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Chemical Constituents of Oil-Cured Tropical Bamboo *Gigantochloa scortechinii*

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Abstract: The chemical constituents of oil-cured 3 years-old tropical bamboo *Gigantochloa scortechinii* were investigated in this study. The bamboo splits were oil-cured using organic palm oil at temperature of 140, 180 and 220°C for duration of 30 and 60 min. The bamboo splits were then grinded into small particles and air-dried prior to the chemical analysis to obtain the compositions: holocellulose, hemicellulose, cellulose, lignin and starch. Untreated samples were used as control for comparison. The results obtained showed an overall reduction in the chemicals constituents after treatments compared to the control. Significant changes were however noted after the bamboo samples underwent treatment at temperature above 180°C. The holocellulose content decreased slightly from 81.4 to 79.7% for treatment conditions at 220°C for 30 min. On the other hand holocellulose content diminished significantly when the sample was treated at 180°C for 30 min and further treatment resulted in 72.7% holocellulose content at 220°C for 60 min treatment. The hemicellulose content of bamboo ranged from 24.1 to 27.8% when treated at 140 and 220°C for 30 to 60 min, respectively. The cellulose content of heat-treated samples ranged 47.4 to 55.2%. Starch contents were largely reduced from 4.1 to 1.9% for control to oil-cured samples at 220°C for 60 min.

Key words: Tropical bamboo, oil cured treatment, palm oil, chemical constituents, starch decreases

INTRODUCTION

Bamboo, considered as the best alternative to replace wood, grow naturally in most of the tropical countries. This fast growing species needs between 3-5 years to mature before they can be harvested and utilized for various purposes such as handicraft and furniture making. However, bamboo either in standing trees or in utilization is prone to fungi and insect attack. They deteriorate rapidly if are not treated with preservatives (Razak *et al*., 2007, 2004; 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 purposes. The uses of preservatives are known to have negative impact to the environment as some of the chemicals present quite harmful to both fauna and flora. Chemicals free treatments are being considered as an alternative.

The oil-curing treatment that has been use for a long time in South East Asia for treating rattan has been recommended by Razak *et al*. (2004, 2005, 2007) to enhance the bamboo durability. In Europe the oil treatment has been used for treating selected wood species (Ruyter, 1989; Leithoff and Peek, 1997).

However to obtain an effective oil treatment process, the temperature must be applied at above 170°C. At this temperature the durability of the bamboo can greatly be improved (Leithoff and Peek, 1997). The chemical composition in the treated woody material are modified at an elevated temperature (Boonstra and Tjeerdsma, 2006).

In the present study, focuses are given on the modification of the bamboo chemical constituents after undergoing the oil curing at 140, 180 and 220°C for 30 and 60 min of treatment.

MATERIALS AND METHODS

The study was conducted from Jan. 2004 to Dec. 2007 at University Malaysia Sabah (UMS), Forest Research Institute Malaysia (FRIM) and University Putra Malaysia (UPM).

Preparation of bamboo samples: Bamboo culms of 3 years old were extracted from randomly selected clumps in Nami, Kedah in Malaysia. The culms were cross-cut into portion of 50 cm in length and split into sizes of about 2 cm before conditioned at 20±2°C and 65% RH to obtain 12% moisture content.

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Heat treatment process: Palm oil (having a boiling point of 320°C) was poured into an oil-curing stainless steel. The oil was poured until it occupied about ¾ of the tank. The oil was then heated up until it reached a temperature of 80°C. Splits bamboo were placed in metallic cage were then immersed in the heated palm oils in the stainless steel tank. The temp of the oil was then increased gradually. The temp increments were controlled by a digital control panel. Three electric heaters were used generated heat from electricity power sources. Data logger connected to thermocouple where four and three channels were placed inside and outside (oil) of the bamboo splits. Data were recorded every 5-10°C interval. In this experiment, temperature was applied at 140, 180 and 220°C with duration 30 and 60 min. Bamboo samples were taken out once they reached targeted temp and treatment duration. Residual oil on splits bamboo surfaces were removed by wiping with cloth to avoid oil absorbance into the bamboo.

Chemical analysis

Preparation of sawdust: Sample of sizes 2 cm × 2 cm × the bamboo culm wall thickness, were cut from each of the heat-treated bamboo. The samples were grinded after the epidermis and the inner layers of bamboo were removed. The grinded particles were then filtered through a BS 40-60 mesh sieve. The particles were air dried for several days until it reached the constant weight prior to chemical analysis. This was done in accordance to the procedure developed by TAPPI Standards (Anonymous, 1974, 1988, 1993).

