

## Thermal Degradation Profile of Chemically Modified Wood Sawdust

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### Abstract

*Gmelina arborea* sawdust was chemically modified using 4% and 8% concentrations of NaOH solution for 30 and 90 minutes soaking times. The influence of this modification on the chemical composition and thermal properties of samples were investigated. Chemical characterization of samples revealed that the percentage content of hemicelluloses decreased from 24.5% in the unmodified sample to 20.8% in samples soaked in 4% NaOH solution for 90 minutes. A further decrease to 17.6% and 15.2% was observed when samples were treated with 8% NaOH for 30 and 90 minutes, respectively. The amount of lignin decreased from 25.0% in unmodified samples to 19.0% when the sample was treated with 8% NaOH for 90 minutes. A moisture content of 9.0% and 2.5% were recorded in unmodified and sample treated with 8% NaOH for 90 minutes, respectively. However, the cellulose content increased from 38.1% in unmodified sample to 60.1% when the sample was treated with 8% NaOH for 90 minutes. The percentage weight loss increased as concentration and modification time increased. Functional groups analysis by Fourier Transform Infrared Spectroscopy (FTIR) showed evidence of reduction in OH and removal of C=O groups associated with the wood polymers after chemical modification. Thermogravimetric analysis revealed that samples treated with 4% NaOH for 90 minutes presented the highest onset and peak degradation temperatures of 274.0°C and 372.1°C, respectively. From these results, it can be concluded that modification with 4% NaOH solution for 90 minutes imparted a significant improvement on the thermal stability of *Gmelina arborea* sawdust.

**Keywords:** Chemical Modification, Fourier Transform Infrared Spectroscopy, Wood Saw Dust, Thermal Degradation, Thermogravimetry

### Introduction

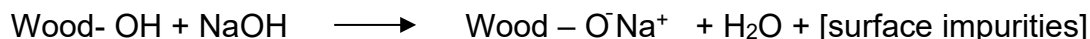
Wood plays vital roles in different industrial sectors, such as in packaging and as attractive reinforcement material in automobiles as well as in building/engineering applications. This is due to its unique qualities such as light weight, renewability, abundance, non-toxicity, low processing energy/cost, less abrasiveness to plastic processing equipment and useful mechanical properties (Fa'bio *et al.*, 2007).

In spite of the several applications of wood in the construction/furniture industries, it has a number of limitations. This includes biodegradability (low microbial resistance), hydrophilicity (high moisture absorption), poor adhesion with hydrophobic polymer matrix, dimensional instability, and thermal instability. Thermal instability results in the restriction of the processing temperature of wood composites to about 200°C since wood polymers undergoes degradation at higher temperature (Maya & Rajesh, 2008; Sultana *et al.*, 2012).

These limitations of wood are due to the presence of hydroxyl groups (OH) in their three main chemical components, namely, cellulose, lignin, and hemicelluloses, which are responsible for the basic chemical and physical properties of wood. Several treatment methods have been employed to alleviate these limitations and enhance better performance (Kallakas *et al.*, 2015; Wu *et al.*, 2016). Chemical treatment is one of the methods of wood modification methods. Generally, chemical modifications of solid wood are intended to improve the material's properties like its dimensional stability, mechanical properties or resistance to biodegradation. However, in the case of wood particles, modifications are aimed at improving the behaviour of the particles or fibres intended for given applications like composites, paper, or new materials (Gérardin, 2016).

Alkalinization by soaking in NaOH is one of the modification methods employed in order to checkmate the limitations of wood (Hakkou *et al.*, 2005; Korkut & Kocaeefe, 2009; Rowell, 2006; Shayesteh & Gregory, 2015; Xiaoli *et al.*, 2015). Reports have shown improved mechanical properties of polymer matrix composites obtained from combining alkaline treated wood saw dust with high density polyethylene (Olanami *et al.*, 2015).

