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Original Article

Effect of pre-treatment and heat treatment on tensile and thermal behavior of Parawood strands

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Abstract

This study was carried out to investigate the effect of pre-treatment and heat treatment at varied temperature and time on tensile and thermal behavior of Parawood strands. A three-way factorial experiment was employed with ten replications. The factors are (1) pre-treatment conditions of three levels (oven-dried strand, EMC strand, boiled strand), (2) temperature of heat treatment of four levels (170°C, 180°C, 190°C, 200°C) and (3) time of heat treatment of four levels (15 min, 30 min, 45 min, 60 min). The specimens prepared from the heat-treated strands were tested in tension parallel to grain and analyzed using thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC). Moreover, the specimens were examined using scanning electron microscopy (SEM) to reveal its internal structure. The results indicated that the Parawood strands subjected to the pre-treatment by boiling in water (100°C) for 30 minutes and subsequently heat treated in a hot air oven at 190°C for 30 minutes yielded highest tensile strength of 57 MPa and highest toughness of 0.75 MPa. The resulted tensile strength and toughness of boiling pre-treated strands are about 30 and 79 percent higher than those of the oven-dried pre-treated strands, respectively. The specimens subjected to the aforementioned treatment also required highest capacity of endothermic heat of 215 J/g for decomposing its modified crystalline structure. This study suggested that the pre-treatment by boiling in water for 30 minutes, followed by heat treatment at 190°C for 30 minutes, could increase tensile strength and toughness of Parawood strands by increasing its modified crystalline regions (a cellulose crystalline region reinforced by hygrothermally modified lignin and hemicellulose molecules).

Keywords: heat treatment, tensile strength, parawood strand, TGA/DSC analyses

1. Introduction

A strand is the main component of several types of wood-composite products, i.e., oriented strand board (OSB), oriented strand lumber (OSL), Laminated strand lumber (LSL), and parallel strand lumber (PSL). A strand is defined as a thin, narrow and long particle produced from wood bolts by using a flaking or stranding machine. It is typically of dimensions 0.635 mm (0.025 inch) thick, 15-25 mm wide and 70-500 mm long (ICBO, 1997). Due to the suitable configuration in transferring loads, a strand thus contributes a lot in improving strength of these wood composites.

A Pararubber tree (*Hevea brasilliensis* Muell. Arg.) is one of the economic species widely planted in Thailand for latex production, especially in the rain-forest in the South. However, the (replanting period) is (typically) only about 25 years. When latex tapping turns uneconomical, the trees are

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felled for replanting and the bolts become the main raw materials for wood industries in spite of their inferior quality. The bolt is usually tapered and crooked and contains a mixture of straight and interlocking grains. The wood is susceptible to insects and fungi attack and inherits a great deal of growth and tapping defects. Quality improvement is undoubtedly essential for the utilization of Parawood.

Heat treatment is one of the environmental-friendly alternative methods for improving wood quality that appeals recently to researchers. Several investigations on solid wood and wood composites found that proper heat treatment could improve its durability and dimensional stability (Militz, 2002; Evans, 2003; Paul *et al.*, 2005; Boonstra *et al.*, 2006). A report on application of heat treatment to wood strands indicated that high temperature treatments (200-260°C) of wood strands resulted in 20% reductions of modulus of rupture and modulus of elasticity (Goroyias *et al.*, 2002). However, several papers reported that heat treatment reduces wood strength, especially tensile and bending strengths, as well as toughness (Bengtsson *et al.*, 2002; Kubojima *et al.*, 2004; Boonstra *et al.*, 2007). An increase of compressive strength parallel-to-grain and modulus of elasticity due to heat treatment were also reported (Boonstra *et al.*, 2007).

Some researchers later modified heat treatment methods by introducing pre-treatment and post-treatment procedures. Recent works reported that heat treatment of highly moist wood sawdust with temperature above 150°C significantly increased the crystallinity of wood (Bhuiyan *et al.*, 2000). On the other hand, heat treated samples following by post-treating in water for one day or in boiling water for one hour yielded no significant change of crystallinity (Bhuiyan *et al.*, 2005). Moreover, Andersson *et al.* (2005) applied heat treatment with steam to Scots pine samples and found that crystallinity increased with temperature above 150°C. These findings suggested that application of moisture or hygrothermal pre-treatments to specimens before heat treatment might be able to improve wood strength since crystalline regions are the main constituents contributing to wood strength. It is therefore the purpose of this study to investigate whether application of a pre-treatment prior to the heat treatment procedure could affect the tensile and thermal behavior of the wood strands.

