



ISSN: 0976-3376

Available Online at <http://www.journalajst.com>

ASIAN JOURNAL OF
SCIENCE AND TECHNOLOGY

Asian Journal of Science and Technology
Vol. 11, Issue, 01, pp.10692-10699, January, 2020

RESEARCH ARTICLE

THE EFFECTIVENESS OF OIL-HEAT TREATMENT IN THE MAIN CHEMICAL CONSTITUTES OF PLANTED 15-YEAR-OLD *ACACIA HYBRID*

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ARTICLE INFO

Article History:

Received 25th October, 2019

Received in revised form

29th November, 2019

Accepted 17th December, 2019

Published online 31st January, 2020

Key words:

Planted *Acacia Hybrid*, Oil-Heat Treatment Process, Holocellulose, Hemicelluloses, Cellulose, Lignin Contents Changes.

ABSTRACT

The hybrids were natural regeneration and were progenies of the two *Acacia* species that had been planted near each other. The *Acacia hybrid* was chosen because it exhibits better properties (It is less susceptible to heart rot disease) in comparison with *A. mangium*. Moreover, *A. hybrid* also widely planted in most countries as sources to replace natural timber. The study focused on variations in the chemical constituents of oil-heat treated planted 15 years-old *Acacia hybrid* investigated. The logs of planted *A. hybrid* were harvested, segregated and cross-cut into the bottom, middle and top portions. The portions then process into respective sizes, and oil-heat treated using organic palm oil at temperature of 180, 200 and 220°C for the time 30, 60 and 90 minutes respectively. The samples were dried and milled into sawdust and air-dried before undergoing subsequent chemical analysis — the untreated woods used as controls. Standard outlined by the Technical Association of the Pulp and Paper Industry (TAPPI) followed. The results highlighted that the chemical composition of the *Acacia* woods changes during the oil-treatment process from 180 to 220°C and occurred at both the sapwood and heartwood. The holocellulose and cellulose decreased from 71.5 to 63.1% and 47.1 to 37.7%, respectively for sapwood while from 73.4 to 64.0% and 48.9 to 38.1%, respectively for heartwood. The hemicellulose content increases from 24.4 to 25.4% in the sapwood and 24.5 to 25.9% for the heartwood. Nevertheless, lignin contents increased 20.8 to 24.0% for the sapwood and 22.4 to 24.9% in the heartwood for treatment temperature from 180 to 220°C.

Citation: Razak Wahab, Mohamad Saiful Sulaiman, Hashim W. Samsi, Ros Syazmini Mohd Ghani and Nasihah Mokhtar, 2020. "The Effectiveness of Oil-Heat Treatment in the Main Chemical Constitutes of Planted 15-Year-Old *Acacia Hybrid*", *Asian Journal of Science and Technology*, 11, (01), 10692-10699.

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INTRODUCTION

The wood industry is currently facing the declining supply of large logs from the natural forest. The industry has no choice but to turn to the smaller diameters logs from the forest's plantation. It is an alternative way to patch the industries demand since plantation forestry rotations are significantly shorter than in natural stands. This situation has become worse, as consumer countries in Europe and North America are sensitive about logging activities from naturally grown species from the natural tropical rain forest. The effort to establish the plantation sector becoming a more concern by government and private sector to cover the demand required from timber industries, in the same way, to conserve the natural forest from continually being vanished by logging activities. *Acacia* species was chosen as one of the pioneer species for the forest plantation project in Malaysian.

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The forest plantation project started in 1982 aims in providing a sustainable supply of timber for the industries which were expected to face a shortfall from the natural forests (Ahmad, 1995). *Acacia* was chosen as a pioneer species due to its rapid growth, and ease adaptation to the local soil (Pinyopusarerk et al., 1993). *Acacia* species such as *Acacia mangium*, *A. auriculiformis* and *A. hybrid* were major fast-growing plantation species not only for timber production but also for greening purposes in the tropical region (Wahab, 2019; Yamamoto et al., 2003; Semsuntud et al., 1991; Hamimi et al., 1989). Furthermore, the wood properties from these planted species tend to be more uniform and of high quality than those harvested from the wild (Wahab, 2019). *Acacia* is categorized as a slightly durable species, especially for exposed condition and in ground contact uses. The preservative treatment has to be used to prolong the utilisation of wood products for specific periods. However, the wood treatment used preservative, which mostly has heavy metals and discharge toxin to the environment. A lot of efforts have been put to develop new wood preservatives.