The reaction between wood and NaOH solution is as shown below:



Also, the thermal properties of composites resulting from alkali treated wood composites were reported to improve over the untreated counterparts (Olanami *et al.*, 2015). Mercerization, the alkaline treatment of natural fibres, is one of the common methods of producing high quality fibres for composites production. Alkaline treatment of natural fibres disrupts the hydrogen bonding in the network structure of the wood polymers, thereby increasing the surface roughness (Rout *et al.*, 2016) It reduces fibre diameter, increases aspect ratio, increases the amount of crystalline cellulose, and removes natural and artificial impurities, thereby producing a rough surface topography (Kalia *et al.*, 2009). Consequently, there is an increase in the number of reactive sites by increasing the amount of cellulose exposed on the fibre surface thereby increasing the surface roughness. In addition, it affects the degree of polymerization and molecular orientation of cellulose crystallites due to the removal of the binding substances like lignin and hemicelluloses (Gassan & Bledzki, 1999).

The type of alkali and its concentration influences the swelling reaction of fibres. Na<sup>+</sup> has an encouraging diameter that can easily pierce into the lattice planes of cellulose with a greater chance of widening the structure compared to the other alkalis (Manna *et al.*, 2017). In addition, NaOH treated fibres has been reported to produce composites with better tensile properties than KOH treated counterparts (Reddy *et al.*, 2019). Liu *et al.*,

(2004) studied the effects of alkaline treatment on the structure, morphology, and thermal properties of native grass fibres as reinforcements for polymer matrix composites. The authors soaked chopped grass fibre in 5% and 10% solution of NaOH for between 1 and 16 hours. Results revealed that the amount of lignin and hemicelluloses reduced with alkali treatment and time. It was also observed that the degradation peak temperature of the grass fibre increased with the treatment and duration, indicating an increase in thermal stability. This was attributed to the removal of hemicelluloses with alkaline treatment. The authors concluded that higher concentration of NaOH causes excessive delignification of natural fibres resulting in weaker or damaged fibre. Taking a cue from the above study, higher treatment times and concentrations of NaOH in alkalination of natural fibres destroys its mechanical integrity. Consequently, in this study, lower concentrations, and treatment times than that employed by Liu *et al.* (2004) were adopted. The analysis of the thermal degradation profile of chemically modified *Gmelina arborea* wood sawdust will help to determine its thermal stability. An understanding of the degradation temperatures will enable the determination of the processing temperature when used as fillers in composites production in order to avoid excessive degradation in the processing equipment, which can lead to poor mechanical properties in the resultant composites. Therefore, this study is aimed at modifying *Gmelina arborea* sawdust with NaOH solutions of different concentrations and time, and to study the effect of alkaline treatment on their thermal degradation profile to ascertain its potential for use as fillers in polymer matrix composites (PMC)

## Methodology

### *Sample Collection and Preparation*

The *Gmelina arborea* sawdust was collected from a sawmill in the timber market in the Federal Capital Territory, Abuja Nigeria, during milling operation. The sample was cleaned manually and dried at 105°C until a constant weight was obtained. Finally, the sample was ground and sieved to obtain particles between 40 and 100 mesh sizes (400-150 µm) and kept in a desiccator for further use.

### *Modification with NaOH Solution*

Modification with NaOH solution was carried out in accordance with the method described by Jayabal *et al.*, (2012) with slight modifications. About 2 g wood sawdust was soaked in 100 cm<sup>3</sup> of five different concentrations (2, 4, 6, 8 and 10%) of NaOH for 30, 60 and 90 minutes. The sample was neutralized with 2% H<sub>2</sub>SO<sub>4</sub> to a pH of 7 and washed several times with distilled deionized water. The modified sample was dried in an oven at 105°C until a constant weight was achieved. The extent of modification was calculated as percentage weight loss which was measured as the differences between oven dry weight of the sample before modification ( $W_1$ ) and after modification ( $W_2$ ) using the formula.

Percentage weight loss =  $[(W_1 - W_2) / W_1] \times 100$

### *Chemical Composition of Samples*

Ash content was assessed using ASTM E-1755 standard while moisture content was determined according to ASTM D-4442 standard. Extractives free wood were prepared according to ASTM D-1105 standard. Acid insoluble lignin was determined using ASTM

D-1106 standard. Cellulose and hemicelluloses content were determined according to the method described by Rowell *et al.* (2005).

### FTIR Analysis

FTIR-ATR spectra of samples were recorded using the FTIR spectrophotometer (Spotlight 400, Perkin Elmer, USA), combined with a universal ATR accessory at a resolution of  $4\text{ cm}^{-1}$  for 64 scans in the range of  $650\text{--}4,000\text{ cm}^{-1}$ . Samples were pressed against the diamond crystal of the ATR unit. A pressure applicator with a torque knob ensured that the applied pressure was the same for all measurements.

