



Chemical Changes in 15 Year-old Cultivated *Acacia Hybrid* Oil-Heat Treated “at 180, 220 and 220°C”

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The research was financed by The Ministry of Science, Technology and Invention, Malaysia, under the Science Fund Project SCF0037-IND-1/2007.

Abstract

The chemical constituents of oil-heat treated cultivated 15 years-old *Acacia hybrid* were investigated. The logs of *A. hybrid* were harvested and cut at bottom, middle and top portions and oil-heat treated using organic palm oil at temperatures of 180, 200 and 220°C for the time 30, 60 and 90 minutes. The wood samples were dried and grinded into sawdust, and air-dried again prior to the chemical analysis. Untreated samples were used as controls. The results on the analysis of the chemical contents in the oil-treated *A. hybrid* shows some changes occurred when treated from 180 to 220°C. The variation occurred in the chemical contents for both the sapwood and heartwood. The holocellulose

contents decrease from 71.5 to 63.1% and 73.4 to 64.0% for sapwood and heartwood respectively. The cellulose contents decreased from 47.1 to 37.7% for the sapwood and 48.9 to 38.1% for heartwood. The hemicellulose content's increases from 24.4 to 25.4% in the sapwood and 24.5 to 25.9% for the heartwood. 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.

Keywords: Cultivated *acacia hybrid*, Oil-heat treatment Process, Wood and chemical changes

1. Introduction

Forest's plantation has becomes importance due to the declining in supply of large logs from the natural forest in Malaysia. It is an alternative way to patch the industries demand, since plantation forestry rotations are significantly shorter than in natural stands. Moreover, nowadays, consumer countries in Europe and North America are very sensitive about logging activities of naturally grown species from natural tropical rain forest. Effort to establish the plantation sector becoming a more concern by government and private sector in order 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. This project which started in 1982, aim for sustainable supply for the timber industries which was expected to face a shortfall from the natural forests (Ahmad, 1995). Acacia species was chosen due to its rapid growth, and ease adaptation to the local soil (Pinyopusarerk *et al.*, 1993). Moreover, acacia species such as *Acacia mangium*, *A. auriculiformis* and *A. hybrid* was major fast growing plantation species not only for timber production, but also for greening purposes in the tropical region (Yamamoto *et al.*, 2003; Semsuntud *et al.*, 1991; Hamimi *et al.*, 1989). Furthermore, the properties of naturally grown species seem to vary much more than that plantation species. Acacia is slightly durable species, especially for exposed condition and in ground contact uses. The preservative treatment has to be used in order to prolong the uses of the wood products for certain periods of time. However, wood treatment used preservative, which mostly have heavy metals and discharge toxin to the environment.

A lot of efforts have been put to develop new wood preservatives. In addition, developed countries have totally banned the use of Chrome-copper Arsenic (CCA) in their woody materials (Berard *et al.*, 2006). Increased in environmental awareness by the general public, combined with an increasingly stringent legislature in recent years has led to greater restrictions in 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 (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 to being non toxin and does not require chemical application.

The aims of this study were 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 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.

2. Materials and methods

i. Materials: Three (3) defects free 15 year-old *A. hybrid* logs were harvested from Sabah Forestry Development Authority (SAFODA) forest plantation in Kinarut, Kota Kinabalu, Sabah. The logs were selected based on their good standing condition, long straight bole, decay-free and minimum branches. The average diameters of the trees selected at breast height were 25 to 30 cm. A chain saw was used for harvesting the trees and wood blocks 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 wood blocks were then transported to UMS for further processing and subsequent testing.

Sample Preparation: The wood blocks were air dried under shed for about a month to reduce the moisture to an equivalent moisture content (15%) and to remove stresses in them. After drying, the wood blocks were 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 comprises 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 were used as 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 30 x 16 x 12 inch (length x width x height) in three inch thickness. The heat of the tank was generated through electricity power sources. Three replicates were used for each treatment condition of temperature and time. Before the treatment, samples were stabilized to 12% moisture content (MC) in a conditional chamber set at 65% RH and temperature 25°C. All together 81 samples were prepared

prior to the treatment. Coding was 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 in nature and has a high boiling point of 320°C (Rafidah, 2009; 2008; Razak *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 were applied at time of 30, 60 and 90 minutes. Samples were 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. Samples were then conditioned in a conditioning chamber at 20±2°C temperature and 65±5% relative humidity to obtain 12% MC. The samples were then cut into respective sizes in accordance to testing procedures.

ii. 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 were 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 conducted using TAPPI T203 cm-99 (1999) and TAPPI T222 OM-01 (2002) standard.

