Effect of Oil Heat Treatment on Chemical Constituents of Semantan Bamboo (*Gigantochloa scortechinii* Gamble)

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Abstract

Effect of oil heat treatment on chemical constituents of 3 years old *Gigantochloa scortechinii* Gamble bamboo was investigated. The bamboo splits within epidermis were heat-treated using crude palm oil at temperature 140°C, 180°C and 220°C for duration 30 and 60 min. After removed the epidermis, the samples were then grind to pass a BS 40-mesh sieve and retained on a BS 60-mesh sieve. The sawdust was air dried for several days before conducted to chemical analyses (cellulose, hemicellulose and lignin) based on TAPPI Standard Methods. The colorimetric method devised by Humprey and Kelly (1960) was adapted to analysis starch in bamboo. Reading was obtained through Baush Lomb UV Spectrophotometer at 650 µm calculated by standard reference using A.R. potato starch. Control was used as comparison for each type of test conducted. There was no significant different between control and condition at 140°C for 60 min (81.4%) of holocellulose content. The value was decreased by 2.1 to 10.7% (79.7 to 72.7%) after heating at 180 to 220°C for 30 to 60 min. The hemicellulose content of bamboo was ranged 24.1 to 27.8% after heating at 140-220°C for 30 to 60 min. The cellulose content of heat-treated bamboo was ranged 47.4 to 55.2% after reduced about 2 to 14%. Lignin content increased about 16% (26%) at 220°C/60 min after reduced approximately 1 to 5% at 140 to 180°C for 30 to 60 min. Starch content was largely reduced about 2 to 54% (4 to 1.9%) at 140 to 180°C for 30 to 60 min of treatment. The results indicated that degradation of cellulose and hemicellulose of heat-treated bamboo was attributed to plasticization of lignin during heating in the same time hydrolysed the starch content.

Keywords: Bamboo, Oil heat treatment, Chemical constituents

1. Introduction

Bamboo is regarded as eco-friendly, which grows and matures quickly has potential to be used as an alternative material for wood. The world is loosing its resource of wood due to higher demand and to recover the resources will take 15 to 20 years. Many researches agreed that the suitable age of the bamboo used is 3 to 4 years old (Thammicha, 1989; Abd. Latif *et al*., 1990; Jamaluddin, 1999).

However, bamboo is easily susceptible to fungi or insect attack during utilization. The properties will deteriorate rapidly if the material is not treated with preservatives (Liese, 1985). Low durability is a major reason for the poor acceptance of bamboo as a building material. They are often considered as a short-term material suitable only for temporary uses (Liese and Kumar, 2003). The used of preservative in bamboo has been recognized as necessary and important for utilization in furniture and construction purposes.

Recent work on the heat treatment process of timber to prepare what is currently called torrefied wood in France, plato wood in the Netherlands and retified wood in the USA has shown that such types of processes can improve the performance of timber in several aspects (Ruyter, 1989). Foremost advantages of the wood treated improve its resistance to fungal decay (Ruyter, 1989 and van Zuylen, 1995) without the need to use any chemical solutions with consequent environmental advantages, water repellency and improve dimensional stability to moisture variations.

Oil heat treatment is another alternative way in treating bamboo without use of preservatives. This process is considered as eco-friendly treatment. Leithoff and Peek, 1997 reported that only temperature above 170°C was effective to enhance durability of bamboo however, Boonstra and Tjeerdsma, 2005 indicated that chemical composition was modified at this condition.   In this study modification of the polymeric of bamboo constituents after oil heat treatment at 140 to 220°C for 30 to 60 min was investigated. The aims of this study were:-

- To analyze the chemical properties of heat-treated bamboo

- To investigate the reaction mechanism occurring during heating at each condition applied.

2. Material and methods

2.1 Preparation of Bamboo Samples

Bamboo culms of 3 years old were extracted from randomly selected clumps in Nami, Kedah in Malaysia. The culms were cut and splitted before conditioned at 20±°C and 65 RH until constant weight. The sample were then subjected to heating at varies temperature and time.

