Physical and Mechanical Properties of Thermally Modified Kauayan-Tinik (*Bambusa blumena* Schltes f.)

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Abstract

The project investigated the effects of thermal modification using high temperature steam in a closed cylindrical vessel on the physico-mechanical properties of 60 cm long quarter-cut kauayan-tinik culm samples. Three temperature levels (150, 175 and 200°C) and two treatment durations (30 and 60 min) were used to modify kauayan-tinik. Two-factor factorial experiments and simple CRD were used to analyze the data.

Results showed that kauayan-tinik properties were altered with exposure to high temperature and long treatment duration. Bamboo color changed from light yellowish brown to dark brown with increasing treatment temperature. No defects due to thermal modification were observed. Treatments resulted in improved dimensional stability as indicated by reduction in equilibrium moisture content (EMC), thickness (radial) and width (tangential) swelling of thermally modified kauayan-tinik. Mechanical properties as measured by modulus of rupture (MOR) and modulus of elasticity (MOE) apparently increased until the temperature of 175°C then decreased significantly at 200°C.

1. Introduction

Bamboo is one of the most important non-timber forest products in the Philippines. Being a very versatile material, it can be made into various designs of furniture, handicrafts and laminated items such as floor tiles and cutting boards. However, like wood, bamboo has its drawbacks. It is dimensionally unstable and susceptible to attack of biodegrading agents such as fungi and insects. Attack of molds, fungi and powder post beetles can occur on bamboo before and after processing into finished products [Giron and Garcia 2005].

Wood biodeterioration is controlled or prevented through the application of prophylactic and preservative chemicals. However, advanced countries are now limiting if not totally banning the use of toxic and harmful chemicals to products that can come in contact to humans; and, furniture and handicrafts are no exceptions.

In Europe, one of the most popular non-chemical treatment methods of making wood dimensionally stable and at the same time preserving it is through thermal modification (TM) or heat treatment at very high temperatures. Commercial names of the wood from this process are: **ThermoWood** from Finland, *Plato-Wood* from Netherlands, *Retified Wood* from France and *Menz Holz (Oil heat treated wood)* from Germany [Rapp, A.O. 2001]. In Asia, Malaysia has *Stellac Wood* which is also from Finland. These TM treatments have been proven as viable methods for protecting wood against biodeterioration aside from improving color and dimensional stability.

Thermal modification is the process of subjecting solid wood close to or above 200^{0}C for several hours in an atmosphere with low oxygen content [Rapp, A.O. 2001]. This condition is achieved through the use of water steam, nitrogen, or oil, which replaces the oxygen inside the treatment chamber to as low as 3.5% [Syrjanen, T. and K. Oy 2001]. The low oxygen content prevents the wood from burning at very high temperatures and at the same time influences changes on the natural properties of wood [Finnish Thermowood Assocation FTA. 2003]. This process alters the physical, mechanical and chemical properties of the wood. Depending on the treatment time and temperature employed, wood properties can be tailored to specific end uses. Among the desired properties are the increase resistance to biodeterioration, dimensional stability of the wood and color change.

Non-wood materials like bamboo have been thermally modified in Japan using the EDS [EDS Manual and pamphlet. 2003] process to improve resistance against fungal decay and insects damage. In Germany [Leithoff, H. and R.D. Peek. 2001], heat treatment of bamboo in a hot oil bath of hemp seed oil using the temperatures of 180, 200 and 220^{0}C and duration of 30, 60 and 120 minutes was carried out. Results showed that the application of temperatures above 200^{0}C caused a clearly enhanced durability against a soft rot fungi but the shock resistance was intensely reduced.

In Malaysia, a study [Wahab, R., et al. 2007] using different treatment temperatures of 140, 180 and 220^{0}C and durations of 30, 60 and 90 minutes showed that oil heat-treated bamboo had improved durability in ground contact tests although with negative but acceptable change in strength properties conducted based on EN 252:1989 and ISO 22157: 2004 standards. In the Philippines, [Manalo, R.D. 2007] a TM of three bamboo species by boiling in grade B virgin coconut oil at temperatures of 160, 180, and 200^{0}C and durations of 30, 60 and 120 minutes resulted to slight improvement of fungal-decay resistance at 180^{0}C for 60 to 120 minutes duration. Above 180^{0}C, considerable loss in bending strength of the bamboos was observed [Manalo, R.D. 2007].