### Thermal Properties

The thermogravimetric (TG) analysis was carried out on between 5 and 7 mg sample using a Perkin Elmer (USA) STA 2000 thermogravimetric analyzer in the temperature range of between  $50^{\circ}\text{C}$  and  $650^{\circ}\text{C}$  under a nitrogen atmosphere at a flow rate of  $20\text{ cm}^3\text{min}^{-1}$  and a heating rate of  $10^{\circ}\text{C min}^{-1}$ . Continuous records of weight loss versus temperature were obtained and analyzed to determine the onset temperature ( $T_{\text{onset}}$ ), degradation peak temperature ( $T_p$ ), temperature at 50% mass degradation ( $T_{50\%}$ ) and degradation temperature range from the derivative thermogravimetric curve, DTG.

## Results and Discussion

### Weight loss

The results of weight loss of chemically modified samples are presented in Table 1. At 30 minutes modification, the weight loss (in %) was 6.1, 8.5, 10.6, 13.2 and 18.3 for samples modified with 2%, 4%, 6%, 8% and 10% NaOH concentrations, respectively. At 60 minutes modification, the weight loss was 10.2, 13.5, 16.4, 18.3 and 22.1 in samples treated with 2%, 4%, 6%, 8% and 10% NaOH concentrations, respectively. The highest weight loss of 13.11, 18.4, 16.2, 22.4 and 24.0% were observed in the samples soaked for 90 minutes when the concentrations of the NaOH solution was 2%, 4%, 6%, 8% and 10% respectively.

**Table 1: Percentage Weight Loss for NaOH Modified Samples**

Soaking Time (Mins)	2% NaOH	4% NaOH	6% NaOH	8% NaOH	10% NaOH
30	6.1 $\pm$ 0.3	8.5 $\pm$ 0.5	10.6 $\pm$ 0.3	13.2 $\pm$ 0.4	18.3 $\pm$ 0.2
60	10.2 $\pm$ 0.2	13.5 $\pm$ 0.3	16.4 $\pm$ 0.1	18.3 $\pm$ 0.2	22.1 $\pm$ 0.4
90	13.1 $\pm$ 0.4	18.4 $\pm$ 0.2	16.2 $\pm$ 0.3	22.4 $\pm$ 0.1	24.0 $\pm$ 0.2

Results are expressed in mean values,  $\pm$  standard deviations.

This result agrees with the findings of Liu *et al.* (2004), who investigated the effect of NaOH modification on weight loss of native grass fiber and reported an increase in weight loss as modification duration and NaOH concentration increased. The observed increase in percentage weight loss as the concentration of NaOH solution and modification duration increased is an indication of the dissolution of hemicelluloses and lignin. Further increase in NaOH concentration and modification duration could lead to the formation of a weak or damaged sample (Mansour *et al.*, 2011). In addition, treatment with 2% NaOH presents the lowest weight loss, an indication that this concentration of NaOH had a minimal effect on the wood flour. On the other hand, samples immersed in 10% NaOH displayed values that can also be obtained when samples were soaked in 8% solution of NaOH. Samples treated with 6% NaOH did not show any justifiable trend. Therefore, samples treated with 4% and 8% NaOH concentrations at 30 and 90 minutes soaking durations were chosen for further investigations.