2.1.1 Heat treatment Process

Palm oil which the boiling point of 320°C fulfilled 3/4 tank. Split bamboo samples were immersed in crude palm oils with original temperature in metallic cage of stainless steel tank. Temperature was rise gradually controlled by digital controller. Three electric heaters were generated heat from electricity power sources. Data logger connected to thermocouple where four and three channels placed inside and outside (oil) of the bamboo samples. Data were recorded every 5-10°C interval. In this experiment, temperature was applied at 140°C, 180°C and 220°C with duration 30 and 60 minutes. Bamboo samples were taken out at reached temperature. Residual oil on bamboo surface was removed by wiping with cloth to avoid absorbance into bamboo samples.

2.2 Chemical Analysis of Bamboo

2.2.1 Preparation of sawdust

A small of bamboo sample (20 mm x 20mm x thickness) was cut from the heat-treated bamboo. The epidermis of bamboo was removed before grind to pass a BS 40-mesh sieve and retained on a BS 60-mesh sieve. The sawdust was air dried for several days until it reached the constant weight prior to chemical analysis. The procedure was referred from TAPPI Standard.

2.2.2 Moisture Content of Sawdust

A small weighing bottle previously cleaned and dried in an oven was weighed on an analytical balance to 0.01 g. 2.0 g air dry sawdust was placed on the weighing bottle and reweigh to 0.01 g. The sample was dried in the oven at 105°C for three hours with the cover off. Then the bottle was removed and placed in the desiccator for 15 minutes to cool, (observing the proper precautions concerning the desiccators lid and the handling of the bottle by using tongs) before weighing. The moisture content on the basis of the air-dry weight was calculated by dividing the loss in weight by air-dry weight.

2.2.3 Determination of Holocellulose content

2 g of air dried extractive-free sawdust was weighed accurately. The sawdust was transferred quantitatively to a 250 ml conical flask. 100 ml water, 1.5 sodium chloride and 5 ml of 10% acetic acid were added and the flask was placed in a water bath maintained at 70°C, swirling the content of the flask at least once every five minutes. The flask was kept closed with a small, inverted Erlenmeyer flask.

5 ml of 10% acetic acid was added after 30 minutes. 1.5 sodium chloride was added after further 30 minutes. Alternative acetic acid and sodium chloride at 30 minutes were continued, after last addition of sodium chloride. The mixture was heated for 30 minutes after last addition of sodium chloride. Residue should be white and retain woody structure. The suspension was cooled in an ice bath.

Residue was filtered into a weighed fruited glass crucible (medium or coarse porosity) and washed with iced distilled water and finally washed with acetone. The residue was air-dried (allowed the residue to strand in the open laboratory for a day or two until it is free of acetone). Covered it with a perforated aluminium foil. The sample was transferred to a desiccator and weighed at daily intervals until the sample reached constant weight. The moisture content was determined on a 0.5 g sample which is afterward.

2.2.4 Determination of Cellulose content

Air-dried holocellulose was used. The experiment was carried out in a water bath at 20°C. About 2 g of sawdust were weighed out accurately and transferred into a 20 ml beaker than placed in a water bath at 20°C. 15 ml of 17.5 NaOH was added and macerated gently with a flattened glass with rod for 1 minute. 10 ml more NaOH was added and the solution was mixed for 45 seconds. Then, 10 ml more was added and mixed for 15 seconds so that at the end of 2

minutes 35 ml of the NaOH have been added. The mixture was stirred and allowed to stand for another 3 minutes. After 3 minutes, another 10 mililitre NaOH was added and mixed with stirring rod in the solution for every 2.5 minutes for 4 times.

The beaker was covered with watch glass and the mixture was left in the water bath for 30 minutes more. Then 100 ml of distilled water were added at 20°C quickly and thoroughly mixed and left the diluted mixture in the water bath for further 40 minutes. The mixture was filtered into a weighed fruited glass crucible (coarse porosity). If suspended fibers are noticed in the filtered, pass it through the cellulose mat again to clarify it. The beaker was rinsed and residue 25 ml of 8.3 NaOH solutions at 20°C and quantitatively transfer all the fiber to the crucible. During the filtration, the cellulose pad covered was always kept with solution to prevent drawing air through the pad.