In China, a similar TM process called *carbonization* is used to modify properties of Moso bamboo for flooring and cutting board manufacture. Pressure heating, boiling, or steam heating at very high temperature results to a bamboo with caramel color and brown grain [Peterson, M. 2007]. This process weakens the strength of bamboo flooring from 20 to 30% but improved the dimensional stability and decay resistance, which are important to the said products. The exact process parameters of how carbonization in industrial scale is done seem to be a trade secret for manufacturers of bamboo flooring and cutting boards.

At FPRDI, pioneering studies on high temperature treatment to improve the durability of wood was made [Mailum, N.P. and C.V. Areanas. 1974]. Five (5) species of Philippine woods (rain tree, bagtikan, guijo, mayapis and palosapis) were subjected to heat treatment for a constant duration at various temperature levels from 90^{0}C to 175^{0}C using a laboratory oven with pure dry air. Results showed increased decay resistance of the 5 species against white rot fungi (*Fomes lividus*) and brown rot fungi (*Lenzites striata*) from 130^{0}C to 175^{0}C. Another study at FPRDI on TM using malapapaya (*Polyscias nodosa*) wood resulted to improved dimensional stability and durability [Jimenez, Jr. P. et al. 2001].

Since TM is proven effective to wood, this may also be effective and advantageous in bamboo processing. Hence, it is imperative to determine the technical feasibility of thermally modifying bamboo and examine the effects of the process on its physico-mechanical properties.

2. Methodology

2.1 Equipment

The steaming cylinder for wood bending was used as TM chamber for bamboo. Installed auxiliary electric heaters inside the cylinder was used to elevate the steam temperature to the desired treatment temperature.

2.2 Raw materials

Partially air-dried kauayan-tinik poles were acquired. The specimens were cut to a length of 60 cm from kauayan tinik culms consisting of basal and middle portions. These were split quarterly, labeled and randomly assigned to each treatment combinations including the control. Twelve (12) pieces each of quartered samples were assigned to the control and to the various treatment combinations. The control samples were kiln dried to moisture content (MC) of 8% to approximate the MC of thermally modified samples.

2.3 Procedure

2.3.1 Thermal modification process

The TM process was done in several batches based on combinations of temperatures and treatment durations. Temperatures used were 150^{0}C, 175^{0}C, and 200^{0}C while durations were 30 and 60 minutes. Real time treatment at nominal temperature started when the desired temperature of the treatment cylinder was reached.

During the TM process, water vapor or steam was continuously injected in the treatment cylinder. Steam injected in the treatment cylinder was used to build up pressure up to 15 psi. Excess pressure was released through the manual pressure release valve. After the treatment, the samples were kept to cool down inside the cylinder and then unloaded when the temperature went down to 30ºC.

2.3.2 Physical and mechanical properties tests

Prior to testing, the thermally treated samples together with the kiln dried control samples were conditioned for six months in a room with an average temperature of 21ºC, RH of 50 - 55% and EMC of approximately 10%.

The physical characteristics (weight loss, MC, EMC, relative density, and swelling/dimensional stability test) of the thermally modified samples were tested using ASTM (1995) procedure with slight modification. The static bending test was performed using a computer controlled Universal Testing Machine.

2.4 Experimental design

A 2-factor factorial in Completely Randomized Design (CRD) was used for the analyses of data on the effects of each treatment on the physico-mechanical properties of thermally modified bamboo. There were six replicates for each treatment (Table 1).

Table 1. Treatment combinations and number of replicates per treatment

Treatment No.	Treatment Combinations		Replicates per Treatment
	Temp (^{0}C)	Duration (min)	
1	150	30	6
2	150	60	6
3	175	30	6
4	175	60	6

5	200	30	6
6	200	60	6
7	Control – Untreated samples		6

3. Results and Discussion

The thermal modification of kauayan tinik samples resulted to significant variation of its physico-mechanical properties. In terms of physical attributes, the TM samples have darker color tones, lower relative density, and higher dimensional stability at higher temperatures and longer treatment duration. On the other hand, there is a reduction of bending strength at the highest temperature level and treatment duration.

