

Keywords: Torrefaction, Wet chemistry, Cellulose, Hemicellulose, Lignin, Leucaena leucocephala

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INTRODUCTION

Renewable energy sources are crucial for meeting the escalating energy demands of the expanding global population and replacing fossil fuels in the long term, with the potential to mitigate greenhouse gas emissions (Chen *et al.,* 2015). Bioenergy offers various options, among which biomass energy exhibits exceptional potential for further advancement. Thermochemical conversion is a primary pathway for biomass-to-energy conversion. Woody biomass, currently used for renewable energy, is a significant source. However, its utilisation encounters challenges such as high moisture content, low bulk and energy density, and inadequate grindability (Wang *et al.,* 2018). Additionally, the heterogeneous nature of woody biomass in terms of chemical composition necessitates implementing effective pretreatment methods.

Wood pretreatment plays a vital role in enhancing wood properties. Different researchers have employed several pretreatment techniques; Xu *et al.* (2206 and Rulkens (2006) used the drying method, Bitra et al. (2009); Estebean and Carrasco (2006) and Kobayashi et el. (2009) did size reduction by pulverisation; Miranda *et al* (2009), Gilbert *et al* (2009) and Demirbas and Sahin-Demibars (2009) densified and pelletised biomass in other to improve different properties of biomass. In addition to these conventional pretreatments, torrefaction is a unique method for enhancing biomass as a fuel or energy feedstock, addressing the limitations associated with woody biomass utilisation. Torrefaction is a thermochemical pretreatment carried out under an inert atmosphere in the temperature range of approximately 200-300°C. This process

significantly enhances the fuel properties of wood, modifies its chemical composition, and reduces moisture content. Torrefaction helps improve the stowing, handling and production of pellets from different biomasses due to the nature and complex polymers of woody biomass consisting of cellulose, hemicelluloses, and lignin as primary polymers. The impact of torrefaction temperature on wood polymers is notable (Wenjia *et al.,* 2013). Torrefaction affects tree species differently regarding the thermal decomposition of hemicelluloses and cellulose, energy value, and combustion properties (Wenjia *et al.,* 2013; Fuwape and Faruwa, 2016). Lignin, a wood component, decomposes within the temperature range of 280 to 500°C (Wenjia *et al.,* 2013), yielding phenol through the cleavage of ether and carbon-carbon linkages. Lignin produces more residual char than cellulose or hemicelluloses (Mohan *et al.,* 2006). Bio-oil yields were reported to be 65% in cellulose pyrolysis, while xylan pyrolysis yielded 53%, and lignin pyrolysis yielded 40% (Qu *et al.,* 2011). Independent decomposition of the woody biomass polymers, without interference, has been demonstrated by Raveendran *et al.* (1995) and Qu *et al.* (2011). Considering that torrefaction influences the organic nature of biomass, studying the chemical alterations of woody biomass becomes imperative. Hence, this study aims to investigate the influence of torrefaction on the hollocellulose, lignin, ash, and extractives content of a twenty-year-old *Leucaena leucocephala* grown under a tropical plantation.

MATERIALS AND METHODS

Torrefaction and Sample Preparation

In this study, *Leucaena leucocephala* wood harvested from the forest plantation of the Federal University of Technology Akure was used. The merchantable tree was divided into the top, middle, and base. Wood samples with 10 x 10 x 60 mm dimensions were prepared and oven-dried at 103 ± 2°C until the moisture content stabilised. Torrefaction was conducted at four different temperature regimes: 225°C, 250°C, 275°C, and 300°C. The torrefaction method used in this study followed the procedure described in Faruwa *et al.* (2021).

Torrefaction

A fixed bed reactor developed at the Federal University of Technology Akure (FUTA) was employed to assess the impact of torrefaction parameters on properties modification. A batch of 300 g of dimensioned wood samples (10 x 10 x 60 mm) was subjected to torrefaction in the fixed bed reactor at the specified temperature range. After torrefaction, the samples were placed in an air-tight chamber to cool down and prepare for further analyses.