The pad was washed with 650 ml distilled water at 20°C. The suction tube was disconnected, filled the crucible with 2N acetic acid at 20°C and the residue was allowed to soap for 5 minutes. Suction was reapplied to remove acetic acid. The residue was washed with the distilled water until it free of acid as indicated by the litmus paper. The bottom and side of the side of the crucible were wiped out with a dry towel and placed in the oven at 50°C, dried to constant weight, then cooled and weighed. Alpha cellulose was calculated as a percentage based on oven dry sample.

2.2.5 Determination of Lignin content

The procedure was referred from TAPPI Standard T 222 os-74. 1 g of air dried extractive free sawdust was weighed out accurately in weighing bottle and transferred in a 50 ml beaker. 10 ml of 72% sulphuric acid were added carefully with a pipetted and the mixture was stirred with a small glass rod (which is left in beaker). 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 3 hour), a crucible was oven dried (fine or medium porosity) for 1 hour at 110°C, then allowed to cooled 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 from act with 250 ml of hot distilled water. The crucible containing the lignin was dried at 110°C for 1 hour, cooled in a desiccator (15 minutes) and weighed. Lignin content was reported as reported as percentage by weight of the dried sample.

2.2.6 Starch Content

The method devised by Humprey and Kelly (1960) was adopted to determine the starch content through the basic reaction of the amylose in bamboo starch with iodine

2.2.6.1 Preparation of powder

Bamboo samples were first ground in order to pass a 200 mesh sieved and triplicate samples of 0.4 g each were dried for 72 hours in desiccator oven containing concentrated sulphuric acid and added with 4-7 ml of 7.2 M perchloric acid in a 50 ml beaker. Reactions were allowed to continue for 10 minutes with occasional stirring. The contents were then transferred into a 50 ml volumetric flask and made up to the volume with distilled water.

After centrifuging, 10 ml aliquots were placed in a 50 ml volumetric flask together with a drop of phenolphthalein and made alkaline with 2N Sodium hydroxide. Then 2N acetic acid was added of 2.5 ml acetic acid, 1.5 ml of 10% weight over volume potassium iodine and 5 ml 0.01N Potassium iodide. Color was allowed to develop for 15 minutes before the absorption (Baush Lomb UV Spectrophotometer) at 650 µm was measured. A blank was prepared without starch aliquot. The starch content was then calculated by applying the formula:

$$\text{Starch} = \frac{0.36778 \times (\text{E. reading} + 0.008)}{\text{Oven-dry weight of sample (g)}} \times \frac{50}{10} \times 100$$

Where,

E reading = differences of absorption between sample and blank

2.3 Statistical Analysis

The statistical analysis was carried out using the statistical analysis software (SAS). All data were analyzed using one-way analysis of variance (ANOVA) and the mean value was separated using Least Significant Difference (LSD) to determine the differences between treatment levels.

3. Result and Discussion

3.1 Effects of heating on chemical properties of G. scortechinii

The mean contents of holocellulose, hemicellulose, cellulose, lignin and starch in the treated and untreated *Gigantochloa scortechinii* after heating treatment are exhibited in Table 3.1. The hemicellulose content was calculated based on the deducted from holocellulose to alpha-cellulose.

3.1.1 Holocellulose content

The results in Table 3.1 show that holocellulose content in the treated bamboo significantly reduced when they were treated at 220°C/60 min. The chemical reduced by 10.7% from 81.4% (untreated) to 72.7%. Even though they was slight reduction for the those treated at higher temperature, the reduction however, was not significant. The holocellulose content were in the range of 77% to 81%. The reduction of holocellulose at higher temperature (above 180°C) was due the depolymerisation of the hemicelluloses and some degradation of the cellulose during heating.

In a previous study on heat treatment of Scots pine, Boonstra and Tjeerdsma, (2005) also found the similar results where the holocellulose content of the wood decreased from 79.7% to 63.3% when it was heated at 180°C.