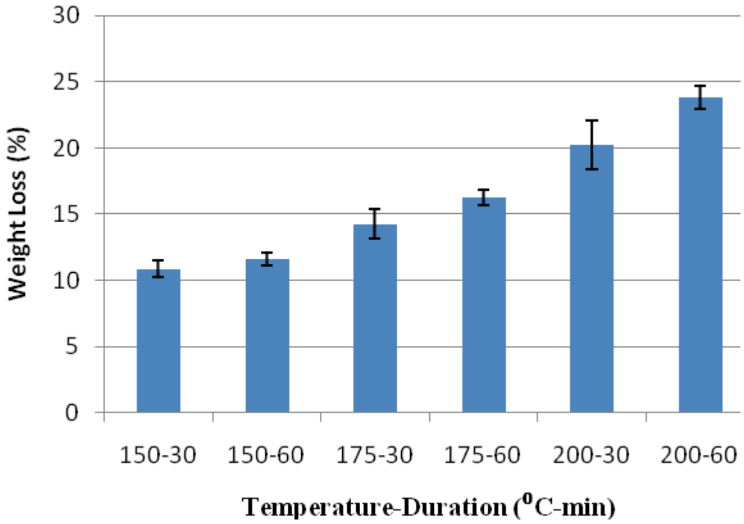
3.1 Color Change

One of the notable changes in the quarter-cut bamboo samples subjected to TM process was the gradual change in color from light to darker shades of brown (Fig. 1). Both the rind and inner side of bamboo turned to darker brown tones as the treatment temperature (150 to 200ºC) and duration (30 to 60 min) increased.

The change in color can be attributed to the complex chemical reaction that occurs when bamboo cell wall components i.e. hemicelluloses, lignin and extractives are heated to elevated temperatures [Sundqvist, B. 2004; Bekhta, P. and Niemz P. 2003; Sehistedt-Persson, M. 2003; Esteves, B.M., et al. 2008; Bremer, M., et al. 2013]. Research done [Nguyen, T.C., et al. 2012] on the thermal modification of 2 Vietnamese bamboo species reported that color difference is influenced more by temperature level rather than duration of treatment.

3.2 Weight Loss

Figure 2 shows the trend on the weight loss of bamboo samples in relation to TM variables. There is an increasing weight loss of the thermally modified bamboo samples as the level of temperature and treatment duration increased. The loss in weight of the samples is not only due to the loss of moisture during the heat treatment. It also involved evaporation of volatile compounds, degradation of cell wall structural components i.e hemicelluloses, amorphous regions of cellulose and demethoxylation and depolymerization of lignin [Bremer, M.S. et al. 2013; Sundqvist, B. 2004; Nuopponen, M., et al. 2002]. Portions of the hemicelluloses are hydrolyzed into monosaccharides or simple sugar components such as glucose, galactose, mannose, arabinose and xylose. The amorphous regions of the cellulose are also broken down into shorter chains by hydrolysis during the TM process.



3.3 Equilibrium Moisture Content (EMC)

The MC of thermally modified (TM) bamboo was lower compared to the MC of the KD control sample. The MC of TM bamboo ranged from 4.31 to 6.59% while the KD bamboo had 7.67% (Table 2). After conditioning of the control and TM samples for 6 months, the MC changed because the room condition to which the samples were exposed had an average EMC of 10%. Thus, having the EMC higher than the MC of the KD control sample and TM samples and being a hygroscopic material, they absorbed moisture from the air.

Table 2. Comparative MC after treatment, after conditioning and change in EMC of the treated samples relative to control

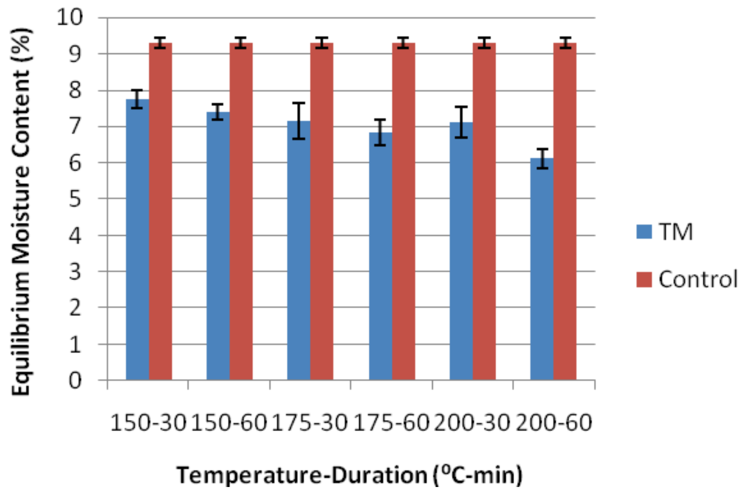
Treatment °C-min	MC (%) after Treatment	EMC (%) after Conditioning for 6 months	Change in EMC
Control	7.67*	9.30 *(0.14)* a	-
150-30	6.59	7.75 *(0.23)* b	1.55
150-60	6.52	7.39 *(0.21)* b	1.91
175-30	5.96	7.15 *(0.49)* b	2.15
175-60	5.36	6.84 *(0.34)* bc	2.46
200-30	5.78	7.12 *(0.42)* b	2.18
200-60	4.31	6.12 *(0.26)* c	3.18
ANOVA		F_{com} = 9.49 df = 6 P < 0.0001	