Moisture content of sawdust: The filtered particles placed in small weighing bottle (previously cleaned and dried in an oven) were weighed on an analytical balance. The air-dried particles of 2.0±0.01 g were placed in the weighing bottle. The bottles with the particles were then put in an oven set at 105°C for 3 h with the cover off. Then the bottle was removed and placed in the desiccator for 15 min for cooling process before reweighing. The moisture content on the basis of the air-dry weight was calculated by dividing the loss in weight by air-dry weight.

Holocellulose content: Air-dried particles of 2 g extractive-free were weighed accurately. The particles were transferred quantitatively to a 250 mL conical flask. One hundred milliliter water, 1.5 sodium chlorite and 5 mL of 10% acetic acid were added and the flasks were placed in a water bath maintained at 70°C, the content of the flask were swirled at least once every 5 min. The flasks were kept closed with a small, inverted Erlenmeyer flask. Five milliliter of 10% acetic acid were added after 30 min. 1.5 sodium chlorite were added after further 30 min. Alternative acetic acid and sodium chlorite at 30 min were added (with sodium chlorite being the last addition). The mixtures were heated for 30 min until the residue turned white and retaining the woody structure. The suspensions were cooled in an ice bath.

Residue were filtered into a weighed fruited glass crucible (medium or coarse porosity) and washed with iced distilled water and finally washed with acetone. The residues were air-dried (to allow the residue to strand in the open laboratory for a day or two until it is free of acetone). The glass crucibles were then covered with perforated aluminum foil. The samples were then transferred to a desiccator and weighed at daily intervals until they reached constant weight. The moisture contents were determined on a 0.5 g sample.

Cellulose content: The air-dried holocellulose prepared earlier were used for the cellulose study. The experiments were carried out in a water bath at 20°C. About 2 g of particles were weighed out accurately and transferred into a 20 mL beaker that were later placed in a water bath at 20°C. Fifteen milliliter of 17.5 NaOH was added and macerated gently with a flattened glass with rod for 1 min. Ten milliliter more NaOH was added and the solution was mixed for 45 sec. Then, 10 mL more was added and mixed for 15 sec so, that at the end of 2 min 35 mL of the NaOH have been added. The mixture was stirred and allowed to stand for another 3 min. After 3 min, another 10 mL NaOH was added and mixed with stirring rod in the solution for every 2.5 min for 4 times.

The beaker was covered with watch glass and the mixture was left in the water bath for 30 min 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 min. 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 min. 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.

Lignin content: The procedure was referred from TAPPI Standard T 222 os-74. One gram of air dried extractive free sawdust was weighed out accurately in weighing bottle and transferred in a 50 mL beaker. Ten milliliter 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 h), a crucible was oven dried (fine or medium porosity) for 1 h at 110°C, then allowed to cooled in a desiccator (15 min 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 h, cooled in a desiccator (15 min) and weighed. Lignin content was reported as reported as percentage by weight of the dried sample.

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.

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 h 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 min 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 0.5 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 min 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:

$$\text{Starch} = \frac{0.36778 \times (\text{E. reading} + 0.008)}{\text{Oven} - \text{dry weight of sample (g)}} \times \frac{50}{10} \times 100 \qquad (1)$$

Where:

E. reading = Differences of absorption between sample and blank

RESULTS AND DISCUSSION

Effects of oil-curing process on chemical properties of *G. scortechinii*: The hemicellulose content was calculated based on the deducted from holocellulose to alpha-cellulose (Table 1).

Holocellulose contents: The holocellulose content in the oil-cured bamboo decreases from 81.42 to 72.74% in the control and treated bamboo at 220°C for 60 min respectively. The changes in the holocellulose content occurred slightly from the control bamboo to temperature of 180°C for 60 min (81.42 to 79.72%). Changes in the contents occurred significantly from 180°C for 60 min to 220°C for 60 min. The reduction of holocellulose at higher temperature (above 180°C) was explained by the depolymerisation of the hemicelluloses and some degradation of the cellulose due to heating (Boonstra and Tjeerdsma, 2006).