Besides, developed countries have banned the use of Chromecopper Arsenic (CCA) in their woody materials (Wahab *et al.*, 2017; Berard *et al.*, 2006). Increased in environmental awareness by the general public, combined with an increasingly stringent legislature in recent years has led to more significant restrictions on the use and disposal of many of the conventional which support the use of renewable resources and environment-friendly chemicals have resulted in high interest in "non-biocidal". Another environment-friendly technique was the use of biodegradable substances in wood protection (Wahab *et al.*, 2017; Hyvonen *et al.*, 2006). One of the new, environmentally friendly techniques used in enhancing the durability of wood is the heat treatment process. Heat treatment seemed a suitable treatment for woods because of its advantage of being non-toxin and does not require a chemical application. This study aimed to investigate the changes that occurred in the main chemical components of oil-heat treated cultivated *A. hybrid*. The chemical constituents are parameters that influenced the durability and stability of the wood. The results of this study will benefit immensely in terms of improving the technologies in treated wood for the local wood industry in producing good quality of Acacia products.

MATERIALS AND METHODS

Research Preparation: Three (3) defects free 15-year-old *A. hybrid* logs harvested from GP Pusaka plantation in Bintulu, Sarawak.

Materials

The logs were selected based on their excellent standing condition, long straight bole, decay-free and minimum branches. The average diameters of the trees chosen at breast height were 25 to 30 cm. A chain saw used for harvesting the trees and woodblocks of 60 cm long were cut from the base, middle and top of each tree corresponding respectively to 50, 30 and 20% of the merchantable height. The woodblocks then transported to UCTS and FRIM for further processing and subsequent testing.

Sample Preparation

The woodblocks were air-dried under the shed for about a month to reduce the moisture to equivalent moisture content of 15% and to remove stresses in them. After drying, the woodblocks split in the middle by using a chainsaw and planned into sizes of 300 mm x 100mm x 25mm (length x width x thickness) with a table saw. These samples comprise of two set of samples consisting of sapwood and heartwood. The wood samples were then oil-heat treated using oil palm oil as the heating medium. Untreated samples used as the control for comparison purposes.

Oil-Heat Treatment Process: The oil-heat treatment was performed in a stainless steel tank, complete with thermocouples and an electric heat generator. The tank size was 100 cm 40 cm x 40 cm (length x width x height) in 3 mm thickness. The heat of the tank generated through electrical power sources. Three replicates used for each treatment condition of temperature and time. Before the treatment, samples were stabilised to 12% moisture content (MC) in a conditional chamber set at 65% RH and temperature 25°C.

Altogether 81 samples were prepared before the treatment. Coding made by using a marker pen on each surface of the samples. The weight of the samples was taken before and after the treatment to determine the weight loss caused by the treatment. Palm oil was used as the heating medium in this study. This oil was chosen because it is organic and has a high boiling point of 320°C (Rafidah, 2009; 2008; Wahab *et al.*, 2005). The tank was filled in with the oil until it reached three-quarters full. Treatment temperature at 180°C, 200°C and 220°C applied at the time of 30, 60 and 90 minutes. Wood samples placed into the hot oil at 80°C. After each treatment period, the samples were removed out from the tank. The samples were then wiped with the clean cloth to avoid excessive oil from inserted the wood tissues. The samples were cooled down. The wood samples then conditioned in a conditioning chamber at 20±2°C temperature and 65±5% relative humidity to obtain 12% MC. The samples then cut into respective sizes following testing procedures.

Chemical Analysis: The evaluations of wood chemical in treated samples were conducted using the alcohol-toluene solubility, holocellulose, alpha-cellulose and klason lignin procedures. All chemical analysis tests conducted separately for heartwood and sapwood. The total amounts of the wood chemical were calculated based on the sapwood or heartwood ratios. Evaluation of chemical analysis was performed using TAPPI T203 cm-99 (1999) and TAPPI T222 OM-01 (2002) standard.