Sample Preparation: Wood samples were divided into the heartwood and sapwood for study on the differences in chemical composition. Samples were chipped for grinding purposes. Samples were ground into powder with Willey's mill in order 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 were chemically analyzed.

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 reweigh 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 were performed with extractive free wood that was derived from the alcohol-toluene extractive test. Additionally, holocellulose was a necessary preparatory stage in order 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 was weighed to an accuracy of 0.01 g in a weighing timble. The timble was 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 were placed on a heating mantle. The extraction of 230ml alcohol-toluene solution was conducted for 8h at the rate of approximately six siphoning per hour. When extraction was 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 a desiccator and weighed until a constant weighed was obtained. The alcohol-toluene solubility content of acacia wood was obtained using Equation 1.

$$\text{Alcohol-toluene solubles(\%)} = (W2/W1) \times 100 \quad (1)$$

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 was 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 was added after 30 minutes of heating. 1.5 g sodium chloride was added after the second 30 minutes. This procedure was repeated until 6 g sodium chlorite and 20 ml of 10% acetic acid was added. At the end of heating, the beakers were placed in an ice water bath until the temperature of the beaker was reduced to 10°C.

The contents of the beaker were filtered into a coarse fritted glass crucible (porosity 1) of known weight. The residua was washed with iced distilled water and acetone. The residue changed colour from yellow to white. The crucible was then air-dried at surrounding area until constant weight was reached. The holocellulose content in acacia wood was determined using Equation 2.

$$\text{Holocellulose content (\%)} = (Y2/Y1) \times 100 \quad (2)$$

Where,

Y1 = Weight of air-dried holocellulose, (g)

Y2 = Weight of oven-dried extraction residue, (g)

Determination of Cellulose: Approximately, 3 g oven-dried sample of holocellulose was placed into a 250 ml beaker. The beakers were 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 was mixed for 45 seconds. Then, 10 ml of 17.5% NaOH more was added and swirled for 15 seconds. The solution was allowed for 3 minutes (total time was 5 minutes). The solution was added again with 10 ml of 17.5% NaOH and swirled for 2 and half minutes. This procedure was repeated for 3 times (total time was 15 minutes). The solution was allowed to react with the solution for 30 minutes. After added and stirred with 100 ml distilled water the solution was allowed to react for 30 minutes and the total time was 75 minutes.

The contents of the beaker were filtered by an aid of vacuum suction into 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 was stopped and 2N acetic acid was filled in the crucible to allow the residue soak for 5 minutes. 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 was oven-dried in an oven at 105±2°C, then cooled in a desiccator and weighed until constant weighed was reached. The Equation 3 was used to obtain the alpha-cellulose content in acacia wood:

$$\text{Oven-dried alpha-cellulose sample (\%)} = (Z2/Z1) \times 100 \quad (3)$$

Where,

Z1 = Weight of oven-dried sample, (g)

Z2 = Weight of oven-dried alpha-cellulose, (g)

Determination of Lignin: Determination of lignin content was referred from TAPPI Standard T 222 om-02. 72% sulfuric acid was prepared by pouring 665 ml of concentration sulfuric acid into 300 ml water. After cooling, another 700 ml of water added to make a 1000 ml solution. The acid solution was 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 pipet and the mixture was 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. While the solution was refluxing (boiled under reflex for 2 hours), a crucible was oven dried for 1 hour at 110°C, then allowed to cool in a desiccator (15 minutes) and accurately weighed.