Hemicellulose contents: The hemicelluloses content decreases slightly from the control to oil-cured bamboo. However, there were no significant differences in the changes observed. The hemicellulose content for control and treated bamboo were in the range 24-28%. The oil-curing process at temperature below 180°C are less effective to depolymerise of hemicelluloses (Boonstra and Tjeerdsma, 2006). Depolymerisation of the hemicellulose increased after temperature were raised to 185°C consequent degraded of hemicellulose content. Hemicellulose are easy to be hydrolysed at high

Table 1: Mean chemical analysis of 3 years old *G. scortechinii* culm after heating in palm oil at 140, 180 and 220°C at 30 and 60 min durations

Oil-curing conditions	Holocellulose (%)	Hemicellulose (%)	Cellulose (%)	Lignin (%)	Starch (%)
Control	81.4a (3.69)	26.2a (3.58)	55.2a (1.75)	22.3ab (3.31)	4.1a (0.18)
140°C for 30 min	80.7a (4.21)	27.2a (2.23)	54.2ab (2.01)	24.23ab (3.04)	4.0a (0.37)
140°C for 60 min	81.4a (4.54)	27.8a (2.78)	53.6ab (2.01)	21.1b (4.09)	4.0a (0.32)
180°C for 30 min	79.7a (5.15)	26.6a (4.96)	53.1ab (0.71)	21.6b (2.98)	3.2b (0.35)
180°C for 60 min	76.5ab (3.36)	24.1a (2.14)	52.4b (3.11)	22.0ab (3.01)	2.8b (0.42)
220°C for 30 min	74.7b (4.76)	25.43a (5.64)	49.5c (2.73)	23.67 (2.95)	2.4c (0.36)
220°C for 60 min	72.7b (6.07)	25.3a (6.26)	47.4c (2.51)	26.1a (3.94)	1.9c (0.20)

Values in parentheses are SD. Means with the same letter(s) are not significantly different ($p<0.05$). Values listed are mean of 6 replicates

temperatures of 200 to 230°C because of its structure and characteristics being non-crystalline, highly disordered and hetero-polymers.

According to Tjeerdsma and Militz (2005), during thermal treatment carbonic acids mainly acetic acid were 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).

Boonstra and Tjeerdsma (2006) 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.

Cellulose contents: The reduction of cellulose contents occurred at 140°C for 60 min to 180°C for 30 min was 2.9 to 3.8%, respectively compared to control. This reduction further increased to 5.1% at 180°C for 60 min and 14.1% at 220°C for 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 off 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*., 2000; 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, 2006). Boonstra and Tjeerdsma (2006) suggested that in hydrothermolysis treatment, (at 165-185°C), an effective treatment time of 30 min while, in curing treatment, (at 170-180°C), an effective treatment time of 4 h.

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 hemicellulose alteration during heating (Carrasco and Roy, 1992).

Lignin contents: There is no specific trend in the decreases of the lignin content in the oil-cured bamboo. The lignin content decreased at treatment temperature 140-180°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 (2006) 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) and Boonstra and Tjeerdsma (2006) 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. The 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*., 1998; 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 (2006) 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 (Kollman 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 hemicellulose than for cellulose and that the degradation rate for lignin was only half of that for cellulose at 150°C (Stamm, 1956).

Starch contents: The starch content decreases from 4.16 to 1.93% in the control and oil-cured bamboo at 220°C for 60 min. The significant differences only occurred once the bamboo are oil-cured at temperature 180°C for 30 min and beyond. The starch content although, decreases slightly between the control and bamboos oil-cured at 140°C for 30 min and 140°C for 60 min but these were not significant. High reduction of starch content was observed when the bamboo were oil-cured at temperature 220°C at 30 and 60 min treatment durations. 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) stated that at 200°C, the starch becomes hydrolyzed.

CONCLUSIONS

Significant changes in the oil-cured bamboo occurred at temp of 180°C either at 30 or 60 min treatment durations. The following main conclusions can be drawn from the study:

- The holocellulose contents of bamboo *Gigantochloa scortechinii* decreases when they were treated in the oil-curing process. The content changed from 81.4% in control bamboo to 72.7% in bamboo treated at 220°C for 60 min. Rapid changes in the holocellulose content occurred at higher temperatures
- The hemicellulose contents of the bamboo remained stable throughout the treatment process. No significant different in the content were observed in the study
- The cellulose contents in the bamboo changes with temperatures. They changed from 55.2 to 47.4%
- The lignin contents changes from 22.3% in control bamboo to 26.1% when oil-cured at 220°C for 60 min
- The starch contents decreases from 4.1 to 1.9% in control and treated bamboo at 220°C for 60 min, respectively.

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