Sample Preparation: Wood samples divided into the heartwood and sapwood for study on the differences in chemical composition. The wood chipped for grinding purposes. Samples were ground into powders with Willey's mill to pass BS 40-mesh sieve and retained on a BS 60-mesh sieved. Then the samples were dried in for 7 days until the MC is in equilibrium with the surrounding before they chemically analysed. A small weighing bottle previously cleaned and dried in an oven was weighed on an analytical balance to the nearest 0.01 g. 2 g air-dried sawdust was placed in the weighing bottle and reweighed to the nearest 0.01 g. The sample was then dried in an oven at 105±2°C for 3 hours with the cover off. Then the bottle was removed and placed in desiccators for 15 minutes to let it cool before weighing it. The moisture content of the sawdust was determined.

Alcohol-toluene Solubility: The alcohol-toluene solubility test was the initial test for many of the chemical analyses. Both lignin and holocellulose content tests performed with extractive free wood that derived from the alcohol-toluene extractive test. Extraction of the holocellulose is a necessary preparatory stage to determine the alpha-cellulose content. The extraction apparatus consisted of a soxhlet extraction tube connected on the top end of a reflux condenser and joined at the bottom to a boiling flask. Approximately, 2 g of air-dried sawdust weighed to an accuracy of 0.01 g in a weighing tibble. The tibble placed in a soxhlet extraction tube. The clean dry round bottle flasks (250 ml) contained a 2:1 solution of 95% ethyl alcohol and toluene, respectively placed on a heating mantle. The extraction of 230ml alcohol-toluene solution conducted for 8h at the rate of approximately six siphoning per hour. When extraction completed, all remaining solutions in a round bottle flask was evaporated with a rotary evaporator over a water bath at 40°C.

The round bottle flasks were oven-dried at 105+2°C, cooled in desiccators and weighed until a constant weighed obtained. The alcohol-toluene solubility content of *Acacia* wood was obtained using Equation 1.

$$\text{Alcohol – toluene soluble (\%)} = \frac{W2}{W1} \times 100$$

Where, W1= weight of oven-dried sawdust, (g), W2= weight of oven-dried extraction residue, (g).

Determination of Holocellulose: Approximately, 2 g of air-dried extractive free sawdust was weighed to the nearest 0.01 g and transferred into a 250 ml beaker. The sawdust was then added and stirred with 100ml distilled water, 1.5 g sodium chlorite and 5 ml of 10% acetic acid on a water bath maintained at 70°C. The solvent constantly swirled for over every 5 minutes. The flask was kept closed with a small round glass through at the heating period. The heating was carried out in a fume hood. Five ml of 10% acetic acid added after 30 minutes of heat. 1.5 g sodium chloride added after the second 30 minutes. This procedure repeated until 6 g sodium chlorite and 20 ml of 10% acetic acid added. At the end of heating, the beakers were placed in an ice-water bath until the temperature of the beaker reduced to 10°C. The contents of the beaker were filtered into a coarse fritted glass crucible (porosity 1) of known weight. The residue washed with iced distilled water and acetone. The residue changed colour from yellow to white. The crucible was then air-dried at the surrounding area until a constant weight was reached.

The holocellulose content in *Acacia* wood was determined using Equation 2.

$$\text{Holocellulose Content (\%)} = \frac{Y2}{Y1} \times 100$$

Where, Y1 = Weight of air-dried holocellulose, (g), Y2 = Weight of oven-dried extraction residue, (g).

DETERMINATION OF CELLULOSE: According to Sulaiman *et al.*, 2016, approximately, 3 g oven-dried sample of holocellulose was placed into a 250 ml beaker. The beakers placed on a stirrer magnetic. 15 ml of 17.5% NaOH was added and stirred for 1 minute. 10 ml more 17.5% NaOH was added and the solution mixed for 45 seconds. Then, 10 ml of 17.5% NaOH more was added and swirled for 15 seconds. The solution allowed for 3 minutes (total time was 5 minutes). The solution added again with 10 ml of 17.5% NaOH and swirled for 2 and a half minutes. This procedure was repeated for 3 times (total time was 15 min.). The solution was allowed to react with the solution for 30 min. After added and stirred with 100 ml distilled water the solution was allowed to react for 30 min. and the total time was 75 min. The contents of the beaker filtered with the aid of vacuum suction through a fritted-glass crucible (porosity 3) of known weight. The residue was rinsed first with 25 ml of 8.3% NaOH, and then with 650 ml distilled water at 20°C. The filter stopped and 2N acetic acid was filled in the crucible to allow the residue soak for 5 min. The suction was reapplied to remove acetic acid. The residue was rinsed with the distilled water until it free of acid as indicated by the litmus paper. The bottom and the side of the crucible were wiped out with a dry towel and the crucible oven-dried in an

oven at 105+2°C, then cooled in a desiccator and weighed until constant weighed reached.