2. Materials and Methods

Branches of a Pararubber tree were adopted for this study since strands could be machined from the small-diameter residues left in replanting areas. The branches of diameter from 60 to 150 millimeters were randomly removed from several Pararubber trees of RRIM-600 variety (Rubber Research Institute, 2002) and about 25-year of age grown in the plantation in Thasala District, Nakhon Si Thammarat Province, Southern Thailand. Each branch was cross cut into 140-mm long bolts limited by the width of the flaker feed-opening (Figure 1-a) as well as sawn into samples for determining moisture content and specific gravity. Each bolt was

(a)

(b)

(c)

Figure 1. Preparation of test specimens from Parawood bolts: (a) Debarked Parawood bolts; (b) Typical Parawood strands; and (c) Specimens for tension test.

then machined into strands by using a CAE 6/36 Laboratory Disc Flaker having conditions set to obtain a target strand dimensions of 0.6-mm thick, 20-mm wide and 140-mm long (Figure 1-b). The green strands were sorted to obtain uniform dimensions by passing through the 12.5 by 12.5-mm sieve of a Gilson screen.

The experiment was carried out using a three-way factorial design assigning the three factors and corresponding levels as follows: Pre-treatment conditions having three levels (oven-dried strand, EMC strand, boiled strand); Temperature of heat treatment having four levels (170°C, 180°C, 190°C, 200°C); and Time of heat treatment having four levels (15 min, 30 min, 45 min, 60 min). By using 10 replications, the treatment combination equals 3×4×4×10 or 480 experimental units.

The prepared strands were divided into three batches for subjecting to the three controlled conditions of the pre-treatment procedures. To obtain the "oven-dried strands", one batch of the green strands was placed in an electric oven maintained at 103±2°C until constant weight was attained. The heated strands were left to be cooled down in a dessicator with dehydrated silica gel. The second batch of green strands was placed in a climate conditioned room maintained at 20°C and 65 % relative humidity until constant weight was likewise attained. Under this condition, the moisture content of the specimens was in equilibrium to the controlled climate,

which was 12 percent. This moisture content is defined as the equilibrium moisture content (EMC) of the specimens and thus this group of specimens was designated as the "EMC strands". The third batch of green strands was dipped in cold water (28±1°C) in a cooking vat and then heated until boiling (100±2°C) for 30 minutes in order to obtain the "boiled strands". These three batches of the pre-treated strands were subsequently subjected to heat treatment in a hot-air oven at four levels of temperature and four levels of heating time according to the designed factorial experiment. All heat-treated strands were prepared into test specimens (Figure 1-c) which were later tested in tension parallel-to-grain via a universal testing machine (Lloyd 5 kN) and according to the procedure in ASTM standard D1822L (ASTM, 2001).

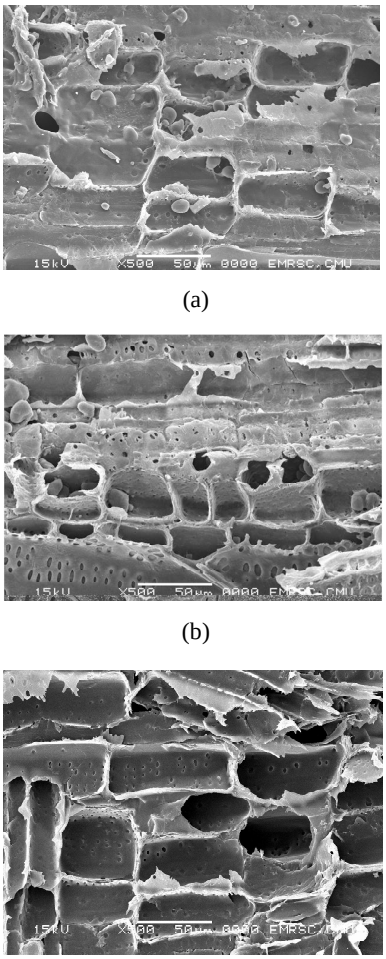
In order to verify the mechanism of tensile failure, the heat-treated strands were accordingly analyzed using thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) (TA Instruments Offices, 2001) to quantify the degree of cellulose crystallinity and examined using scanning electron microscopy (SEM) to reveal the internal structure. Each run of the thermogravimetric and calorimetric analyses was performed using 20 grams of granular sample prepared from the heat treated wood strand for placing in the ceramic crucible of the furnace. The TGA/DSC profiles were continuously recorded from ambient temperature (30°C) up to 600°C. The collected data were finally analyzed by a Statistical Analysis System (SAS) computer package in order to determine the level of significance of the treatment comparison.