When the refluxing was completed, the insoluble lignin was recovered by filtration through the crucible after allowing the lignin to settle to facilitate filtration. The lignin free was washed with 250 ml of hot distilled water. The lignin containing the lignin was dried at 110°C for 1 hour, cooled in a desiccator (15 minutes) and weighed. The Equation 3 was used to obtain the lignin content in the *A. hybrid*:

$$\text{Lignin content in acacia wood (\%)} = [(W4-W3)/(100 \times W2)] \times (100- W1) \quad (4)$$

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)

3. Results and discussion

Table 1 shows 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 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. However, the result shown, chemical composition in heartwood of *A. hybrid* was higher than sapwood.

Holocellulose: The result in Figure 1 and 2 respectively shows that holocellulose content for the treated sapwood and heartwood was 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%.

It has been observed in several studies 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 were decreased between 79.7% and 63.3%. According to Hill (2006), 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 and 2 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 degrade 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 confirming 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 predominate 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. Moreover, in the pyrolysis of hemicelluloses, more gaseous products and less charred residue are formed 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, resulting in the production of methanol, acetic acid and various volatile heterocyclic compounds (Hill, 2006). The acetic acid is generated when the acetylated hydroxyl groups of the hemicellulose chains are split off (Johansson, 2008). It is suggested that volatile organic acids formed due to 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 were between 98% and 80% for sapwood, while the values for heartwood were between 97% and 78%. The result (Table 3) showed that minor degradation of celluloses can be seen when wood treated at 180°C in 30 minutes and it continually to decrease with the increase of temperature and time of treatment.

Cellulose is more resistant to hydrolysis than hemicelluloses, pectins and starch, and it has generally a more regular and crystalline structure with considerably higher molecule weight (Sundqvist, 2004). As stated by Yilidiz *et al.* (2006), 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 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 degradation rate of cellulose content (Boonstra & 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 is reduced if 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 start to increase slowly when temperature is increase and this reaction continue to occur due to the extended time of exposure. From the result (Table 1), 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 changed in the lignin structure of *A. hybrid*. This reinforced by the earlier

studies that has 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).

Sarni *et al.* (1990) found that an increase in the syringly ratio of lignin during heating was observed as the treatment temperature increased. While, Mburu *et al.* (2008) also found that there was an increment in lignin content of *Grevillea robusta* wood with treatment time. Sudo *et al.* (1985) noted that the changed in the lignin were strongly dependent upon the temperature and time of treatment. This 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 content of lignin.

According to Sandermann and Augustin (1964), the loss of polysaccharides material on heating leads to an increase in the lignin content of the wood. Kotilainen (2000) also reported that the relative mass proportion of lignin increased with both elevated temperature and treatment time, with a simultaneous decrease in the mass proportion of carbohydrates. However, according to Brito *et al.* (2008), an increment in lignin composition could not be explained based on the assumption that there could have been a synthesis of lignin during heat treatment. Still, 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 (Inari *et al.* 2007; Yildiz *et al.* 2006). Since lignin presents a higher resistance to heat, there was a significant and proportional increase in its content (Brito *et al.*, 2008).

However, there are also a researchers found that the degradation in lignin content due to heat treatment of wood. Those findings contradict with this present result in this study. The lignin of 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. A partial depolymerization has been reported at 135°C for beech (Kosikova *et al.*, 1999). Though, lignin is not changed 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. Above 200°C the lignin degradation rate and the concentration of radicals that is formed are reported to strongly increase.

Sundqvist (2004) reported that noticeable changes in the lignin structure start at temperatures around 120°C and with rising temperatures the changes increase. 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 repolymerization occurs and simultaneously, some of the functional groups are cleaved and gaseous low-molecular mass products are evolved precursors light hydrocarbons.

Analysis of Variance on Chemical Composition

The summary of ANOVA of chemical compositions of the *A. hybrid* is given in Table 2. Almost all of the chemical components were significantly affected by temperature, treatment time and wood types.

The summary of ANOVA showed that there were a significant difference between the treatment temperature, time and wood type of the treatment relating to the content of holocellulose, cellulose and lignin. Therefore, it can be concluded that the effects of temperature, treatment time and wood types were a caused of the change in chemical composition of those chemical component.