#### *Chemical Composition Analysis*

Table 2 presents the percentage composition of unmodified and modified samples. The moisture content decreased with NaOH concentration and soaking time. The highest moisture content of 9.0% was recorded in the unmodified sample, while the lowest (2.5%) was obtained for sample soaked in 8% NaOH solution for 90 minutes. On exposing the samples to NaOH solution, swelling of the sample and formation of hydrophobic ionic groups (Cellulose-O<sup>-</sup>Na<sup>+</sup>) on the surface of the sample occurred. This could lead to the decrease in its moisture absorption capacity, thus leading to the reduction in the observed moisture content of the sample. The decrease could also, possibly, be attributed to the reduction in number of hydroxyl (OH) groups in the wood sawdust components as well as the partial removal of hemicelluloses and lignin by NaOH solution (Akinrinola *et al.*, 2014). Similarly, the unmodified samples present the highest extractives content (3.1%) than the modified samples. The hemicelluloses (24.5%) and lignin (25.0%) contents decreased to 15.2% and 19.0% respectively, after soaking the samples with NaOH solution. This reduction is consistent with literature reports (Vinod *et al.*, 2019). Also, from the results, the amount of ash and cellulose showed an increase after modification. The observed increase in percentage content of cellulose in the modified samples could be because of the partial hydrolysis of hemicelluloses and the depolymerization of lignin leading to the formation of sugar and phenolic groups which are soluble in water (Rajeshkumar *et al.*, 2016). The hydroxyl groups in cellulose were converted to alkoxides (Cellulose-O<sup>-</sup>Na<sup>+</sup>) during modification and to inorganic salt on neutralization with 2% H<sub>2</sub>SO<sub>4</sub>. However, on rinsing with water, the sodium ion (Na<sup>+</sup>) that was linked to cellulose was removed which then led to transformation of native cellulose I to cellulose II with a new crystalline structure, hence reducing hydrophilicity (Mohammed & Kesava, 2014).

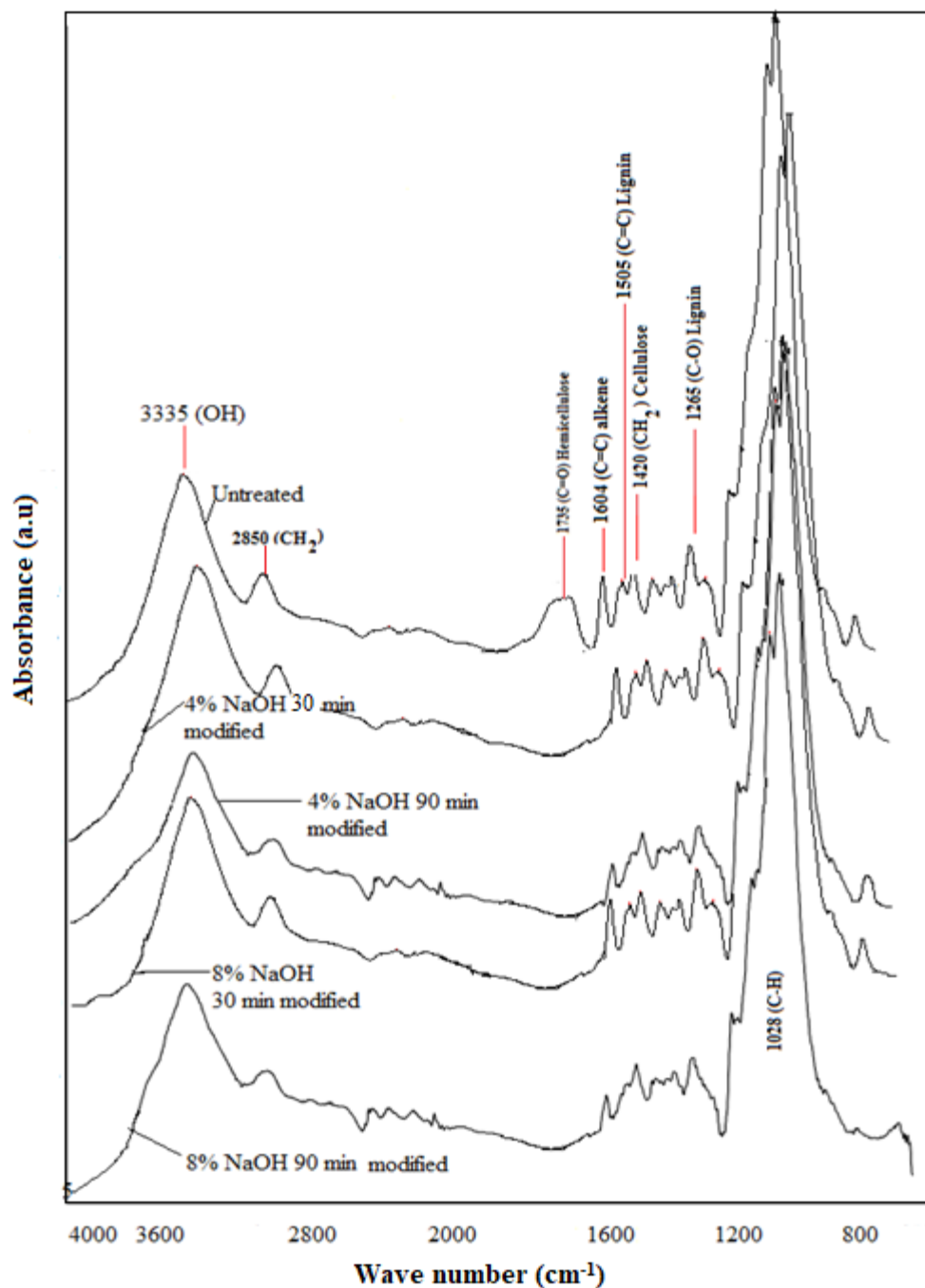
**Table 2: Percentage Composition of Cell Wall Polymers Present in Unmodified and Modified Samples**