3. Results and Discussions

Results of the examination by a scanning electron microscope revealed the internal structure of the heat-treated Parawood strands as shown in Figure 2. It is obvious that boiling pre-treatment removes most of the extractives included in the cell cavities resulting in a clean structure comparing to the oven-dried and EMC pre-treatments.

The results of mechanical tests revealed a typical load and extension diagram of the heat-treated strand subjected to tension parallel-to-grain as shown in Figure 3. The diagram obviously indicates elastic behavior and yields the maximum load for calculating ultimate tensile stress as well as the area under the curve up to failure for computing work to maximum load (area under the curve in N.mm divided by loaded volume of specimen in mm^3 equals N/mm^2 or MPa) which represents toughness. The resulted mean values of ten replications as well as the corresponding moisture content and specific gravity at test are presented in Table 1. In addition, the TGA/DSC analyses yield typical curves as depicted in Figures 4, 5 and 6.

For thermal analysis, Figures 4, 5, and 6 illustrate two typical curves, i.e., the TGA curve (dotted line) and the DSC curve (solid line) which represent weight loss and heat flow, respectively. These two curves together describe the behavior of the samples (heat-treated strands) subjected to heat



(a) (b) (c)

Figure 2. Internal structure of the Parawood strands subjected to different conditions of pre-treatment : (a) Oven-dried strand; (b) EMC strand; and (c) Boiled strand.

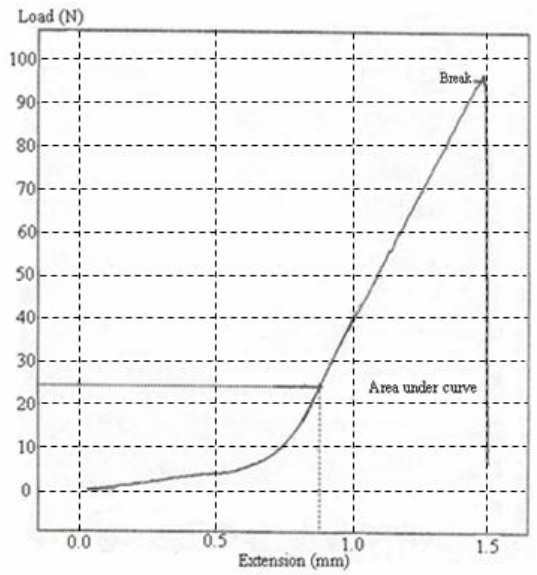


Figure 3. A typical load-extension diagram of a heat-treated Parawood strand tested in Tension parallel to grain.

Table 1. Values of moisture content at test, specific gravity at test, ultimate tensile stress parallel to grain, work to maximum load and endothermic heat capacity of oven-dried strand, EMC strand, and boiled strand under heat-treatment at 170, 180, 190 and 200°C for 15, 30, 45 and 60 minutes (average values for 10 replications).