For hemicellulose, the summary of ANOVA showed that there was no significant difference with wood type of the treatment relating to the content of hemicellulose. Therefore, it can be concluded that the wood types din not affected the changed 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 changed in chemical content in wood was increased with the increasing in treatment temperature. This means that most of the chemical composition in *A. hybrid* wood is highly affected by treatment temperature, time and wood types in oil heat treatment process.

4. Conclusion

The oil-heat treatment process caused some features changed in the chemical composition of *A. hybrid* wood. The degradation in holocellulose, cellulose, and hemicellulose contents occurred when *A. hybrid* woods were 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. From the observation, the changed in chemical content in wood was increased with the increasing in treatment temperature. This means that most of the chemical composition in *A. hybrid* wood is highly affected by treatment temperature, time and wood types in 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 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 degrade 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 wood type of the treatment relating to the content of hemicellulose. Therefore, it can be concluded that the wood types did not affected the changed 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. There was a significant difference between the temperatures and time of the treatment relating to content of lignin. Therefore, it can be concluded that the effects of temperature were a cause of increment of lignin.

There were significant difference between the treatment temperature, time and wood type of the treatment relating to the content of holocellulose, cellulose and lignin. Therefore, it can be concluded that the effects of temperature, treatment time and wood types were a caused of the change in chemical composition of those chemical component.

References

- Ahmad Shakri, M. S. (1995). Finishing Properties of *Acacia mangium*, *Paraserianthes falcataria* and *Gmelina arborea* Timbers: Some Important Parameters. *Journal of Tropical Forest Products*, 1(1): 83-89.
- Berard, P., Laurent, T. and Dumonceaud, O. (2006). Use of Round Wood of Chestnut Tree Coppices: Crack Risk and Effects of a Hot Oil Bath Treatment. *Journal of Holz als Roh-und Werkstoff*, 64: 287-293. DOI 10.1007/s00107-005-0086-4.
- Boonstra, M. J. and Tjeerdsma, B. (2005). Chemical Analysis of Heat Treated Softwoods. *Journal of Holz als Roh-und Werkstoff*, DOI 10.1007/s00107-005-0078-4.
- Brito, J. O., Silva, F. G., Leao, M.M and Almeida, G. (2008). Chemical Composition Changes In Eucalyptus and Pinus Woods Submitted to Heat Treatment. *Wood. Journal of Bioresource Technology*, DOI 10.1016/j.biortech.2008.03.069.
- Charrier, B., Charrier, F., Janin, G., Kamdem, D.P., Irmouli, M. and Goncalez, J. (2002). Study of Industrial Boiling Process on Walnut Colour: Experimental Study Under Industrial Conditions. *Journal of Holz als Roh-und Werkstoff*, 60: 259-264. DOI 10.1007/s00107-005-0082-8.
- Fengel, D. and Wegener, G. (1989). *Wood Chemistry, Ultrastructure, Reactions*. Walter de Gruyter and Co. Berlin, New York.
- Hamimi, M. S., Razali, A. K. and Kee, K. T. (1989). Gluing Properties of Three Fast-growing Plantation Species. *Journal of Malaysian Forest*, 52 (2): 46-60.
- Hill, C. A.S. (2006). *Wood Modification: Chemical, Thermal and Other Processes*. John Wiley & Sons, Ltd. England.
- Hyvonen, A., Piltonen, P. and Niinimäki, J. (2005). Tall Oil/Water- Emulsions as Water Repellents for Scots Pine Sapwood. *Journal of Holz als Roh-und Werkstoff*, 64: 68-73. DOI 10.1007/s00107-005-0040-5.
- Inari, G. N., Petrisans, M. and Gerardin, P. (2007). Chemical Reactivity of Heat-treated Wood. *Journal of Wood Science and Technology*, 41: 157-168.
- Johansson, D. (2008). *Heat Treatment of Solid Wood; Effects on Absorption, Strength and Colour*. Ph.D. Thesis. Lulea University of Technology. ISSN: 1402-1544.
- Kosikova, B., Hricovini, M. and Cosentino, C. (1999). Interaction of Lignin and Polysaccharides in Beech Wood (*Fagus sylvatica*) During Drying Processes. *Journal of Wood Science and Technology*, 33: 373-380.
- Kotilainen, R. (2000). *Chemical Changes in Wood During Heating at 150°C-260°C*. Ph.D Thesis. Jyväskylä University, Finland.
- Manninen, A. M., Pasanen, P. and Holopainen, J.K. (2002). Comparing the VOC Emissions between Air-dried and Heat-treated Scots Pine Wood. *Journal of Atmospheric Environment*, 36: 1763-1768.
- Mburu, F., Dumarcay, S., Bocquet, J.F., Petrisans, M. and Gerardin, P. (2008). Effect of Chemical Modifications Caused by Heat Treatment on Mechanical Properties of *Grevillea robusta* Wood. *Journal of Polymer Degradation and Stability*, 93: 401-405. DOI: 10.1016/j.polymdegradstab.2007.11.017.
- Pinyopusarerk, K., Liang, S. B. and Gunn, B.V. (1993). Taxonomy, Distribution, biology and Use As An Exotic. *Proceeding of Acacia Mangium Growing and Utilization*. Bangkok, Thailand. 1-19.