Chemical Reagents and Methods

All chemicals and reagents used in this study were of analytical grade and commercially available. The compositional analysis of lignocellulosic biomass was carried out using the gravimetric method, as detailed by Ayeni *et al.* (2013, 2015). This method was chosen for its proven result reproducibility, representing biomass analysis, and its economic viability, making it suitable for developing countries. The ash content and wood extractives (WE) were quantified using the gravimetric analysis method, following the procedure reported by Rabemanolontsoa (2011) and TAPPI (1988). The extractives, including lipophilic wood components and low molecular phenolic compounds, were extracted using acetone. The extractives were expressed as a total percentage weight for all extractives. Wood extractives (WE) were calculated using the formula:

$$WE = \frac{W_0 - W_1}{W_0} [\%] \ldots\ldots (1)$$

Where:
WE = Wood Extractive, in %;
where: WE = Wood Extractive, in %,
W0 = Weight of Wood before extraction, in g,
W1 = Weight of wood after extraction, in g.
Proximate analysis was conducted on all torrefied samples to determine the percentage of ash content (PAC), following the procedures outlined by Kurada (2000).

Hemicellulose Determination

To determine the hemicellulose content, 1 g of extracted biomass (W2, g) was transferred into a 250 mL Erlenmeyer flask. Accurately measured, 150 mL of 0.5 M NaOH was added to the flask, and the mixture was boiled in a water bath for 3.5

hours. The boiled mixture was allowed to cool at room temperature. The slurry was then filtered through vacuum filtration, and the residues were dried at 103 ± 2°C until a constant weight was achieved. The differential value before and after the treatment account for the hemicellulose content (Ayeni *et al.,* 2013, 2015; Blasi *et al.,* 1999; Li *et al*., 2004).

Lignin Content Determination

200 mg of acetone-extracted wood samples were placed in small test tubes to determine the lignin content. Each sample was treated with 2 mL of 72% H_2SO_4 and incubated at 300°C for 60 minutes with stirring. The resulting hydrolysates were poured into an Erlenmeyer flask and mixed with distilled water. The slurry underwent a second hydrolysis step in an autoclave for 30 minutes. After cooling, the hydrolysates were filtered, and the acid-insoluble lignin was dried and accounted for ash content. The acid-soluble lignin fraction was determined by measuring absorbance at 320 nm. The lignin content was calculated by summing up the acid-insoluble and acid-soluble lignin fractions.

Cellulose content Determination

The cellulose content was calculated by subtracting the percentages of extractives, hemicellulose, lignin, and ash from 100. These methods are based on previous studies (Sluiter *et al.,* 2008; Ayeni *et al*., 2013; Blasi *et al.,* 1999; Li *et al.,* 2004)

RESULTS

Moisture Content (MC)

The ANOVA results (Table 1) showed no significant differences among the different parts (Top, Middle, Base) of the torrefied samples for this study. However, significant differences were observed in moisture content across the temperature regimes. The mean values (Table 2) indicated that the raw sample had the highest moisture content at 10.29%, while the temperature of 300°C had the lowest at 3.12%.

Wood Extractives

The statistical analysis (Tables 1 and 2) revealed no significant differences in extractive content among the different temperature regimes and tree parts considered in this study. The mean value for the torrefied samples ranged between 3.32 % (250 ºC) and 4.77 %(300ºC); this suggests that the mean values of extractive content were not statistically different for the leucaena wood samples at different temperatures.

Ash Content

The analysis of variance (Table 2) indicated significant differences in the ash content of Leucaena leucocephala across different tested parameters of this study. The mean separation results showed that the ash content was not statistically different between temperatures 250°C and 275°C, but they were significantly different from temperatures 225°C and 300°C. The mean values revealed increased ash content with increasing temperature, ranging from 1.57% to 2.71% for temperatures 225°C and 300°C, respectively.