Temperature (°C)	Time (min)	Oven-dried strand					EMC strand					Boiled strand				
		MC %	SG	σ_{tll} (MPa)	W (MPa)	Heat (J/g)	MC %	SG	σ_{tll} (MPa)	W (MPa)	Heat (J/g)	MC %	SG	σ_{tll} (MPa)	W (MPa)	Heat (J/g)
170	15	11.58	0.54	41.18	0.34	39.62	11.53	0.59	45.61	0.43	69.22	11.55	0.59	49.94	0.49	90.17
	30	11.58	0.54	41.53	0.35	39.89	11.56	0.57	46.51	0.49	65.02	11.54	0.57	42.67	0.40	90.65
	45	11.62	0.54	41.34	0.38	40.13	11.61	0.56	48.86	0.50	68.20	11.54	0.55	41.38	0.39	95.23
	60	11.54	0.57	40.78	0.38	40.33	11.55	0.55	39.86	0.35	66.85	11.46	0.55	40.26	0.33	91.47
180	15	11.55	0.55	40.48	0.36	38.28	11.65	0.59	45.02	0.46	60.01	11.45	0.59	43.81	0.41	163.23
	30	11.46	0.56	40.36	0.38	39.41	11.51	0.58	45.04	0.43	67.53	11.61	0.58	44.28	0.41	170.26
	45	11.53	0.56	41.17	0.38	41.45	11.53	0.58	45.27	0.45	66.33	11.52	0.57	42.17	0.35	167.97
	60	11.57	0.57	40.73	0.36	38.06	11.61	0.57	43.98	0.41	58.18	11.52	0.55	41.02	0.58	94.75
190	15	11.49	0.55	43.92	0.42	44.51	11.54	0.57	39.00	0.40	54.75	11.52	0.57	50.74	0.56	177.70
	30	11.49	0.55	43.49	0.41	41.22	11.50	0.57	50.20	0.56	71.49	11.53	0.55	56.98	0.75	215.26
	45	11.46	0.55	32.23	0.23	44.43	11.49	0.57	42.19	0.35	57.91	11.46	0.55	47.21	0.44	197.49
	60	11.51	0.56	29.36	0.18	40.14	11.59	0.56	45.71	0.43	63.14	11.55	0.54	45.98	0.41	112.07
200	15	11.56	0.56	35.62	0.28	40.51	11.47	0.56	39.22	0.31	70.89	11.48	0.56	43.89	0.43	196.45
	30	11.56	0.56	34.30	0.25	38.87	11.55	0.56	39.03	0.28	69.28	11.53	0.55	42.54	0.41	184.16
	45	11.49	0.57	34.32	0.26	40.78	11.56	0.56	39.38	0.32	71.24	11.48	0.54	40.87	0.32	197.30
	60	11.89	0.58	28.75	0.21	41.23	11.57	0.55	38.31	0.28	53.71	11.61	0.54	37.71	0.31	155.84

Note: EMC = Equilibrium moisture content
MC = Moisture content
SG = Specific gravity
σ_{tll} = Ultimate tensile stress parallel to grain
W = Work to maximum load
Heat = Endothermic heat capacity

under inert conditions. The behavior can be explained by dividing the curves into six ranges. In Figure 4, Range I (0-100°C) shows the release of moisture from the sample as indicated by a small weight loss in TGA curve and a small endothermic peak in DSC curve. Range II (100-160°C) shows a rapid buildup of temperature by DSC curve and a constant weight by TGA curve. Range III (160-305°C) shows a small weight loss by TGA curve and a shallow endothermic peak by DSC curve which indicated a loss of amorphous structure of the cell wall (hemicellulose and some portion of lignin). Range IV (305-370°C) reveals a very fast loss of weight by TGA curve and a large endothermic peak by DSC curve. This important behavior indicates the disintegration and evaporation of the majority of cell wall structure (cellulose crystallites and lignin) which corresponds to the well established knowledge reported in several papers that cellulose crystallites did not decompose until the temperature reaches 340°C (Kim *et al.*, 2001; Wikberg, 2004). The DSC curve specifies the temperature of cell wall rupture at 355.30°C and the amount of thermal energy needed of 44.88 J/g. Range V (370-475°C) exhibits further pyrolysis of the remaining cell wall structure by ensuing small loss of weight of the TGA curve and an exothermic peak of the DSC curve. Range VI (above 475°C) shows the deterioration of carbon left as a residue from the pyrolysis as evident from a continued small