- Rafidah, S., Razak, W., Zaidon, A. & Hashim W. S. (2009). Chemical constituents of bamboo *Gigantochloa scortechinii* oil-cured at 140°C, 180°C and 220°C. *Journal of Applied Science*, 9(1): 149-154. ISSN 1812-5654. Asian Network for Scientific Information.
- Rafidah, S., Razak, W. & Zaidon, S. (2008). Effect of Oil Heat Treatment on Chemical Constituents of Semantan Bamboo (*Gigantochloa scortechinii* Gamble). *Journal of Sustainable Development*, 1(2): 91-98. Canadian Center of Science and Education.
- Razak, W., Aminuddin, M., Hashim, W.S. & Othman, S. (2005). Effect of heat treatment using palm oil on properties and durability of Semantan bamboo. *Journal of Bamboo and Rattan*, 4 (3): 211-220. ISSN: 1569-1568, International Network for Bamboo and Rattan.
- Resch, H., Hansmann, C. and Pokorny, M. (2000). The Colour of Wood from White Oak. *Journal of Holzforchung*, 54(1): 13-15
- Rowell, R. M., Pettersen, R., Han, J. S., Rowell, J. S. and Tshabalala, M. A. (2005). Cell Wall Chemistry. In Rowell R.M. (ed.). *Handbook of Wood Chemistry and Wood Composites*, pp. 37-72. Madison: CRC Press.
- Sandermann, W. and Augustin, H. (1964). Chemical Investigations on the Thermal Decomposition of Wood. Part III: Chemical Investigation on the Course of Decomposition. *Journal of Holz als Roh-und Werkstoff*, 22(10): 377-386.
- Sarni, F., Moutounet, M., Puech, J. L. and Rabier, P. (1990). Effect of Heat Treatment on Oak Wood Extractable Compounds. *Journal of Holzforchung*, 44(6): 461-466.
- Semsuntud, N. and Nitiwattanachi, W. (1991). Tissue Culture of *Acacia auriculiformis*. In Advance in Tropical Acacia Research, ACIAR. 35: 64-66
- Shafizadeh, F. (1984). The Chemistry of Pyrolysis and Combustion. In The Chemistry of Solid Wood, Rowell, R.M. (Ed). ACS Symposium Series, 207, pp. 489-529.
- Sudo, K., Shimizu, K. and Sakurai, K. (1985). Characterization of Steamed Wood Lignin from Beech Wood. *Journal of Holzforchung*, 39(5): 281-288.
- Sundqvist, B. (2004). *Colour Changes and Acid Formation in Wood during Heating*. Ph.D. Thesis. Lulea University of Technology.
- Technical Association of the Pulp and Paper Industry. (1999). TAPPI Standard T203 cm-99. Atlanta, USA.
- Technical Association of the Pulp and Paper Industry. (2002). TAPPI Standard T222 om-02. Atlanta, USA.
- Viitaniemi, P. (2001). The Thermal Modification of Wood with Heat Treatment. *VTT Building and Transport*, pp. 1-21, Espoo, Finland.
- Yamamoto, K., Sulaiman, O., Kitingan, C., Choon, L.W. and Nhan, N. T. (2003). Moisture Distribution in Stems of *Acacia mangium*, *A. auriculiformis* and Hybrid *Acacia* Trees. *JARQ*, 37(3): 207-212.
- Yildiz, S., Gezer, E. D. and Yildiz, U. C. (2006). Mechanical and Chemical Behaviour of Spruce Wood Modified by Heat. *Journal of Building and Environment*, 41:1762-1766.

