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MOISTURE SORPTION AND QUANTITATIVE ASSESSMENT OF ACETYLATED AFRICAN WHITEWOOD WITH INFRARED SPECTROSCOPIC METHODS

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ABSTRACT

In this study, moisture absorption and reactivity of acetylated Triplochiton scleroxylon wood and its cell wall components was investigated. Wood samples were treated with acetic anhydride at 120 °C for 60, 120,180, 240 and 300 minutes. The moisture absorption was determined at relative humidity of 65% for eight weeks. The chemical constituents of the wood were determined by wet chemical analysis. The efficiency of the acetylation was assessed by Fourier Transform Infrared (FT-IR) Spectroscopy analysis. The ratio of the intensity of the major peaks at carbonyl (C=O) stretch region (1739 - 1730 cm** $^{-1}$**), carbon-oxygen (C-H) stretch region (1370-1365 cm**$^{-1}$**) and carbon-hydrogen (C-O) stretch region occurring at (1245-1000 cm**$^{-1}$**) of the untreated samples to acetylated ones were determined. The moisture absorption (MA) of the untreated samples after 4 weeks and 8 weeks was 4.25% and 8.24% respectively while the acetylated ranged from 1.57-2.3% and 2.64-3.53% for 4 and 8 weeks respectively. The extracted acetylated and untreated wood components yielded milled wood lignin (6.24-9.8% and 5.40%), hemicelluloses (69.80-73.10% and 67.83%) and celluloses (42.40-44.0% and 42.20%) respectively. The ratio of intensity of IR transmission at the major peaks for acetylated whole wood, holocellulose, hemicellulose and lignin samples were found to be 3.4, 4.9, 1.4 and 4.2 times greater than their untreated counterparts. This suggested that all the wood components were chemically modified, and this is responsible for their significant lower moisture absorption.

Keywords: Acetylation, cell-wall components, spectroscopy, moisture absorption, *Triplochiton scleroxylon*

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INTRODUCTION

In recent times, there has been great concern on the need for the preservation of the environment and sustainability of resource use. In particular, there is a need for humans to reduce their dependence on non-renewable resources and increase the use of renewable resources. Thus, the utilization of forest resources is receiving renewed interest as an alternative to non-renewable resources in material technology. Wood can serve as a substitute for fossil fuels in many applications because it is a renewable and low carbon resource. Many non-renewable materials can be replaced by wood, a stable and durable biomass materials (Domke *et al.*, 2008)

Wood is considered to be a future oriented material, even though, it is one of the oldest and most common structural and building materials.Wood is an economical, renewable, recyclable and biodegradable raw material that requires low processing energy and is aesthetically pleasing (Bruce and Lucy, 2006). It is made up of interconnecting matrix of cellulose (up to 45%), hemicelluloses (20-30 %) and lignin (25-30 %) with minor amounts of inorganic elements and organic extractives. These polymers have free -OH groups that adsorb and release water subject to temperature and relative humidity changes. These changes are responsible for most of the physical and chemical properties exhibited by wood such as changes in dimensional stability and biodegradation by fungi (Hill, 2006; Giridhar, *et al.*, 2017). The formation of hydrogen bonds with water molecules causes are responsible for any chnges in dimensional stability in wood. This occurs in sorption sites which are located in hemicelluloses followed by cellulose and lignin (Engelund *et al*., 2013). Due to configuration of microfibril aggregates in cellulose, the possible sorption sites are on the surface, however in hemicelluloses and lignin, the amount of sorption sites is much greater, thus, increasing adsorption of water molecules at these sites (Engelund *et al.,* 2013).

Water absorption in wood leads to decrease in strength resulting to dimensional changes until the wood reaches the fibre saturation point (FSP). Variations in moisture contents leading to dimensional changes in wood cause internal stresses, checks and other undesirable defects (Čermák *et al.,* 2022). Hence, there is need to protect wood in service especially those used in outdoor conditions from moisture to prevent too much of swelling and shrinking, as well as biological attacks by fungi. To overcome these problems, many different wood modification methods have been used. These methods usually modify the physical properties of the wood by making the wood more hydrophobic devoid of harmful preservative substances (Rowell, 2006) and acetylation is one of such methods that could achieve this. Acetylation is the chemical reaction between acetic anhydride and the hydroxyl groups of the wood polymers, resulting in the esterification of accessible hydroxyl groups in the cell wall (Rowell, 1983). This reaction brings about a change in the chemical constitution of the cell wall thereby affecting most of the physical properties of the wood. There is a decrease in equilibrium moisture content of acetylated wood, a lower maximum cell wall moisture content (Rowell, 2006) also displays less fibres per cross section area compared to it is unmodified due to bulking of the cell wall (Rowell, 1996).