Equation 3 was used to obtain the alpha-cellulose content in *Acacia* wood:

$$\text{Oven – dried alpha – cellulose sample (\%)} = \frac{Z2}{Z1} \times 100$$

Where, Z1 = Weight of oven-dried sample, (g), Z2 = Weight of oven-dried alpha-cellulose, (g).

Determination of Lignin: Determination of lignin content referred from TAPPI Standard T 222 om-02 and Sulaiman *et al.*, 2019. 72% sulfuric acid prepared by pouring 665 ml of concentrated sulfuric acid into 300 ml of water. After cooling, another 700 ml of water added to make a 1000 ml solution. The acid solution cooled in a refrigerator to 10 - 15°C before being used. Approximately, 1 g of air-dried extractive free sawdust was weighed out accurately in weighing bottle and transferred in a 50 ml beaker. 15 ml of 72% sulphuric acid was added carefully with a pipette and the mixture stirred with a small glass rod. The mixture was left quantitatively with a wash bottle (water) to a 500 ml round-bottle flask and diluted with water until the final volume is 300 ml. The solution was refluxing (boiled under reflux for 2 hrs.), a crucible was oven dried for 1 hr. at 110°C, then allowed to cool in desiccators (15 min.) and accurately weighed. When the refluxing completed, the insoluble lignin was recovered by filtration through the crucible after allowing the lignin to settle to facilitate filtration. The lignin-free washed with 250 ml of hot distilled water. The lignin-containing the lignin was dried at 110°C for 1 hr., cooled in desiccators (15 min.) and weighed.

Equation 3 was used to obtain the lignin content in the *A. hybrid*:

$$\begin{aligned} \text{Lignin content in } Acacia \text{ wood (\%)} \\ = \frac{(W4 - W3)}{(100 \times W2)} \times (100 - W1) \end{aligned}$$

Where, W1 = Alcohol-toluene extractive content, (%) W2 = Weight of oven-dried extractive free sample, (g), W3 = Weight of oven-dried crucible, (g), W4 = Weight of oven-dried residue and crucible, (g).

RESULTS AND DISCUSSION

Table 1 and Figure 1 showed the chemical composition of the treated *Acacia* wood through the oil-heat treatment process. For sapwood and heartwood, the results clearly showed that the changes of chemical components occurred when wood was treated at different temperature and time (Table 1). The changes in the wood chemical components were due to the loss of polysaccharide material which becomes particularly significant at a temperature above 180°C; however, it depends on the treatment condition (Hill, 2006). For treated sapwood, holocelluloses contents varied between 63.1% and 70.8% while hemicelluloses contents varied between 23% and 26.1% with cellulose between 37.7% and 46.2%, and lignin between 19.2% and 24.9% at 180-220°C with 30 to 90-minute treatments. On the other hand, holocelluloses contents for treated heartwood varied between 64% and 71.7%, hemicelluloses contents varied between 23.2% and 26.2% with