Bamboo holocellulose contains all carbohydrates in bamboo also the sum of the cellulose and hemicellulose. The reduction of holocellulose was possibly attributed to the degradation of hemicelluloses and cellulose.

3.1.2 Hemicellulose content

There is no specific trend of hemicellulose content of bamboo. Even though there was a reduction by 8% of the chemical when treat at 180°C/60 min and above, the reduction was not significant. The hemicellulose content for treated and untreated bamboo were in the range 24-28%.

During heating, acetic acid is formed from aceylated hemicelluloses by hydrolysis. The released acid serves as a catalyst in the hydrolysis of hemicelluloses to soluble sugars. In addition, the acetic acid that has formed depolymerises the cellulose microfibrils in the amorphous area. The acid hydrolyses the bonds joining the units of glucose, breaking cellulose into shorter chains. However, the breaking of a hemicellulose chain does not reduce as much the strength of the bamboo as breaking of cellulose chains would do.

Tjeerdsma and Militz (2005), found hemicellulose hydrolysed more easily than cellulose. Dehydration reactions occur naturally during thermal treatments of polysaccharides, causing a decomposition of hydrolysed sugars (Fengel and Wegener, 1989). Pyranosidic ring structures will be dehydrated into hydroxymethylfurfural and furanosidic ring structures in furfural (Bobleter and Binder, 1980).

According to Abatzoglou *et al.* (1990), hemicellulose are easy to hydrolysed at high temperatures between 200 to 230°C even without the presence of an acid catalyst because of its structure and characteristics for example non-crystalline, highly disordered and heteropolymers (Ishii and Shimizu, 2001).

Boonstra and Tjeerdsma (2005), stated that the heat treatment at 180°C are less effective to depolymerise of hemicellulose however depolymerisation increased after temperature was raised to 185°C consequent degraded of hemicellulose content.

According to Tjeerdsma and Militz (2005), during thermal treatment carbonic acids mainly acetic acid will be formed as a result of cleavage of the acetyl groups of particular hemicelluloses (Bourgois and Guyonnet, 1988). Depending the on acid concentration and temperature applied, hemicellulose as the most reactive wood component will be hydrolysed into oligomeric and monomeric structures (Carrasco and Roy, 1992).

According to Garrote *et al.* (1999), depending on the conditions, polysaccharides (mainly hemicelluloses) are depolymerized to oligomers and monomers and the correspondent sugars (pentoses and hexoses) can be dehydrated to furfural and hydroxymethylfurfural, respectively.

Boonstra and Tjeerdsma (2005), indicated a depolymerization of carbohydrate mainly of the hemicellulose at higher temperature during hydro-thermolysis at the same time effect on the concentration of the soluble fraction because of the formation of furfural and some degradation of the lignin wood component.

Fengel and Wegener (1989), investigated that the acidic hydrolysis of hemicellulosic polysaccharides in wood is mainly a matter of breaking the glucosidic bonds (1→4) between monosaccharide units of the chain, the liberation of ester-bonded side groups such O-acetyl and the breaking of other ether-bonded side groups such as α-D-galactopyranose. This will lower the molecular size such as reduce the degree of polymerization for the polysaccharide and fragments of low molecular by products such as mono- and disaccharides will be solubilized.

3.1.3 Cellulose content

The cellulose content of heated and unheated bamboo is presented in Table 1. The reduction of cellulose from treatment at 140°C/60 min to 180°C/30 min was 2.9% to 3.8% respectively compared to control. This reduction further increased to 5.1% at 180°C/60 min and 14.1% at 220°C/60 min. This result shows that the degradation of bamboo cellulose increased when temperature and time of treatment increases.

Bamboo cellulose is considered to have major influence of bamboo strength. The bamboo cellulose structure is changed and that amorphous part can be degraded close to and above 220°C. The amorphous part of the bamboo cellulose possibly hydrolyzed first, leaving a residue of cellulose with reduced degree of polymerisation and increased crystallinity.