The reduction in equilibrium moisture content of acetylated wood is attached to the presence of acetyl groups covalently bonded in the cell-wall matrix, which therefore does not available for sorption of water molecules (Papadopoulos and Hill, 2003; Popescu *et al*., 2014). Thus, water uptake into the cell wall is reduced, which limits the dimensional changes of wood and enhances its decay resistance. African whitewood (*Triplochiton scleroxylon*) is one of the tropical wood species that is found in most of West Africa countries, especially Nigeria. Obeche wood is desired due to its properties as it is a light wood and this makes it very easy to work with both hand and machine tools (TRADA, 2022). However, it has some drawbacks such as dimensional instability and low resistance to fungi and insects when the moisture content of the wood changes below the fiber saturation point. Thus, it would be of a great value if its properties could be modified. Therefore, the objective of this study was to determine the chemical constituents of the acetylated obeche wood as it affects the rate of moisture absorption.

MATERIALS AND METHODS

Preparation of wood blocks and modification procedures

The wood blocks for this study were obtained at breast height from the 22 years old tree of Obeche (*Triplochiton scleroxylon*) and dimensioned to 20 × 20 × 60 mm (radial × tangential × longitudinal) samples with no defects. Chemical modification of wood using acetic anhydride was performed according to Adebawo *et al*., (2016) and Adebawo *et al*., (2019).

Determination of moisture absorption

The moisture absorption for the treated and untreated wood samples were determined according to the method reported by Islam *et. al*. (2014). The samples were oven dried at 103 ±2°C and then placed in a conditioning chamber at a temperature of 20±2°C and a relative humidity of 65% for six weeks. The weight of each sample was measured and the Equilibrium moisture content (EMC) was calculated as in equation 1.

$$EMC\ (\%) = \frac{M_2 - M_1}{M_1}\ X\ 100\ \ldots\ldots (1)$$

Where,

EMC = Equilibrium moisture content

M_2 = weight of the wood samples at moisture absorption equilibrium

M_1 = oven-dry weight of the wood samples

Determination of chemical constituents by wet chemical analysis

The chemical constituents of acetylated and unmodified wood of *T. scleroxylon* were quantitatively determined by wet chemical analysis.

Extractives free wood preparation

The extractives in the wood species were removed with acetone using Soxhlet apparatus. Wood for chemical analysis were ground to a fine particle size with a Willey mill to a fine particle size to pass a 0.4 mm (40-mesh) in order to permit complete reaction of the wood in the reagents used in the analysis according to TAPPI-264 (1997).

Determination of Acid Insoluble Lignin (Klason lignin) and Milled wood Lignin (MWL)

Acid insoluble lignin percentage was calculated from the extractive free wood, according to TAPPI Standard (TAPPI- T222, 2002). Milled wood lignin was determined using the method of Bjork-man (Lin and Dence, 1992). The extractive-free samples (1.0 g) were ball-milled (Microwolf-VFD-A, Torrey Hill Technology, LLC, USA) in a zirconium (zirconium dioxide, 95 %) grinding bowls (5 cm diameter, 4 cm height) in the presence of 8 zirconium balls (10 mm diameter). The milling process was conducted at room temperature at 350 rpm for 40 min with 20 min cooling intervals for a total of 72 h. The 72-h milled wood sample was dispersed in dioxane: water (96:4, v/v) and mechanically stirred (1 g of milled wood in 25 ml of dioxane: water) for 48 h. The extract was then dried in a rotary vacuum evaporator to give a crude MWL.

Extraction of Holocellulose

The acid chlorite method was carried out according to procedure used by Rowell, *et. al*, (1994) as adopted by Sadeghifar *et al.,* (2014). The mixture was centrifuged and holocellulose was collected as the residue, washed with distilled water, air-dried overnight, and then vacuum oven-dried at 40 ℃ overnight.