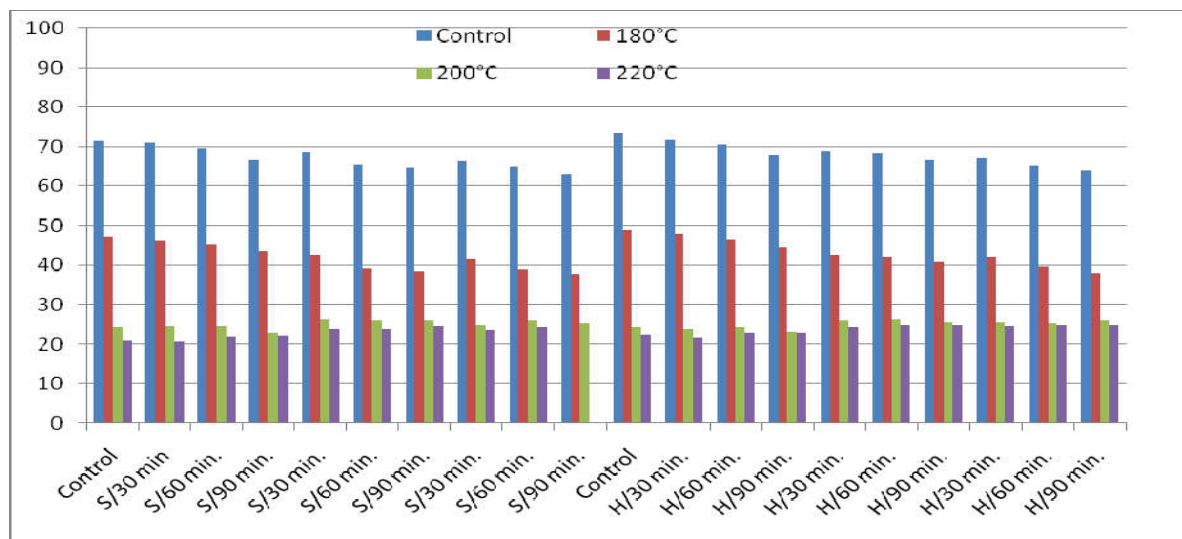
Table 1. Chemical constituents of 15-year-old planted *Acacia* hybrid

Wood type	Temp. °C	Treat time (min.)	Chemical constituents				
			Holocellulose	Cellulose	Hemicellulose	Lignin	
Sapwood	Control	Control	71.5 (0.0)	47.1 (0.0)	24.4 (0.0)	20.8 (0.0)	
	180	30	70.8 (-1.0)	46.2 (-1.9)	24.6 (0.8)	20.7 (-0.5)	
	180	60	69.7 (-2.5)	45.1 (-4.2)	24.6 (0.8)	22.0 (5.8)	
	180	90	66.6 (-6.9)	43.6 (-7.4)	23.0 (-5.7)	22.1 (6.3)	
	200	30	68.6 (-4.1)	42.5 (-9.8)	26.1 (7.0)	23.9 (14.9)	
	200	60	65.3 (-8.7)	39.2 (-16.8)	26.0 (6.6)	23.8 (14.1)	
	200	90	64.5 (-9.8)	38.5 (-18.3)	26.0 (6.6)	24.7 (18.8)	
	220	30	66.4 (-7.1)	41.6 (-11.7)	24.8 (1.6)	23.7 (13.9)	
	220	60	64.9 (-9.2)	38.9 (-17.4)	26.0 (6.6)	24.3 (16.8)	
	220	90	63.1 (-11.7)	37.7 (-20.0)	25.4 (4.1)	24.0 (15.4)	
	Heartwood	Control	Control	73.4 (0.0)	48.9 (0.0)	24.5 (0.0)	22.4 (0.0)
		180	30	71.7 (-2.3)	47.8 (-2.3)	23.9 (-2.5)	21.7 (-3.1)
180		60	70.6 (-3.8)	46.3 (-5.3)	24.3 (-0.8)	22.9 (2.2)	
180		90	67.9 (-7.5)	44.7 (-8.6)	23.2 (-5.3)	23.0 (2.7)	
200		30	68.8 (-6.3)	42.6 (-12.9)	25.9 (5.7)	24.5 (9.4)	
200		60	68.3 (-6.9)	42.1 (-14.0)	26.2 (6.9)	24.9 (11.2)	
200		90	66.5 (-9.4)	40.9 (-16.4)	25.6 (4.5)	25.0 (11.6)	
220		30	67.1 (-8.6)	42.0 (-14.1)	25.7 (4.9)	24.7 (10.3)	
220		60	65.1 (-11.3)	39.7 (-18.8)	25.4 (3.7)	24.8 (10.7)	
220		90	64.0 (-12.8)	38.1 (-22.1)	25.9 (5.7)	24.9 (11.2)	

Table 2. ANOVA on the chemical constituents of the 15-year-old planted *Acacia* hybrid.