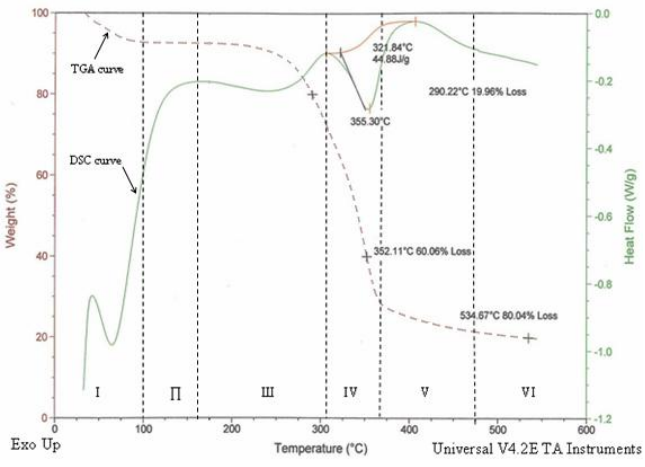


Figure 4. The typical TGA and DSC curves resulted from thermal analysis of an oven-dried strand.

loss of weight in TGA curve and a small drop of heat flow in DSC curve. The curves in Figure 5 are similar to those of Figure 4 except the release of moisture in Range I and the area of endothermic peak in Range IV are larger. Moreover, there is no endothermic peak in Range III of Figure 5, which implies that the destruction of amorphous structure of the EMC strands requires less thermal energy than the oven-dried strands presented in Figure 4. Figure 6 is interesting because the curves in Range III disappear and the area of endothermic peak in Range IV is very large while its left tail extends to the end of Range II. This is probably due to the strengthening effect of hemicellulose and lignin to cellulose crystallites which consequently requires large amount of thermal energy to decompose the cell wall of the Parawood strands subjected to boiling pre-treatment. This result seems to agree with the findings reported by Bhuiyan *et al.* (2000) who stated that "Our results suggested that other components accompanying wood cellulose were involved in the increase of crystallinity by heat treatment, and that wood cellulose contained more quasicrystalline regions than pure cellulose." They also reported that heat treatment under a highly moist condition causes significant increase in crystallinity of Spruce (*Picea sitchensis* Carr.), but negligible effect on pure cellulose from cotton.

The amount of endothermic heat used in decomposing the crystalline structure of the heat-treated strand samples was calculated and presented in a normalized unit (Joule per gram or J/g) as listed in Table 1. The resulted data of mechanical tests and thermal analysis were subsequently plotted in three dimensions as depicted in Figures 7, 8, and 9. The resulted F-tests of the analysis of variance indicated that

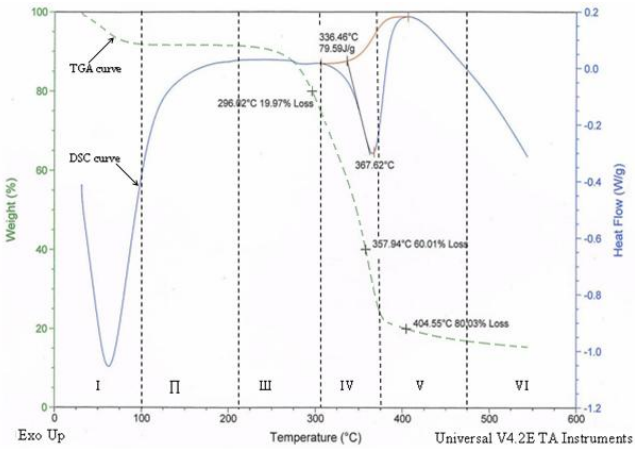


Figure 5. The typical TGA and DSC curves resulted from thermal analysis of an EMC strand.