Sample	Moisture content (%)	Ash content (%)	Extractives (%)	Hemicelluloses (%)	Cellulose (%)	Lignin (%)
Unmodified	9.0± 0.1	0.4 ± 0.0	3.1 ± 0.2	24.5 ± 0.2	38.1 ± 0.1	25.0 ± 0.3
4% NaOH 30 minutes	6.4± 0.1	2.2 ± 0.1	2.3 ± 0.1	22.9 ± 0.5	42.7 ± 0.6	23.5 ± 1.0
4 % NaOH 90 minutes	5.4± 0.2	2.4 ± 0.0	1.3 ± 0.2	20.8 ± 0.4	49.9 ± 0.1	21.1 ± 0.1
8% NaOH 30 minutes	3.2± 0.1	2.8 ± 0.0	0.9 ± 0.4	17.6 ± 0.2	57.5 ± 0.4	19.2 ± 1.5
8% NaOH 90 minutes	2.5± 0.5	3.1 ± 0.0	0.6 ± 0.1	15.2 ± 0.3	60.1 ± 0.2	19.0 ± 0.2

Results are expressed in mean values, ± standard deviations.

#### *FTIR Analysis*

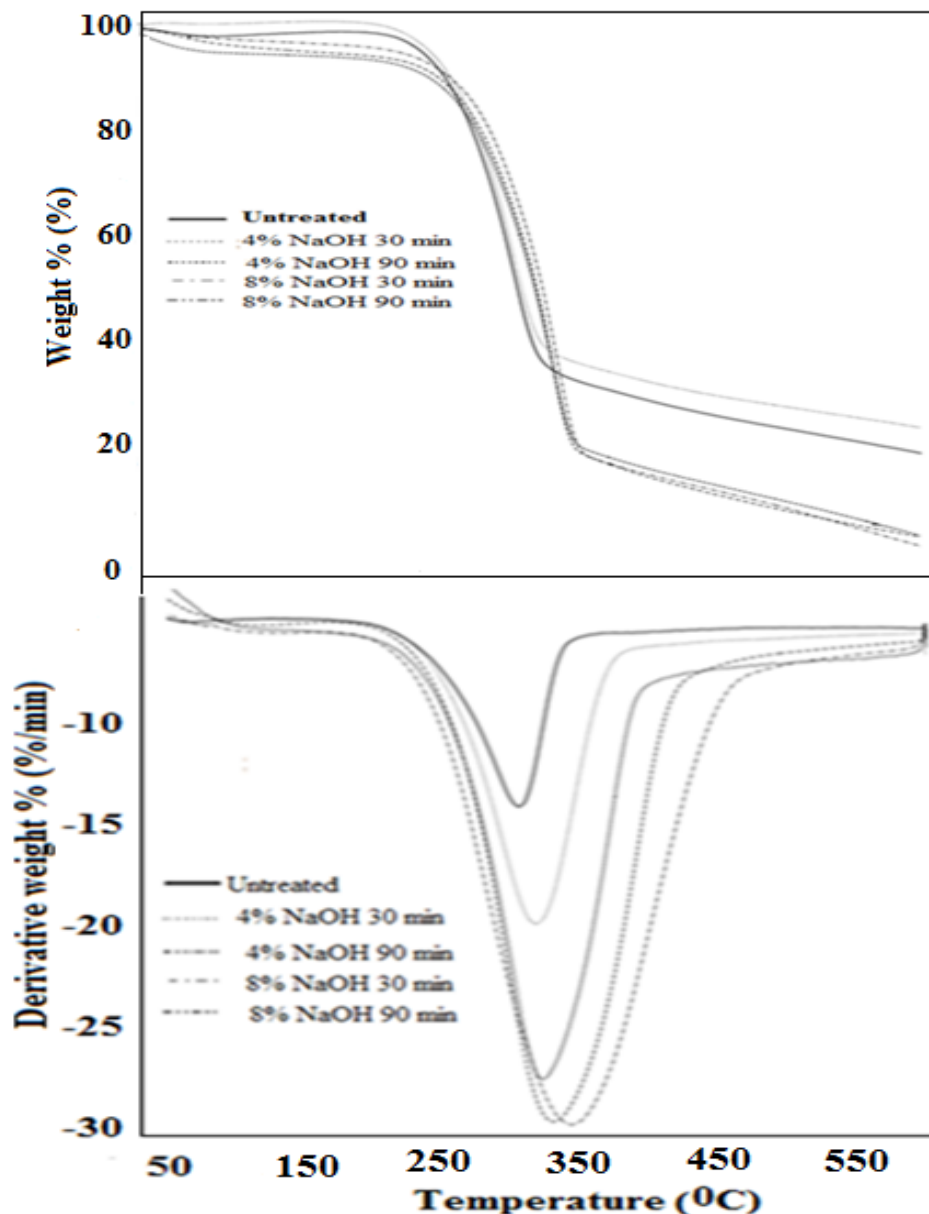
The FTIR spectra of unmodified and modified samples are shown in Figure 1. All the spectra band revealed in the samples are those typical of lignocellulosic materials. The spectra band around 3335 cm<sup>-1</sup> showed a reduction in intensity to 3320 cm<sup>-1</sup> after modification, suggesting a reduction in the hydroxyl groups of the three main wood components. Also, the characteristic band at 1735 cm<sup>-1</sup> disappeared in the modified samples, indicating the removal of carbonyl groups C=O in hemicelluloses. This is in agreement with the findings of Muhammad et. al. (2017). The vibration peak at 1507 cm<sup>-1</sup> assigned to the aromatic C=C in-plane benzene stretching of lignin showed a slight shift to 1505 cm<sup>-1</sup> confirming its partial removal with NaOH solution. The vibration peak at 1265 cm<sup>-1</sup> belonging to C-O stretching vibration of acetyl groups in hemicelluloses and lignin component of the sample showed a remarkable reduction in wave number to 1254 cm<sup>-1</sup> in the modified samples and decreased in intensity. The spectra peak at 1317 cm<sup>-1</sup> assigned to CH<sub>2</sub> wagging vibration in cellulose increased to 1320 cm<sup>-1</sup> in the modified samples confirming the observed increase in the cellulose content. These results indicate that modification with NaOH could lead to a partial removal of hemicelluloses and lignin (Bodîrlău & Teaca, 2007; Deepa et al., 2011; Zhu et al., 2015).