S.V.	Dependent variable	Sum of square	df	Mean square	F-Ratio
Temperature, °C	Holocellulose	1618.96	3	539.65	564.15*
	Hemicellulose	139.12	3	46.37	85.82*
	Cellulose	2460.23	3	820.08	1018.75*
	Lignin	368.90	3	122.97	461.45*
Duration	Holocellulose	250.82	2	125.41	131.10*
	Hemicellulose	7.43	2	3.72	6.88 *
	Cellulose	210.26	2	105.13	130.60*
	Lignin	12.69	2	6.35	23.81*
Wood type	Holocellulose	96.80	1	96.80	101.20*
	Hemicellulose	0.01	1	0.01	0.01 ^{ns}
	Cellulose	95.20	1	95.20	118.27*
	Lignin	52.22	1	52.22	195.94*

* Significant at p ≤ 0.01, ns = not significant.



S=Sapwood, H=Heartwood, x-axis representing the wood sample at different treatment temp and treatment duration, y-axis representing percentage of the chemical constituents.

Figure 1. Changes in the chemical constituents between the controls and oil heat-treated of planted *Acacia* hybrid at various temperatures and treatment durations

cellulose between 38.1% and 47.8%, and lignin between 22.4% and 27.0% at 180 - 220°C with 30 to 90-minute treatments. The result showed chemical composition in heartwood of *A. hybrid* was higher than sapwood.

Holocellulose: The result in Figure 1 respectively show that holocellulose content for the treated sapwood and heartwood slightly reduced with the increasing of treatment temperature and time of heating exposure in comparison to both untreated sapwood and heartwood.

Holocellulose content determined for both wood types after heat treatment was between 98% and 83% for sapwood, while the values for heartwood were between 96% and 82%. Earlier studies showed that heat treatment resulting in a decrease of the holocellulose content. A previous study done by Inari *et al.*, (2007) found that holocellulose content of beech and pine after heat treatment was between 50% and 60%. While Boonstra and Tjeerdsma (2005) also found that holocellulose content of heat-treated Scots pine heated at 180°C decreased between 79.7% and 63.3%. According to Hill (2006); Wahab *et al.*, (2012), the holocellulose content can decrease when wood-heated at a temperature above 100°C. The content decrease is associated with the loss of cellulose and hemicellulose during heating.

Hemicellulose: Figure 1 shows the results of the chemical analyzed on hemicelluloses content for both the treated sapwood and heartwood. The results showed fluctuation values heading to no specific trend during this treatment. This might be due to the structural heterogeneity of hemicellulose, which is a complex matter to reveal their thermal behaviour (Manninen *et al.*, 2002; Kotilainen, 2000). From the observation, hemicelluloses content for sapwood degrades 5.7% when treated at a temperature 180°C in 90 minutes, while heartwood's hemicelluloses content degraded from 2.5% to 5.3% when treated at a temperature 180°C from 30 to 90 minutes. The increment in lignin content in wood with treatment time showed higher susceptibility of the hemicelluloses to thermal treatment (Mburu *et al.*, 2008). There are variations in the literature regarding the exact temperature for the onset of hemicelluloses degradation although it was well known that hemicelluloses are less thermally stable than cellulose (Hill, 2006). Rowell *et al.*, (2002), stated that hemicelluloses degradation is predominant at temperatures below 200°C. As refer to Kotilainen (2000), the lower thermal stability of hemicellulose compared to cellulose is usually explained by the lack of crystallinity. In the pyrolysis of hemicelluloses, more gaseous products and less charred residue formed if compared to cellulose. Theander and Nelson (1988) reported that upon heating of wood, degradation of hemicelluloses starts with the liberation of acetic acid and it has been found in emissions from drying of Radiata pine at 100°C. When wood is heated, the most thermally labile of the hemicelluloses begin to degrade, resulted in the production of methanol, acetic acid and various volatile heterocyclic compounds (Hill, 2006). The acetic acid generated when the acetylated hydroxyl groups of the hemicellulose chains are split off (Johansson, 2008). The volatile organic acids formed due to the heating of wood are trapped in the process and promote the degradation rate (Viitaniemi, 2001).