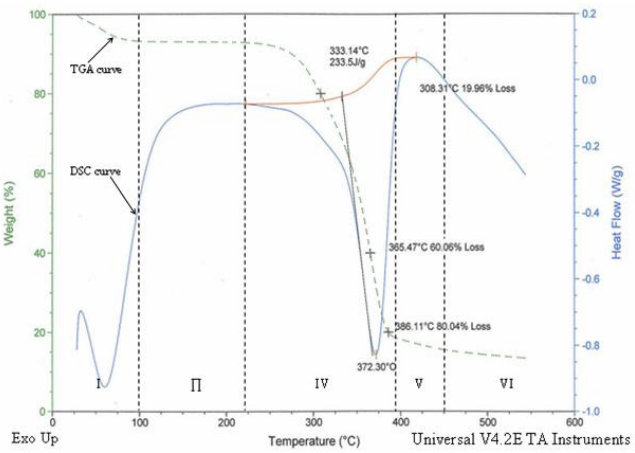


Figure 6. The typical TGA and DSC curves resulted from thermal analysis of a boiled strand.

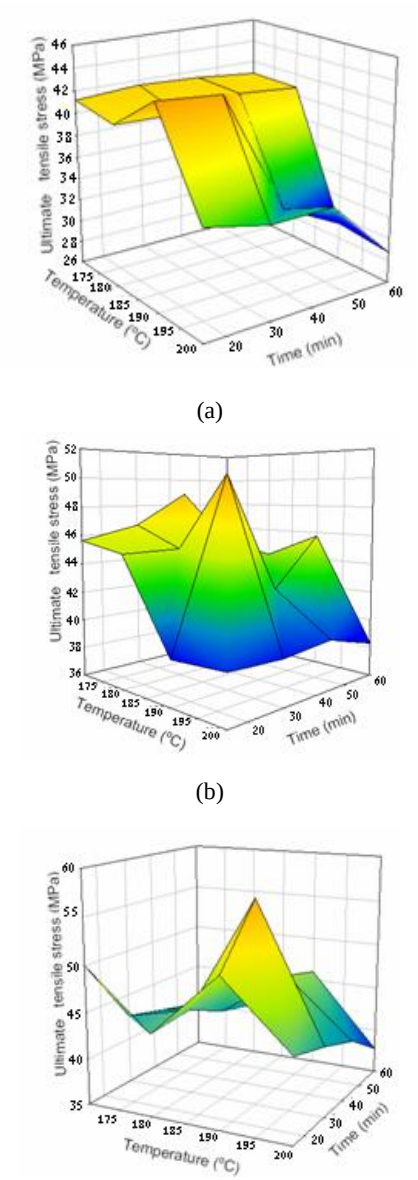
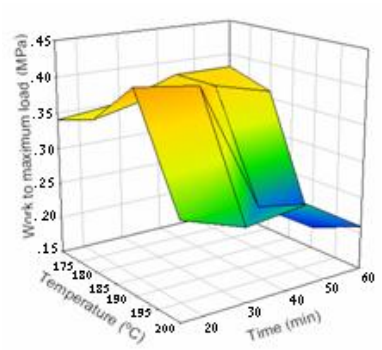
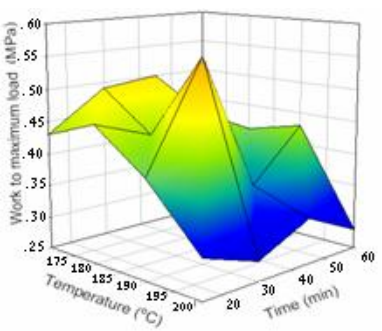


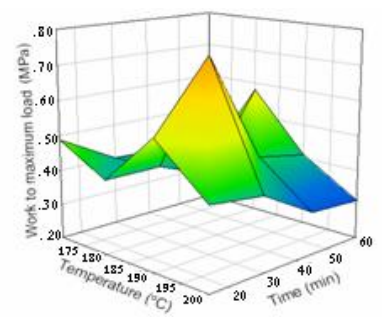
Figure 7. Plots of ultimate tensile stress parallel to grain versus temperature and time: (a) Oven-dried strand; (b) EMC strand; and (c) Boiled strand.