Table 1: ANOVA table for Moisture content, *extractives, hemicelluloses, lignin and celluloses* of *Leucaena leucocephala*

Moisture Content					
Source	**SS**	**df**	**MS**	**F**	**Sig.**
Torrefaction Temprature	107.09	4	26.77	4.89	.03
Tree parts	27.83	2	13.92	2.54	.14
Error	43.79	8	5.47		
Total	**178.71**	**14**			
Extractives					
Source	**SS**	**df**	**MS**	**F**	**Sig.**
Torrefaction Temprature	16.81	4	4.20	2.06	.18
Tree parts	7.36	2	3.68	1.80	.23
Error	16.34	8	2.04		
Total	**40.52**	**14**			
Hemicelluloses					
Source	**SS**	**df**	**MS**	**F**	**Sig.**
Torrefaction Temprature	131.60	4	32.90	6.09	.02
Tree parts	12.13	2	6.08	1.12	.37
Error	43.20	8	5.40		
Total	**186.93**	**14**			
Lignin					
Source	**SS**	**df**	**MS**	**F**	**Sig.**
Torrefaction Temprature	347.22	4	86.81	5.32	.02
Tree parts	184.48	2	92.24	5.65	.03
Error	130.65	8	16.33		
Total	**662.35**	**14**			
Celluloses					
Source	**SS**	**df**	**MS**	**F**	**Sig.**
Torrefaction Temprature	336.36	4	84.09	10.34	.00
Tree parts	135.37	2	67.68	8.32	.01
Error	65.06	8	8.13		
Total	**536.79**	**14**			

Table 2: Mean value for Moisture content, extractives, Hemicelluloses, lignin and Celluloses of Leuceana leucocephala

Temperature (ºC)	**MC (%)**	**WE (%)**	**Hemicelluloses (%)**	**Ash (%)**	**Lignin (%)**	**Celluloses (%)**
Raw	10.29^{a}	6.67^{a}	27.23	1.44	36.70	27.96
225	5.69^{bc}	4.27^{a}	33.00	1.57^{c}	47.32^{b}	18.11
250	5.10^{ab}	4.00^{a}	30.57	2.10^{b}	46.06^{b}	17.27
275	4.89^{bc}	3.32^{a}	29.00	2.55^{b}	47.31^{b}	17.82
300	3.12^{c}	4.77^{a}	24.00	2.70^{a}	50.39^{a}	18.14

Values with different superscripts are significantly different along the column.

Hemicelluloses

The ANOVA analysis showed a significant difference in hemicellulose content among the different temperatures of the samples (P value ≤ 0.05), but no significant difference was observed among the different parts. The mean values for Leucaena indicated a hemicellulose content ranging from 24% to 33%. The result showed a decrease in the quantity of cellulose with an increase in temperature. The reduction in hemicellulose content was 7.9 % from 225°C to 250°C, while the decline between 250°C to 275°C was about 5.4 and a 20 % reduction was observed between 275°C and 300°C.

Lignin Content

The ANOVA revealed significant differences in lignin content across different temperatures and parts of the samples (P ≤ 0.05). The mean values showed that the un-torrefied sample had the lowest lignin content at 36.70%, while a temperature of 300°C had the highest content at 50.39%. The lignin content increases with the increase in temperature used in this study, except at 250°C, where a reduction was recorded between 225°C and 250°C.

Cellulose Content

The ANOVA (Table 1) indicated that cellulose was significantly impacted by temperature across different tree parts. The cellulose content ranged from 13.84% to 18.14%(Table 1). The result further revealed a slight change in the quantity of cellulose across the varying temperature.