Extraction of Hemicellulose with Sodium hydroxide (NaOH) (Rowell et. al., 1994)

A weight of 1 g of air-dried sample was stirred in 50 ml of a 12 % NaOH solution, and nitrogen gas was bubbled through for 1 min. The flask was stoppered and stirred gently for 24 h at 24℃. The sample was then filtered by suction over a coarse glass filter. The residue was then extracted three times with 50 ml of 7.1 % NaOH and the two extracts were mixed, and the solution was neutralized with hydrogen (0.5N) chloride and evaporated by rotary evaporator. The extract was then dissolved in 100 ml of deionized water. The solution was adjusted to pH 4 with acetic acid and the hemicellulose was precipitated with ethanol and hemicellulose was collected by centrifugation. The sample was finally dried in a vacuum oven at 40 ℃, a fine, light-yellow powder was obtained.

Extraction of Hemicelullose with Dimethyl sulfoxide (DMSO)

The hemicellulose was extracted from the holocellulose (acetylated and unmodified) using organic solvent extraction methods due to the hydrolytic instability of acetyl groups under alkaline conditions. (Teleman *et al.,* 2003; Goncalves *et al.,* 2008; Sadeghifar *et al.,* 2014). A weight of 1 g of pulverized holocellulose was mixed with 50 mL of DMSO at 45 ℃ over 24 h.

The cellulose content was determined by subtracting the hemicellulose fraction from the holocellulose content.

Fourier Transform Infrared (FT-IR)

The infrared spectra of acetylated and unmodified woods and its components were determined using an ATR with Perkin Elmer FT-IR spectrometer Frontier by accumulation of 64 scans at a resolution of 4 cm^{-1} at 600 - 4000 cm^{-1} as described in our previous reports (Adebawo *et al.,* 2019).

RESULTS

Moisture Absorption

The moisture absorption was measured and the mean values for the moisture absorption of untreated and acetylated Obeche wood specimens after 4 and 8 weeks are presented in Table 1. The untreated wood samples displayed a higher percentage of moisture absorption (4.25 and 8.21 %) after 4 and 8 weeks respectively than the acetylated wood samples. The moisture absorption for acetylated samples ranged from 1.57- 2.30 and 2.64- 3.53 % for a period of 4 and 8 weeks respectively, while the least moisture absorption was observed in the 240- and 300-min acetylated samples. The analysis of variance results depicted that there was significant difference in the moisture absorption of untreated and acetylated samples for the period of 4 and 8 weeks. However, the results revealed the influence of acetylation time on moisture absorption in which there was no significant different in the moisture absorption of 60-, 120- and 240-min acetylated samples exposed for 4 weeks and also no significant difference observed among 180-, 240- and 300-min acetylated samples. For the 8 weeks exposure, follow-up test showed that there was no significant difference between 60- and 120-min acetylated samples, and also no significant difference was seen among 120-, 180-, 240- and 300-min acetylated samples.

Table 1. Influence of acetylation time on moisture absorption (MA) of untreated and acetylated wood of *Triplochiton scleroxylon* wood samples

Acetylation time (Min)	MA after 4 weeks	MA after 8 weeks
Untreated	4.25 ± 0.40^a	8.24 ± 0.39^a
60	2.30 ± 0.46^b	3.53 ± 0.64^b
120	1.86 ± 0.48^c	3.15 ± 0.47^b
180	2.03 ± 0.51^b	3.06 ± 0.47^b
240	1.67 ± 0.67^c	2.83 ± 0.34^c
300	1.57 ± 0.31^c	2.64 ± 0.30^c

Means followed by the same letter in the same column are not significantly different (p>0.05)
**MA-moisture absorption*

Wet Chemical Analysis

The chemical composition of untreated and acetylated samples is presented in Table 2. The untreated wood sample showed lower klason lignin (28.8%) than the acetylated ones (29 - 31.19%). The data showed that the yield of extracted milled wood lignin and hemicellulose were improved in the acetylated samples. The yields of milled wood lignin in the unmodified samples was 18.75% of the determined Klason lignin for unmodified wood samples. However, for the acetylated wood samples at 60, 120, 180, 240 and 300 min, the yields were increased to 21.5%, 26.2%, 28%, 28.4% and 31.4%, respectively. The major changes were noted for hemicellulose extracted with DMSO. The yield of hemicellulose (DMSO extraction) for non-acetylated samples from milled holocellulose was 48.6% (based on the total hemicellulose extracted by NaOH). After acetylation, the hemicellulose extraction yields were increased to 59%, 67%, 72.7%, 72.7% and 74.5% in 60, 120, 180, 240 min treated samples, respectively. The holocellulose content increment was between 2-7% for the acetylated samples. There was no much changes for the cellulose content for the acetylated samples, however, it increased for about 4% for 300 min acetylated samples.