Table 1. Mean result of chemical composition of *Acacia* hybrid

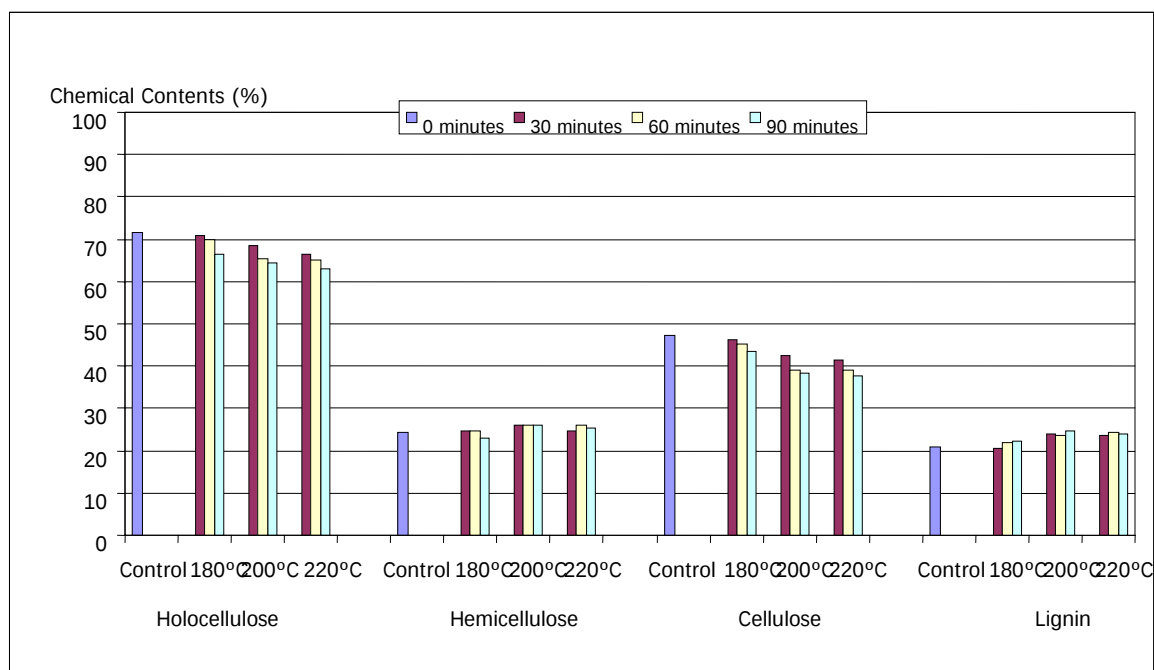
Wood type	Temp. (°C)	Treatment time (min)	Chemical Composition (%)			
			Holocellulose	Cellulose	Hemicellulose	Lignin
Sapwood	Control	Control	71.5	47.1	24.4	20.8
		30	70.8 {-1.0}	46.2 {-1.9}	24.6 {0.8}	20.7 {-0.5}
		60	69.7 {-2.5}	45.1 {-4.2}	24.6 {0.8}	22.0 {5.8}
		90	66.6 {-6.9}	43.6 {-7.4}	23.0 {-5.7}	22.1 {6.3}
	200	30	68.6 {-4.1}	42.5 {-9.8}	26.1 {7.0}	23.9 {14.9}
		60	65.3 {-8.7}	39.2 {-16.8}	26.0 {6.6}	23.8 {14.4}
		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}
		60	64.9 {-9.2}	38.9 {-17.4}	26.0 {6.6}	24.3 {16.8}
		90	63.1 {-11.7}	37.7 {-20.0}	25.4 {4.1}	24.0 {15.4}
	180	Control	73.4	48.9	24.5	22.4
		30	71.7 {-2.3}	47.8 {-2.3}	23.9 {-2.5}	21.7 {-3.1}
		60	70.6 {-3.8}	46.3 {-5.3}	24.3 {-0.8}	22.9 {2.2}
		90	67.9 {-7.5}	44.7 {-8.6}	23.2 {-5.3}	23.0 {2.7}
Heartwood	200	30	68.8 {-6.3}	42.6 {-12.9}	25.9 {5.7}	24.5 {9.4}
		60	68.3 {-6.9}	42.1 {-14.0}	26.2 {6.9}	24.9 {11.2}
		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}
		60	65.1 {-11.3}	39.7 {-18.8}	25.4 {3.7}	24.8 {10.7}
		90	64.0 {-12.8}	38.1 {-22.1}	25.9 {5.7}	24.9 {11.2}