In a study of thermal treatment, degree of polymerisation of cellulose reduced and leveled of to values around 600 to 800 (Roffael and Schaller, 1971). Cellulose degradation can contribute to the loss of mechanical strength in wood under high temperature treatment (Sundqvist, 2004). Changes in the cellulose of wood during thermal have also been known as increase in crystallinity (Bhuiyan *et al*., 2000; Kubojima *et al*., 2001; Sivonen *et al*., 2002). Organic acids such as formic and acetic acid are liberated during the process which may affect the properties of wood (Risholm-Sundman *et al*., 1998; Garrote *et al*., 2001; Manninen *et al*., 2002).

Rubio *et al*. (1994), reported that cellulose degradation reactions started at an above 210-220°C and degree of degradation in the same way cellulose was significantly affected at temperature more than 270°C (Biermann *et al*., 1984). Different process conditions and treatment time applied during heat treatment may influence degradation rate of cellulose content (Boonstra and Tjeerdsma, 2005). Boonstra and Tjeerdsma, 2005 suggested that in hydrothermolysis treatment, (at 165-185°C), an effective treatment time of 30 minutes while in curing treatment, (at 170-180°C), an effective treatment time of 4 hours).

In hydrothermal at high temperatures, the heating time can be reasonably elongated in contrast with the duration of the isothermal reaction. This is important to a substantial hemicelullose alteration during heating (Carrasco & Roy, 1992).

3.1.4 Lignin content

The lignin content of bamboo that had been heat treated in a palm oil is displayed in Table 1. There is no specific trend of lignin when treated at different treatment conditions. From Table 1, it shows that lignin content decreased at treatment temperature 140°C-180°C however, it increased when the treatment temperature was raised to 220°C. At 180°C, lignin content was higher when it was heated longer. A higher lignin found in the treated bamboo compared to the control indicates that the degradation of some hemicelluloses. Furthermore, changes in structure of bamboo lignin (plasticization) at high temperature were probably attributed to the increment of lignin content.

Kamden *et al.* (2002), found that increases in lignin content can be attributed to the loss of hemicellulose or fragile pentoses and hexoses during the heat treatment. They also initiated that increase in lignin content does not involve the formation of lignin during the process but the reduction of other wood components. Boonstra and Tjeerdsma (2005), found a similar trend of lignin content during treatment process in *Picea abies*, *Pinus sylvestris* and *Pinus radiate* D. They also observed that the lignin content of heat-treated wood is increased mainly due to depolymerisation of the carbohydrates. Polycondensation reactions result in a further cross-linking of the lignin network also contributes to increase of the lignin content. The reduction of water adsorption favours the effect of increased cross-linking of the lignin network while the proportion of free hydroxyl groups still available after heat treatment.

Tjeerdsma *et al.* (1998); Boonstra and Tjeerdsma (2005), stated that the condensation reactions of lignin at 185°C, was probably contributed to higher lignin content and more hemicelluloses cleavage products.

According to Westermark *et al.* (1995), the changes in the lignin structure starts at temperature around 120°C in which the changes increased with the temperature. Kishimoto and Sano (2002), stated that lignin condensation was probably due to homolytic cleavage of ether bonds and subsequent rearrangement reactions. At temperature around 180°C, homolytic cleavage of β-ether linkages and formation of radicals condensation products and possible cross links between lignin and polysaccharides (Tjeerdsma *et al*., 1998b; Kosikova *et al.,* 1999; Sivonen *et al.,* 2002). Lai (1991), observed that 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 (Sivonen *et al.,* 2001).

Boonstra and Tjeerdsma (2005), reported a depolymerization of carbohydrate mainly of the hemicellulose at higher temperature during hydro-thermolysis at the same time effect on the concentration of the soluble fraction because of the formation of furfural and some degradation of the lignin wood component. They also reported that heat treatment at higher temperatures (185°C) result instead in a further decrease of water adsorption, in which decrease in free hydroxyl groups.

Tjeerdsma and Militz (2005), stated that lignin is the least reactive wood component, but at high temperatures, bonds within the lignin complex will be cleaved, resulting in a higher concentration of phenolic groups (Kolmann and Fengel, 1965). This state of increased reactivity of the lignin exposes the occurrence of various condensation reactions of aldehydes and autocondensation of lignin.