Table 2. Chemical composition of untreated and acetylated wood samples based on extractives-free materials (triplicate analyses).

Wood components	Amount in wood %(w/w)					
	Reaction time (min)					
	untreated	60	120	180	240	300
Klason Lignin	28.80	28.92	29.44	30.56	30.68	31.19
Milled wood Lignin	5.40	6.23	7.72	8.56	8.71	9.80
Holocellulose	67.83	69.80	70.50	72.15	72.40	73.10
Total Hemicellulose	25.63	27.40	27.60	28.75	28.70	29.10
Extracted Hemicellulose with DMSO	12.46	16.30	18.56	20.89	20.86	21.68
Cellulose	42.20	42.40	42.90	43.40	43.70	44
Extractives	1.54	1.65	1.44	1.55	1.68	1.66

**Dimethyl sulfoxide - DMSO*

Intensity of major peaks of Fourier Transform Infrared (FT-IR) Analysis

The FT-IR spectra of untreated and acetylated wholewood, holocellulose, and MWL of *Triplochiton scleroxylon* has been reported in our previous work (Adebawo et al., 2019). The normalized transmission of the carbonyl band stretch frequency for all samples including the ratio of the intensity between untreated to acetylated wood samples and wood components were also calculated. The ratio of the intensity of the major peaks at carbonyl (C=O) stretch region (1739 - 1730 cm^{-1}), carbon-oxygen (C-H) stretch region (1370-1365 cm^{-1}) and carbon-hydrogen (C-O) stretch region occurring at (1245-1000 cm^{-1}) of the untreated samples to acetylated ones were determined (Figures 1 – 3).

The ratio of the IR transmission of the carbonyl group (C=O) at 1739-1730 cm^{-1} for the untreated and acetylated whole wood, holocellulose, hemicellulose and lignin sample are presented in Figure 1. The transmissions at this peak for the acetylated whole wood, holocellulose, hemicellulose and lignin samples were found to be 3.4, 4.9, 1.4 and 4.2 times higher than the untreated samples respectively. Moreover, the IR transmissions of acetylated whole wood, holocellulose, hemicellulose and lignin samples at frequencies occurring at 1245-1000 cm^{-1} (C – O) presented in Figure 3 were found to be 2.4, 2.0, 2.6 and 2.3 times higher than the untreated samples respectively.

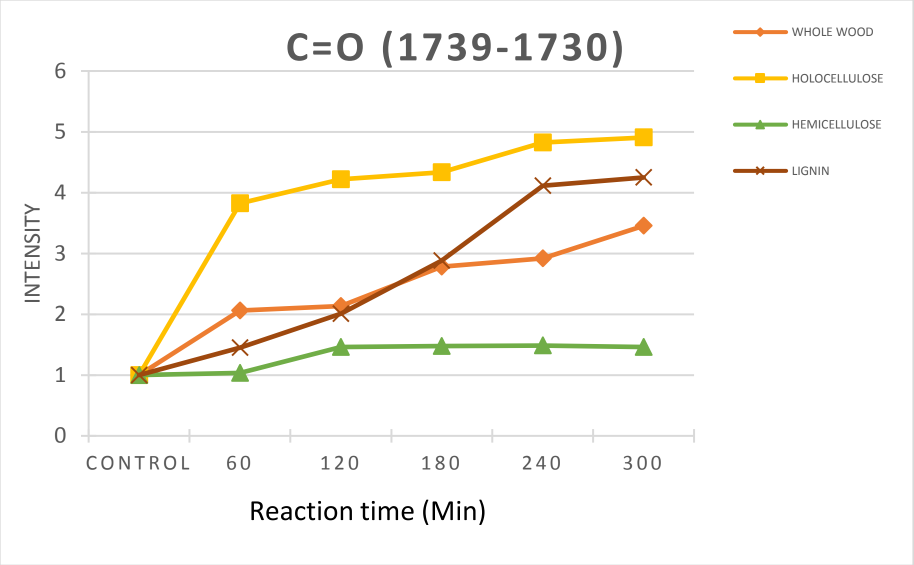

Figure 1: Intensities of IR transmission for the C=O bond in the *T. scleroxylon* wood and its components at varying reaction time