Note: { } = % change from control

Table 2. ANOVA of chemical compositions of the 14 year-old *Acacia* hybrid

Source of Variance	Dependent Variable	Sum of Squares	Df	Mean Square	F-Ratio
Temperature	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**
Time	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 types (Sap and Heartwood)	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**

Note: ** = significant difference at $p \leq 0.01$, * = significant difference at $p \leq 0.05$, ns = not significant

Figure 1. Chemical composition of *Acacia* hybrid of the treated sapwood

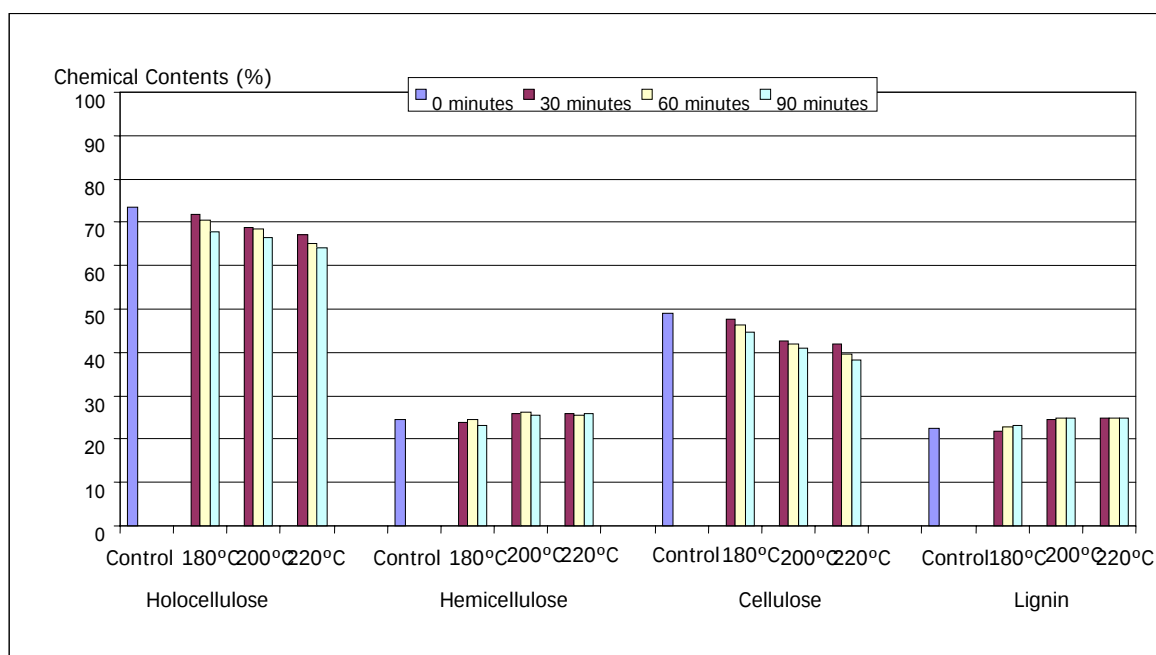


Figure 2. Chemical composition of *Acacia hybrid* of the treated heartwood