DISCUSSION

The moisture content in this study, as presented in Table 2, was within the reported value of Jim *et al.* (2000), where the highest moisture content value was reported not to exceed 12%. Also, previous studies on the torrefaction of reed canary grass and wheat straw by Bridgeman *et al.* (2008) showed a moisture content reduction from a 4.7% to 0.8% initial value. The decrease in moisture content can be attributed to the loss of bond water within the cell wall at elevated temperatures due to disintegration in the hydroxyl group (Faruwa *et al.,* 2021).

Previous studies revealed varying trends in extractive content with increasing torrefaction temperature. Some studies observed an increase in extractable content up to a specific temperature, followed by a decrease. Others reported a continuous increase or decrease. In this study, the extractive content ranged between 2-8%, which is consistent with the findings of Sixta (2006), but at variance with the study of Shebani *et al* (2008), where a lower value between 2-5 % was reported and further suggested that the valued could be up to 15 % in some species of trees. The result of this study aligns with the investigations of Hakkou *et al.* (2005), Windeisen *et al*. (2007), and Pierre *et al.* (2011), which indicated that extractive within the cell tends to increase at insignificant torrefaction temperatures (around 240°C) and decrease as temperature increases. This change in extractive content can be attributed to the depolymerisation of different extractable materials from wood polymers during torrefaction processes. The trend observed for ash in this study aligns with a similar study conducted by Chun-Te and Far-Ching (2012) and Ho *et al.* (2014) for Larch wood, which reported an increasing ash content with higher reaction temperatures. This increase could be attributed to the rise in the number of inorganics constantly disintegrated due to an increase in temperature and the alteration in the quantity of other substances in the torrefied samples.

The hemicellulose content in this study is consistent with the reported work of Sixta (2006) and Juliano et al. (2014), where hemicellulose content ranged from 25% to 35%. The variation in hemicellulose content observed in this study may be attributed to differences in the reactor and species used, as suggested by Bourgeois and Guyonnet (1988) and Tooran *et al.* (2014) compared to other studies. Previous research on torrefaction's effect on hemicelluloses has shown that at lower torrefaction temperatures (e.g., 240°C), hemicellulose decomposition results in the formation of char and extractable materials, with volatile compounds such as carbon monoxide, carbon dioxide, water, and various organic acids (Prins *et al.,* 2006). Likewise, the close variation in lignin content across different temperatures suggests that the temperature used in this study did not significantly affect lignin degradation. The lignin results align with the findings of Evandro *et al.* (2015) and Cagnon (2013), where lignin content ranged from 30% to

50%. The cellulose content of this study is consistent with the study conducted by Sundquist (2004), where cellulose content ranged from 11% to 28%. The degradation of cellulose increased progressively with higher temperatures, which is in agreement with other studies using TGA and methanolysis methods (Torran *et al.,* 2014).

CONCLUSION

This research investigated how torrefaction temperature influences the polymer composition of *Leucaena leucocephala* across three significant parts (top, middle and base). This study likewise showed that the composition of carbohydrate polymers, such as hemicelluloses and cellulose, was influenced by temperature and varied across different tree parts. The ash content increased with higher torrefaction temperatures, while lignin content showed less pronounced changes. Cellulose content decreased with increasing temperature. This study revealed that an increase in temperature is a crucial influence on wood polymer in terms of compositional variations in polymers of *Leucaena leucocephala, also* different tree parts because as the torrefied temperature increases, different degrees of breakdown resulted in various quantities of other polymers at varying temperatures. Both species' Extractive and Ash content show a similar trend with temperature. The result revealed that with an increase in temperature, the cellulose content had an upward trend in quantity, especially at above 250°C. In comparison, the lignin content showed less pronounced changes because the lignin degradation starts effectively actively around 280°C; hence no major breakdown in this study will significantly alter the compositional makeup of lignin as observed. Considering the changes observed in the chemical composition in this study for the species used, it becomes imperative to determine the end use of the biomass before torrefaction as this will influence the temperature for pretreatment and eventual properties of the torrefied sample to be produced.

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