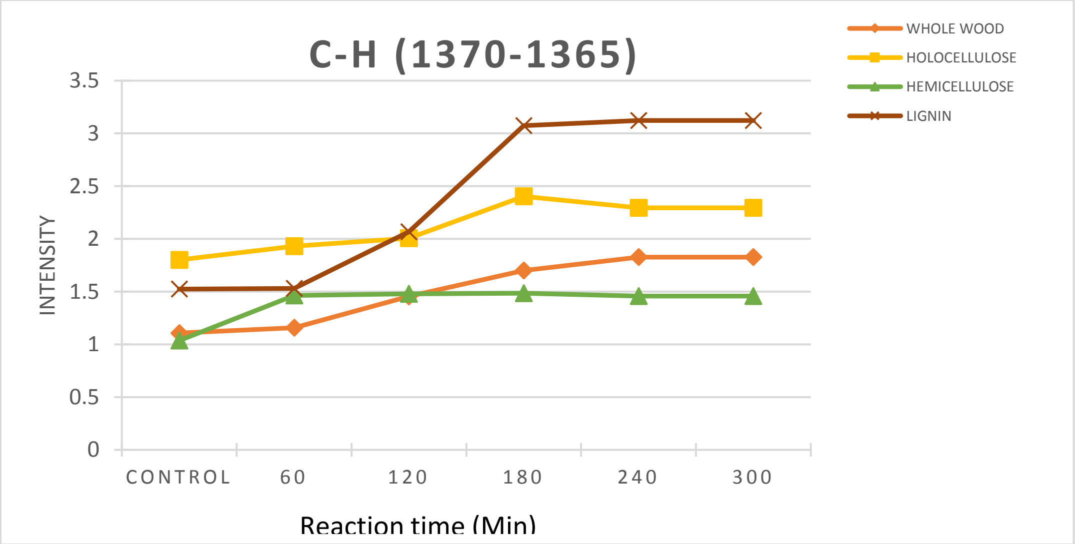

Figure 2: Intensities of IR transmission for the C-H bond in the *T. scleroxylon* wood and its components at varying reaction time

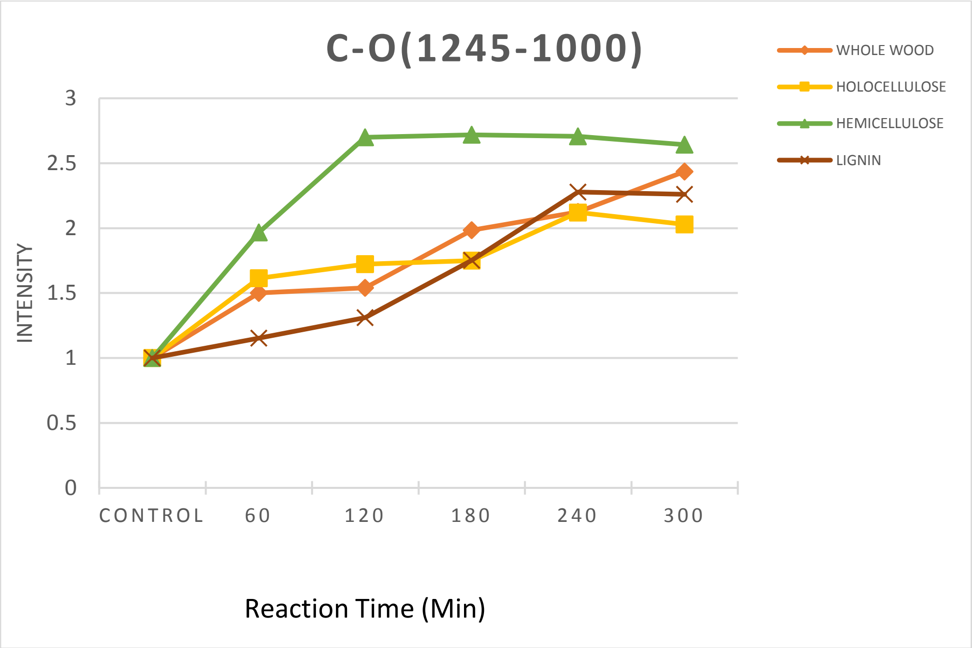

Figure 3: Intensities of IR transmission for the C-H bond in the *T. scleroxylon* wood and its components at varying reaction time

DISCUSSION

Moisture Absorption

The untreated samples displayed a higher percentage of moisture absorption than the acetylated wood samples. This was expected based on the mechanism that hydrophilic hydroxyl groups will absorb moisture to their surfaces through the formation of hydrogen bonds (Rozman *et al.*, 1998; Islam *et al.*, 2014). The replacements of the hydroxyl groups with acetyl groups in the acetylated samples significantly reduced moisture absorption compared to the untreated samples. Acetylated wood has reduced permeability to gases compared with untreated wood (Kumar *et al.*, 1979). This is thought to be due to the bulking chemical restricting pore space. This reduction in permeability also reduces the moisture absorption as well as overall water resistance.