**Figure 1: FTIR Spectra of unmodified and modified Sawdust Samples**

*Effect of Modification on Thermal Characteristics*

The thermogravimetric (TG) and derivative thermogravimetric (DTG) curves of samples are shown in Figure 2. Data extracted from these curves are presented in Table 3. There are two main degradation events as shown in the DTG curves. The first event occurred at between 50°C and 150°C.



**Figure 2: TGA and DTG curves of sawdust samples at  $10^{\circ}\text{Cmin}^{-1}$  heating rate**

This could be attributed to the evaporation of water and, possibly, the volatilization of extractives. The results show that the unmodified sample had the highest percentage weight loss of 1.88% around this temperature region when compared with the modified samples that recorded weight loss as low as 0.132%. The high extractives content in unmodified sample promotes its thermal degradation at lower temperature due to their high volatility and could be responsible for this observation. The second event occurring within the range of  $220^{\circ}\text{C}$  and  $400^{\circ}\text{C}$ , could be attributed to the degradation of hemicelluloses, cellulose, and lignin. This is similar to the reports of Baysal *et. al.*, (2017). It has been observed that lignocellulosic materials, being chemically active, decomposes thermo-chemically in the range of  $150^{\circ}\text{C}$  to  $500^{\circ}\text{C}$ . Hemicelluloses degrades between  $150^{\circ}\text{C}$  to  $350^{\circ}\text{C}$ , cellulose between  $240^{\circ}\text{C}$  to  $350^{\circ}\text{C}$  and lignin between  $250^{\circ}\text{C}$  and  $500^{\circ}\text{C}$



(Yang *et al.*, 2005). This improvement in thermal stability may also be as a result of the fact that hemicelluloses are removed from the wood flour by alkaline treatment. This is expected to have possibly improved the thermal properties by reducing its heat transfer coefficient (Kotalainen *et al.* 1999; Esteves & Pereira, 2008; ).