According to Kamdem *et al.* (2002), the chemical degradation of wood occurs in the order of hemicellulose, cellulose and lignin. A limited decomposition of lignin is observed at a temperature as low as 220°C with the presence of phenolic substances such as vanillin, coniferaldehyde and syringyl aldehyde (Sandermann and Augustin, 1964).

A partial depolymerization reported at 135°C for beech (Kosikova *et al.,* 1999) in addition, splitting of β-aryl ethers and formation of lignin condensation products at 100°-120°C was found for maple and hemolytic cleavage of phenolic β-aryl ether in wood lignin was found 130°C (Westermark *et al.,* 1995). At temperature of 150°-250°C, major changes

in the wood components occur. Both degradation and modification which thermal treatment in model systems was four times higher for hemicelulose than for cellulose and that the degradation rate for lignin was only half of that for cellulose at 150°C (Stamm, 1956).

3.1.5 Starch content

The mean starch content value in the bamboo culms is shown in Table 1. The starch content of heat-treated bamboo did not differ significantly between control and treatment conditions of 140°C/60 min. At 180°C/30 min the starch content was reduced by 22% to 3.2%, whilst at 180°C/60 min, it was reduced by 34% to 2.7%. A high reduction of starch content was demonstrated at 220°C/60 min. The value was reduced by 54% to 1.9%. The reduction of starch may probably hydrolysis of this component during heating. Removal starch from bamboo can improves the durability since fungus or pest depends on starch as a favourite food to survive.

Sundqvist, 2004 reported that pectin and starch has a similar molecular structure such as hemicellulose. He also stated that at 200°C, starch become hydrolysed under this conditions same with hemicellulose.

4. Conclusion

Holocellulose content of boil-treated bamboo did not differ to each other when it was heated at 180°C however, the content reduced significantly when treated at 220°C/60 min**.** Hemicellulose content of bamboo was not affected by the treatments. Cellulose content in bamboo was only reduced when it was treated at temperature higher than 180°C. At 140°C to 180°C, the lignin content in the treated bamboo decreased, but the chemical content value increased when the bamboo was heated at 220°C. Starch content in the bamboo was found to be inversely proportional to treatment temperatures.   At 180°C, duration of treatment did not significantly affect the starch content. The results indicated that degradation of cellulose and hemicellulose of heat-treated bamboo at higher temperature and time was attributed to plasticization of lignin during heating in the same time hydrolysed the starch content.

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Table 1. Mean chemical analyses of 3-years-old *G. scortechinii* culm after heating in palm oil at varies temperatures and times

Heating conditions	N	Holo-cellulose (%)	Hemi-cellulose (%)	Cellulose (%)	Lignin (%)	Starch (%)
Control	6	81.4 [a] (3.69)	26.2 [a] (3.58)	55.2 [a] (1.75)	22.3 [ab] (3.31)	4.1 [a] (0.18)
140°C/60	6	81.4 [a] (4.54) {0}	27.8 [a] (27.8) {6.21}	53.6 [ab] (2.01) {-2.9}	21.1 [b] (4.09) {-5.4}	4.0 [a] (0.32) {-2}
180°C/30	6	79.7 [a] (5.15) {-2.1}	26.6 [a] (4.96) {1.42}	53.1 [ab] (0.71) {-3.8}	21.6 [b] (2.98) {-3.1}	3.2 [b] (0.35) {-22}
180°C/60	6	76.5 [ab] (3.36) {-6}	24.1 [a] (2.14) {-7.98}	52.4 [b] (3.11) {-5.1}	22.0 [ab] (3.01) {-1.3}	2.8 [b] (0.42) {-34}
220°C/60	6	72.7 [b] (6.07) {-10.7}	25.3 [a] (6.26) {-3.63}	47.4 [c] (2.51) {-14.1}	26.0 [a] (3.94) {16.6}	1.9 [c] (0.20) {-54}

Note: Values in parentheses are standard deviations

     Means with the same letter are not significantly different (p<0.05)

    {   } = % change from control

   N = number of sample