Wet Chemical Analysis

Wood is essentially composed of cellulose, hemicelluloses, lignin, and extractives**.** Analysis of wood chemical components of untreated and acetylated wood revealed the extent of modification in each of this component. The results indicated that the chemical constituents of wood were changed after chemical modification and are dependent on reaction time. The increase in klason lignin and holocellulose at higher reaction time suggested that this component reacted well during acetylation. Since reactivity of cellulose was very low, this suggested that hemicellulose part of the holocellulose gave the higher percentage. These observations agree with the research findings of Rowel *et al.*, (1994) and Sadeghifar *et al.*, (2014) who showed that chemical transformation and extraction of wood components increased during acetylation. Apparently, acetylation of wood facilitates the lignin and holocellulose constituents' extraction from wood.

Intensity of major peaks of Fourier Transform Infrared (FT-IR) Analysis

The IR transmission for the C=O bond in the holocellulose of the acetylated samples increased sharply after 60 min and and after 240 min, it was greater (4.9 times greater) than the whole wood and other wood components and no visible changes occurred till the end of the reaction. This was followed by lignin which showed a progressive increase in intensity with increase in reaction time.The whole wood also showed an increase in the intensity as the reaction time increases, this was just 3 times greater even after 300 min of reaction. The least reacted was hemicellulose, there was no change in the intensity until after 120 min and this remained unchanged till the end of the reaction time. A continuous increase in the intensities of specific bands (C=O) at 1739-1730 cm^{-1} observed was due to an increase in the weight percent gain (WPG) and the increasing number of acetyl groups resulting in acetylation of wood (Adebawo et al., 2016). The dissapearance of this band which remained unchanged till the end of the reaction time confirmed the loss of hemicellulose. The disappearance of the C=O bond at the peak in the region of 1740 cm-^{1}, is attributed to the loss of hemicelluloses and this is in line with the report of Cetin and Ozmen (2011).

The intensities of the IR transmissions occurring at 1370-1365 cm^{-1} (C – H) in acetylated whole wood, holocellulose, hemicellulose and lignin samples presented in Figure 2 were found to be 1.8, 2.3, 1.4 and 3.1 times higher than their untreated samples, respectively. The IR transmission for the C – H bond intensities showed that lignin was the most reactive, even though the intensity remain unchanged until after 60 min of reaction. After 180 min, the intensity was 3 times graeater and remain unchanged till the end of reaction. Holocellulose was next, there was an increase in the intensity till the reaction time reached 180 min and the intensity was 2.3 times greater. The whole wood and hemicellulose showed gradual increase till 60 min reaction time and remained unchaged till the end of the reaction.

These bands are assigned to aliphatic C–H deformation vibrations of the CH_3 group (1374cm^1), from lignin, which is overlapped by the latter one with increasing number of acetyl groups (Manfred et al., 2013). Since this band is related to lignin, the band intensity for lignin was greater than other wood constituents as shown in figure 2.

The IR transmission for the C–O bond intensities revealed that hemicellulose had the highest intensity and increased sharply from the beginning of the reaction till 120 min reaction time with an intensity that was 2.6 times greater but remained unchanged after this time till the end of the reaction time. The holocellulose also increased till 240 min reaction time with intensity of 2.2 times greater and dropped to about 2 after this time. The whole wood and lignin intensities also increased, however the intensities were just 2.4 and 2.3 times greater respectively. The (C ─ O) stretch at 1245–1000 cm^{-1} due to the carbonyl deformation in the ester bond in ligin and xylan in acetylated wood and its components is responsible for the high intensities at this peak for the hemicelluloses (Adebawo *et al.*, 2019; Blanco and Alfaro, 2014).

CONCLUSION

There was a significant moisture reduction in the acetylated *Triplochiton scleroxylon* wood compared to their untreated counterparts. The isolation of the wood constituents was greater in the acetylated samples than in untreated samples. The pronounced increase in the intensities of all the peaks related with acetylation for the whole wood, holocellulose, hemicelluloses, and lignin indicated that all the wood components were acetylated.

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