Cellulose: Cellulose content determined for both wood types after heat treatment between 98% and 80% for sapwood, while the values for heartwood were between 97% and 78%. The result showed that minor degradation of celluloses can be seen when wood treated at 180°C in 30 minutes and it continued to decrease with the increase of temperature and time of treatment. Cellulose is more resistant to hydrolysis than hemicelluloses, pectins and starch, and has a more regular and crystalline structure with considerably higher molecule weight (Sundqvist, 2004). As stated by Yildiz *et al.*, (2006), the crystalline structure of cellulose is not changed or even can improve up to a certain temperature, which may be as high as 200°C depending on the conditions involved.

From the analysed of the molecule size of cellulose in heat treatment by using intrinsic viscosity measurement, it showed that heat treatment results in a considerable reduction in molecule size of cellulose. Different process conditions and treatment time applied during heat treatment can influence the degradation rate of cellulose content (Boonstra and Tjeerdsma, 2005). Fengel and Wegener (1989), found that the degree of polymerization (DP) of cellulose is already decreased in thermally treated spruce at temperatures above 120°C due to cleavage of the glucosidic bonding that is accelerated by the presence of acids that are catalyzing the reaction. With extended heating, chain scission of the cellulose occurs, producing alkaline soluble oligosaccharides, with a concomitant decrease in the cellulose DP and degree of crystallinity (Hill, 2006). CO₂ and CO are produced when cellulose is heated at 170°C (Shafizadeh, 1984) and heating for a longer periods results in an increase in carbonyl groups at the expense of carboxylic moieties (Fengel and Wegener, 1989). According to Hill (2006), the amorphous regions of cellulose are more susceptible to thermal degradation and probably exhibit similar thermal properties to the hexose components of hemicelluloses. The rate of cellulose degradation reduced if the water is present, which is assumed to be due to enhanced ability of the amorphous regions to change structure to produce more thermally stable crystalline region (Fengel and Wegener, 1989).

LIGNIN: Lignin composition in both the sapwood and heartwood of the *A. hybrid* increased with an increase in treatment temperature and time. The lignin starts to increase slowly when temperature increased, and this reaction continues to occur due to the extended time of exposure. Table 1 showed an increment in lignin composition of *Acacia* sapwood was obtained from 20.8% in the untreated wood to 24% in the heat-treated wood while lignin composition of *Acacia* heartwood was obtained from 22.4% in the untreated heartwood to 25% in the heat-treated wood. The changed in lignin composition showed that there was a change in the lignin structure of *A. hybrid*. This reinforced by the earlier studies that have been observed by several researchers that heat treatment causes an increment in lignin composition in wood (Brito *et al.*, 2008; Mburu *et al.*, 2008; Inari *et al.*, 2007; Yildiz *et al.*, 2006; Sarni *et al.*, 1990). Increase in the syringe ratio of lignin occurred during heating as the treatment temperature increases (Sarni *et al.*, 1990). Similar finding occurred in *Grevillea robusta* wood where there is an increment in lignin content with treatment time (Mburu *et al.*, 2008). The changed in the lignin were strongly dependent upon the temperature and time of treatment (Sudo *et al.*, 1985). This is reinforced by the analysis of variance in Table 2 which showed that there was a significant difference between the temperatures and time of the treatment relating to the content of lignin. The loss of polysaccharides material on heating leads to an increase in the lignin content of the wood (Sandermann and Augustin, 1964). The relative mass proportion of lignin increased with both elevated temperature and treatment time, with a simultaneous decrease in the mass proportion of carbohydrates (Kotilainen, 2000). The increment in lignin composition could not be explained based on the assumption that there could have been a synthesis of lignin during heat treatment (Brito *et al.*, 2008). It is possible to suppose that as there was a reduction in sugar contents, lignin would have increased proportionally, because it is practically inert to degradation caused by heat, depends on the treatment