(a)

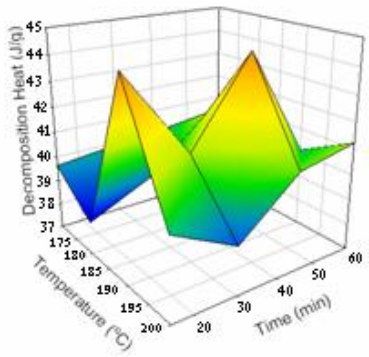


(b)

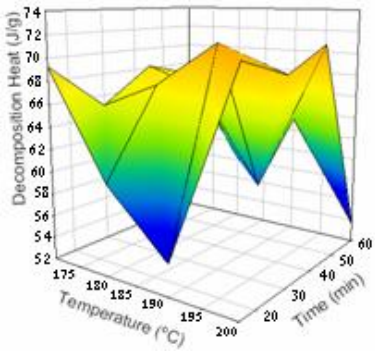


(c)

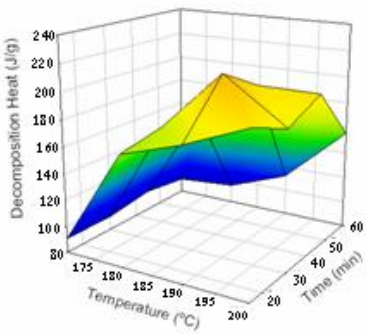
Figure 8. Plots of work to maximum load versus temperature and time: (a) Oven-dried strand; (b) EMC strand; and (c) Boiled strand.



(a)



(b)



(c)

Figure 9. Plots of decomposition heat versus temperature and time: (a) Oven-dried strand; (b) EMC strand; and (c) Boiled strand.

the differences were highly at 0.01 significant.

Regarding mechanical tests Plotted in Figure 7 reveal a clear peak for EMC strands (50.20 MPa) and for boiled strands (56.98 MPa), but it is not evident for oven-dried strands. It is interesting to note that the peaks occurred at the same temperature (190°C) and time (30 minutes) of heat treatment. These indicate that 190°C and 30 minutes are the optimum parameters for heat treating Parawood strands to increase its tensile strength and that moisture pre-treatment has significant effect on Parawood strands. Comparing the peaks, it was found that the results of boiled strands produce the highest peak of 56.98 MPa which is about 30 percent and 14 percent higher than oven-dried strands and EMC strands, respectively. Similarly, Plots in Figure 8 show that work to maximum load (toughness) is also highest for boiled strands (0.75 MPa) at 190°C and 30 minutes of heat treatment and that the peak value is about 79 percent and 33 percent higher than oven-dried strands and EMC strands, respectively. The results of mechanical tests therefore suggest that tensile strength and toughness of Parawood strands could be increased by pre-treatment in boiling water (100°C) for 30 minutes and immediately subjected to heat treatment at 190°C for 30 minutes.

For thermal analyses, it is obvious from Figure 9 that

the peaks of the decomposition-heat capacity of EMC strands and boiled strands coincide at the same heat-treating temperature and time of 190°C and 30 minutes except those of oven-dried strands which are slightly different. The results indicate that the highest endothermic heat capacity required to decompose the "modified crystallites" occurs for Parawood strands subjected to boiling pre-treatment in water for 30 minutes and heat treatment at 190°C and 30 minutes. It is interesting to note that the results of thermal analyses agree very well to the results of mechanical tests in this study. Unfortunately, reports on works similar to the present study (tensile and thermal behavior of heat treated wood strands) could not be found. Comparison of the findings is therefore not possible.

4. Conclusions

The following findings are drawn from the results of this study:

(1) The Parawood strands subjected to the pre-treatment by boiling in water (100°C) for 30 minutes and subsequently heat treated in a hot-air oven at 190°C for 30 minutes yields highest tensile strength of 57 MPa which is about 30 percent and 14 percent higher than the strands subjected to oven-dried pre-treatment and to EMC pre-treatment, respectively.

(2) The Parawood strands subjected to the treating process stated in (1) also yields highest toughness of 0.75 MPa which is about 79 percent and 33 percent higher than the strands subjected to oven-dried pre-treatment and to EMC pre-treatment, respectively.

(3) The Parawood strands subjected to the treating process stated in (1) requires highest amount of endothermic heat (215 J/g) for decomposing its modified crystallite structure.

(4) The pre-treatment by boiling in water could increase the tensile strength and toughness of heat-treated Parawood strands by strengthening cellulose crystallites to form a modified crystallite (a cellulose crystalline region reinforced and enlarged by hygrothermally modified lignin and hemicellulose molecules).

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