*Control's MC of 7.67% was obtained after kiln drying. Each mean is the average of six replicates;

means followed by the same letter are not significantly different with each other using DMRT at $\alpha = 0.05$. Italicized value in parenthesis is the standard error. F_{com} = F computed; df = degrees of freedom

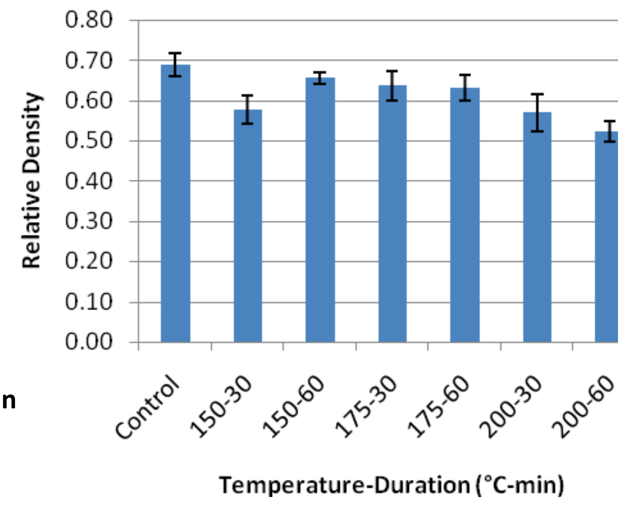
As presumed, results show that the EMC of the thermally modified bamboo is lower than the EMC of the KD control sample (Fig. 3). As temperature level and treatment duration increased, the degree of change in EMC also increased. The control's EMC was significantly different from the treated samples ($p < 0.0001$). The lowest EMC change was observed at 150-30 with 1.55% change in EMC and the highest change was observed at 200-60 with 3.18% (Table 2). This finding is similar to other researches on bamboo thermal modification involving hot oil [Salim, R., et al. 2010; Nguyen,T.C., et al. 2012].

The chemical modification of bamboo cell wall components during heat treatment caused the changes in EMC level. The hemicelluloses were degraded while the amorphous regions of the cellulose are hydrolyzed breaking it to shorter chains with reduced free hydroxyl groups. TM samples subjected to higher temperatures have lower free hydroxyl groups; thus, less hygroscopic or have lower EMC.



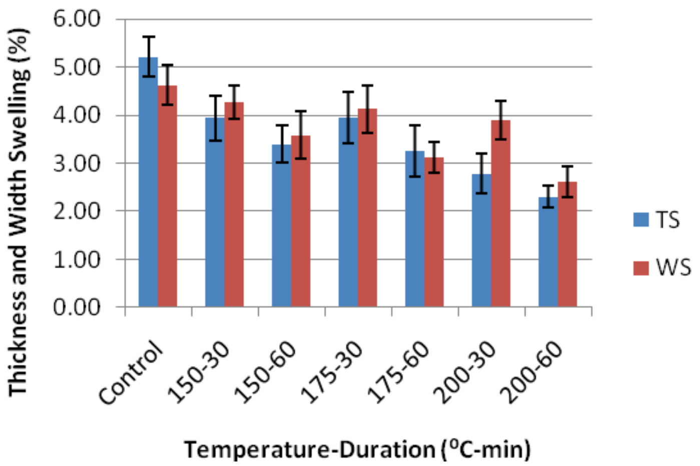
3.4 Relative Density

The relative density (RD) of the control samples was found to be higher than the RD of the treated samples (Fig. 4) and was found to be significantly different particularly to those treated at 200ºC ($p = 0.0144$). This can be attributed to the mass loss of the treated samples due to decomposition of hemicelluloses at very high temperature [Bremer, M., et al. 2013]. The significantly lower RD of the treated samples at 150-30 might be attributed to unintentional error in the selection of samples used in the project. Some replicates of test samples might have inferior inherent quality such as immature cells due to younger age class compared to those assigned to other treatment combinations. Nevertheless, a decreasing trend of RD in other TM samples was observed with increasing temperature level and treatment duration.



3.5 Thickness and Width Swelling

Figure 5 shows the comparison of the thickness (radial) and width (tangential) swelling of the control and TM bamboo samples after 24-hour soaking in water.