condition used in the study (Izyan *et al.*, 2010; Inari *et al.*, 2007; Yildiz *et al.*, 2006). The lignin presents higher resistance to the heat, there was a significant and proportional increase in its content (Brito *et al.*, 2008). The lignin in the wood is affected in approximately 100°C for wet conditions are considered to be the plasticization temperature of wood which is related to changes in lignin structure. Partial depolymerization occurred at 135°C for beech (Kosikova *et al.*, 1999). The lignin does not change as much as the hemicelluloses when subjected to hydrolytic conditions below 200°C. Mild acidic hydrolysis of lignin is proposed to be the result of the breaking of cyclic α -aryl ether bonds giving various lignin fragments such as lignols. At above 200°C the lignin degradation rate and the concentration of radicals that is formed are reported to strongly increase. Noticeable changes in the lignin structure start at temperatures around 120°C and with rising temperatures the changes increase (Sundqvist, 2004). At temperature around 180°C, the degradation of lignin in heat-treated wood is considerable. Relatively weak aliphatic bonds breakdown and hydrocarbon fragments are released. As secondary reactions, hydrocarbon fractions are degraded further and re-polymerization occurs, and simultaneously, some of the functional groups are cleaved and gaseous low-molecular-mass products are evolved precursors light hydrocarbons.

The summary of ANOVA of chemical constituents of the *A. hybrid* given in Table 2. Almost all of the chemical components significantly affected by temperature, treatment time and wood types. The summary of ANOVA showed that the significant difference between the treatment temperature, time and wood type of the treatment relating to the content of holocellulose, cellulose and lignin. It can be concluded that the effects of temperature, treatment time and wood types were caused by the change in the chemical composition of that chemical component. The summary of ANOVA for hemicellulose showed that there was no significant difference with wood type of treatment relating to the content of hemicellulose. It can be concluded that the wood types did not affect the change in hemicellulose content in oil heat-treatment process. The effects of temperature and treatment time were a cause of decrement of hemicellulose composition. From the observation, the change in chemical content in the wood increased with the increase in treatment temperature. This means that most of the chemical composition in the *A. hybrid* wood is profoundly affected by treatment temperature, time and wood types in oil heat-treatment process.

CONCLUSION

The chemical constituents' changes occurred in the *A. hybrid* wood during the oil-treatment process. The degradation in holocellulose, cellulose, and hemicellulose contents occurred when *A. hybrid* woods exposed to oil-heat treatment process at temperatures higher than 180°C. The degradation shows some variation from temperatures 180 to 220°C at different exposure times. Changes in chemical constituents in the wood increases with the increase in the treatment temperature. Most of the chemical constituents in *A. hybrid* wood are highly affected by the treatment temperature, time and wood types in the oil heat-treatment process. Holocellulose and cellulose degraded with the increasing of treatment temperature and time of heating exposure, while lignin showed the increment in the content through this treatment. Holocellulose content determined for

both wood types after heat treatment was between 98% and 83% for sapwood, while the values for heartwood were between 96% and 82%. Hemicelluloses content for sapwood degrades 5.7% when treated at a temperature 180°C in 90 minutes while heartwood's hemicelluloses content was degraded from 2.5% to 5.3% when treated at a temperature 180°C from 30 to 90 minutes. There was no significant difference with the wood type of treatment relating to the content of hemicellulose. Therefore, it can be concluded that the wood types did not affect the change in hemicellulose content in oil heat-treatment process. The effects of temperature and treatment time were a cause of decrement of hemicellulose composition. Cellulose content determined for both wood types were between 98% and 80% for the sapwood and the values for heartwood were between 97% and 78% after undergoing the oil-heat treatment process. An increment in lignin composition of *A. hybrid* increases from 20.8% in the control sapwood to 24% in the treated wood, while the lignin composition heartwood increases from 22.4% in the control heartwood to 25% in the heat-treated wood. The significant difference occurred between the temperatures and time of the treatment relating to the content of lignin. It can be concluded that the effects of temperature were a cause of increment of lignin. There was a significant difference between the treatment temperature, time and wood type of the treatment relating to the content of holocellulose, cellulose and lignin. It can be concluded that the effects of temperature, treatment time and wood types caused by the change in chemical composition of that chemical component.

Acknowledgement

This study financed by the University College of Technology Sarawak Grants (UCTS/RESEARCH/4/ 2017/01) and (UCTS/RESEARCH/2/2018/01). The authors expressed their gratitude to University College of Technology Sarawak (UCTS) and Forest Research Institute Malaysia (FRIM) for permission in using their laboratory and workshop equipment in the preparation and analysis parts of the study.

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