**Table 3: Thermal Degradation data for unmodified and samples modified with different concentrations of NaOH and time**

Samples	T <sub>onset</sub> (°C)	T <sub>p</sub> (°C)	T <sub>50%</sub> (°C)	Degradation temperature range (°C)	Moisture (%)
Unmodified	230.3	260.2	265.0	220.2-358.1	1.8
4% NaOH 30 min	256.4	337.0	345.3	220.1-401.2	1.6
4% NaOH 90 min	274.0	372.1	361.2	226.2-413.3	0.1
8% NaOH 30 min	267.2	366.1	355.3	226.0-409.2	0.2
8% NaOH 90 min	270.4	367.5	356.2	222.1-412.0	0.3

The high T<sub>p</sub> value for wood flour treated with 4% NaOH solution for 90 minutes is in agreement with the findings of Olakanmi, *et al.*, (2015), who investigated the use of alkaline treated *Daniela oliveri* wood flour as reinforcement in virgin HDPE and reported that wood flour treated with 4% NaOH solution for 150 minutes produced composites that exhibited the highest T<sub>onset</sub> and T<sub>p</sub> of 375°C and 495°C respectively, indicating the highest thermal stability among the samples. Although, samples treated with 8% NaOH for 30 minutes and 90 minutes presented lower values of T<sub>p</sub>, there is no observable trend in the T<sub>p</sub> of the samples. The lower values at this concentration and soaking time may be due to the depolymerization of the native cellulose resulting in excessive delignification of the wood flour treated with NaOH concentrations greater than 4% (Olakanmi, *et al.*, (2015). This indicates that modification with 4% NaOH solution for 90 minutes enhanced the thermal stability of the wood flour used in this study. T<sub>p</sub> in wood degradation profile has also been attributed to the volatilization of the hemicelluloses, cellulose, and lignin (Guida *et al.*, 2019). In general, it has been observed that alkalination of wood flour with NaOH at specified concentration and time has the ability to improve the thermal properties of wood flour (Kelly, *et al.*, 2010; Okoroigwe, 2015). It is therefore necessary to note that samples modified with 4% NaOH for 90 minutes can be conveniently compounded with a polymer matrix at temperatures as high as between 250°C and 300°C.

## Conclusion

The results of this study revealed an increase in weight loss in the samples as the concentration of NaOH and time of soaking increased, indicating the dissolution of hemicelluloses and lignin. The moisture, hemicelluloses, lignin, and extractives content decreased with modification, whereas the cellulose content showed an appreciable increase with concentration and time. FT-IR spectra showed evidence that modification of wood sawdust by NaOH solution had taken place as the absorption peak at 1735 cm<sup>-1</sup> assigned to carbonyl group disappeared after modification, suggesting that a dissolution

of hemicelluloses and lignin in NaOH had occurred. The peak at  $3335\text{ cm}^{-1}$  which corresponds to hydroxyl (OH) molecules present in absorbed water and the three main polymeric constituents in the samples showed a reduction in both intensity and absorbance, a possible proof that the OH groups of the wood polymers had decreased. Thermogravimetric assessment of the samples indicates that the  $T_{\text{onset}}$  and  $T_p$  of the samples are higher in the modified wood flour relative to the unmodified counterparts. This indicates that the degradation of hemicelluloses, the component of the wood polymer with the lowest thermal stability, has led to the overall thermal stability of the modified samples. From the results, it can be concluded that chemical modification with 4% NaOH for 90 minutes has resulted in significant improvement in the thermal properties of *Gmelina arborea* sawdust. This sample can be used as fillers in polymer matrix composites, as it can be processed conveniently at temperatures between  $250^{\circ}\text{C}$  and  $300^{\circ}\text{C}$  without damage to the thermal properties of the wood flour.

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