Both the control and TM have practically zero moisture before soaking because these were ovendried at 103±2ºC. It was very evident that there was a significant difference in the thickness swelling ($p = 0.0014$) and width swelling ($p = 0.0187$) of the control and TM samples. As seen in Fig. 5, the TM samples are more dimensionally stable than the KD control sample both in the thickness and width swelling. Generally, as the temperature level and treatment duration increased, lower swelling or higher dimensional stability of the thermally modified bamboo was observed. Similar results were obtained by other researchers [Manalo, D.R. and M.N. Acda 2009] on their study using hot oil to thermally modify bamboo.

The lower affinity to water or less hygroscopic structure of TM samples due to decomposition of hemicelluloses and reduction of free hydroxyl groups in the amorphous regions of the cellulose as mentioned earlier, also caused the lower swelling of samples thermally modified at higher temperatures. High dimensional stability, in wood or bamboo, is very important in their utilization for joinery and laminated products. Loosening of joints and premature delamination of glued products are associated to raw materials with high swelling and shrinkage when used or exposed to conditions with fluctuating humidity and temperature.

3.6 Static Bending

3.6.1 Modulus of elasticity (MOE)

Thermally treated samples MOE varies from 3570 MPa (200-60) to 5182 MPa (175-60) (Table 3). Relative to control (4966 MPa), treatments resulted to a negative change in MOE by 11-28% and to positive change in MOE by 2-4%. ANOVA shows that, this change in the MOE was significantly different. The samples treated at the highest temperature and duration (200-60) was significantly different from the control and had the lowest MOE value. All other combinations, except for the MOE of 150-30, were not significantly different from the control. For the MOE value in the 150-30 combination, the significantly different value (3672 MPa) from the control was attributed to its low relative density compared to control and other treatments. Some replicates of samples that were randomly assigned to this treatment might have inherent inferior quality as explained earlier hence, resulting to significantly lower value of MOE.

The decrease in MOE due to increased temperature level and treatment duration was also observed by other researchers [Manalo, R.D. and M.N. Acda, 2009] who reported a 16-22% decrease in MOE for the 3 bamboo species they studied.

Table 3. Mechanical Properties of Thermally Modified Kauayan-tinik

Treatment °C-min	Modulus of Elasticity (MPa)	Modulus of Rupture (MPa)
Control	4966 *(243)* a	49.9 *(3.3)* ab
150-30	3672 *(399)* b	34.8 *(5.0)* bc
150-60	5058 *(464)* a	47.8 *(4.5)* ab
175-30	4385 *(567)* ab	40.8 *(8.9)* abc
175-60	5182 *(429)* a	53.3 *(5.5)* a
200-30	4406 *(369)* ab	37.9 *(6.7)* abc
200-60	3570 *(315)* b	24.0 *(2.3)* c
ANOVA	F_{com}= 2.54 df = 6 P = 0.038	F_{com}= 3.31 df = 6 P = 0.011

Each mean is the average of six replicates; means followed by the same letter are not significantly different with each other using DMRT at $\alpha = 0.05$. Italicized value in parenthesis is the standard error. F_{com} = F computed; df = degrees of freedom

3.6.2 Modulus of rupture (MOR)

Among the treated samples, it appears that only the one treated at 200ºC-60 min (24.0 MPa) combination had the significantly different MOR from the control (49.9 MPa). All other values of MOR in other treatment combinations were not significantly different from the control (Table 3). Relative to control, MOR changed negatively from 4.9% (150-60) to 51.9% (200-60). Positive change was observed to 175-60 by 6.7% though not significantly different from the control.

The insignificant difference of values of MOR obtained from 150-30 to 200-30 might be attributed to some variation in the samples because these were cut from mixture of basal and middle portions of the culm and taken from various clumps. However, it can be seen that even the samples were mixed, the change in MOR value was still detected at the highest treatment combination (200-60). Compared to the results of other researchers [Manalo, R.D. and M.N. Acda. 2009] who obtained a MOR reduction of 31-60%, the reduction of MOR in this study is lower (5 to 52%).

4. Summary and Conclusion

Thermal modification of Kauyan tinik resulted to:

- Darker color of samples as temperature level and duration of treatment increased.
- Increased weight loss (10.8 to 23.8%) as temperature and duration of treatment increased

- Decreased relative density (5.1 to 24.1%) as degree of temperature and duration of treatment increased which could be attributed to weight loss due to degradation of hemicelluloses
- Decreased hygroscopicity (1.6 to 3.2%) as shown by the lower EMC values of the treated samples compared to control.
- Improved dimensional stability as shown by the reduced thickness (radial) swelling (24.4 to 55.9%) and width (tangential) swelling (7.5 to 43.4%) of the treated samples.
- Decreased MOE by 11.3 to 28.1% and MOR by 4.9 to 51.9%.

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