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Enhancing the Properties of Low Density Hardwood *Dyera costulata* Through Impregnation with Phenolic Resin Admixed with Formaldehyde Scavenger

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Abstract: The formaldehyde emission, properties and decay durability of *Dyera costulata* wood, impregnated with low molecular weight phenol formaldehyde (LmwPF) resin mixed with urea were investigated. The air-dry wood was impregnated with 20-40% LmwPF (M_w , 600) mixed separately with urea (30% based on solid PF), partially cured at 60°C for 30 min and subsequently heated at 150°C for 60-120 min. The treatments had successfully reduced the formaldehyde emission (FE) of the impreg and the degrees of reduction depend on curing time. Statistical analyses showed that the concentration of resin affected the density, stiffness, water absorption and thickness swelling, while the curing time affected the density, polymer loading, water absorption and thickness swelling. Impreg product had higher MOR, MOE and dimensional stability compared to the untreated wood. The treatments rendered the impreg product with approximately 20% in anti-swelling efficiency and had also changed the wood into highly resistant to fungal decay.

Key words: Impreg, phenol formaldehyde, urea, scavenger

INTRODUCTION

Chemical treatment of wood has been intensively investigated to improve the properties of low density wood and make them more usable in assessing the shortfall of wood supply. Numerous treatment systems have been proposed to improve wood properties, particularly for wood used under conditions where it is exposed to moisture or biological attack.

Impregnation with Phenol Formaldehyde (PF) resin followed by curing under heat has been known to provide tremendous enhancement in properties, which include mechanical strength, dimensional stability (Hill, 2006) and durability against decay and termite attack (Ryu *et al.*, 1991; Aikfei, 2010). This product is known as impreg (Rowell, 2005). Kajita and Imamura (1991) used low molecular weight formaldehyde resin (LmwPF) to improve the physical and biological properties of particle boards while, Anwar *et al.* (2006) and Loh *et al.* (2011), respectively, used this resin to enhance the properties of laminated bamboo and oil-palm stem veneer. The LmwPF resin managed to swell the cell wall without bonding to it. The PF resin with a molecular weight (MW) of 290-480

was able to penetrate the cell wall and increased stability (Rowell, 2005) but the PF resin with MW of 820 just remained in the cell lumen without gaining much stability (Furuno *et al.*, 2004). Impregnation of low density wood with PF followed by compressing at high temperature has also been proven in enhancing the bending strength, dimensional stability and durability against fungal attack (Zaidon *et al.*, 2010). This product, known as compreg, normally has Modulus of Rupture (MOR), Modulus of Elasticity (MOE) and hardness greater than the untreated wood due to the increase in density.

However, besides the positive effects of this treatment, the treated product releases free formaldehyde. It is generated from free, unreacted formaldehyde during the process of curing. When exposed to high temperature and humidity during polymerization, some of the methylol groups in the oligomeric chains of the LmwPF resin will release undesirable free formaldehyde into the surrounding (Hoong *et al.*, 2010). One way to capture this free formaldehyde is by using the formaldehyde catcher/scavenger (Roffael, 1993). There are many formaldehyde scavengers that have been introduced into the treatment system which include urea, ammonium

phosphate, potassium sulphite and sodium thiosulphate. However, urea is preferable due to its low cost and has been proven able to reduce the formaldehyde emissions from the *compreg* products made from low density wood, sesenduk (*Endospermum diadenum*) and mahang (*Macaranga* spp.) (Zaidon, 2009). It is expected that the introduced urea will react with the free formaldehyde and form a rigid cross-linked polymer of urea formaldehyde.

In this study, urea was mixed with the LmwPF solution before it was used in the impregnation of low density wood. This study reports the formaldehyde emission, properties and durability of the impreg made from the low density hardwood jelutong (*Dyera costulata*). The effect of PF concentration and curing time on the properties of the treated wood were also evaluated.

MATERIALS AND METHODS

Materials: The jelutong wood used in this study was obtained from Ayer Hitam Forest Reserve, Puchong, Selangor. The treating solution was composed of low molecular weight phenol formaldehyde (LmwPF) resin (M_w 600, 45% solid) purchased from Malaysian Adhesive Chemical, Shah Alam. Urea granules were used as the formaldehyde scavenger which was mixed with the PF resin.

Samples preparation: PF resin was diluted with distilled water to 20%, 30% and 40% concentrations. Urea was mixed separately into the three solutions. In the earlier work, it was found that the urea concentration in the range of 10-30% (based on solid PF) was able to reduce FE from the impreg and compreg made from the low density wood, *Endospermum diadenum* and the FE reduction increased with the urea concentration (Zaidon, 2009). In this case, 30% of urea (based on solid PF) as the formaldehyde scavenger was used. The pre-conditioned wood (MC = 12%) which was free from defects, was flat sawn into the samples (the width is the tangential surface) with a nominal dimension of 150 mm long, 50 mm wide and 5 mm thick. The untreated samples served as the control for comparison purposes.

Impregnation treatment process: The pre-weighed samples were vacuum-pressure impregnated with the solution separately in an impregnation apparatus. The treating cycle consisted of a 15 min initial vacuum of 85 kPa followed by filling the set-up with the treating solution under vacuum. A final pressure of 340 kPa was applied for 30 min at an ambient temperature. The impregnated samples were immediately weighed before

being partially cured at 60°C for 30 min and subsequently cured at 150°C for 60, 90 and 120 min. In a preliminary work, Zaidon (2009) found that a minimum of 60 min was required to cure the PF resin in the wood of the same thickness. All the treated samples were then conditioned at 65±2% RH and 25±2°C until a constant weight was obtained. The density and Weight Percent Gain (WPG) for each impreg were then determined using the following equations:

$$\text{Density (k gm}^{-3}\text{)} = (W_f/V_f) \quad (1)$$

$$\text{WPG (\%)} = 100 [(W_f - W_i) / W_i] \quad (2)$$

where, W_f is the weight of the sample after curing and conditioning, kg, V_f is the volume of the sample after curing and conditioning m^3 and W_i is the initial weight of the sample, kg.

Formaldehyde emission (FE) test: The impreg samples with or without admixed urea were subjected to the FE test after conditioned to a constant weight. The test was carried out following the method specified in the Malaysian Standard (MS, 2005) Wood-based panels-Determination of Formaldehyde Emission by the Dessicator Method. Calibration curve was first produced from a standard formaldehyde solution by iodometric titration. About 8-10 test pieces (total surface area of 1800 cm^2) were placed in a desiccator above a plastic container filled with 300 mL of distilled water. They were left in the laboratory for 24 h at an ambient temperature. The background formaldehyde was prepared using a desiccator without the test piece. The formaldehyde absorbance was measured photometrically at 412 nm wavelength and these values were used to determine the FE using Eq. 3, as follows:

$$G = f \times (A_d - A_b) \times 1800/S \quad (3)$$

where, G is the concentration of formaldehyde due to the test pieces, ppm, A_d is the absorbance of the solution from the desiccators containing the test pieces, A_b is the absorbance of the background formaldehyde solution, f is the slope of the calibration curve for the standard formaldehyde solution, ppm and S is the surface area of the test pieces, cm^2 .

FT-IR spectroscopy analysis: The infrared spectrum analysis was performed on the impreg samples using a PerkinElmer Spectrum 100 Series FT-IR, equipped with a CsI detector attached to the ATR unit. The FTIR-UATR spectra were recorded in the wave number ranging from 4000-400 cm^{-1} .

Evaluation of dimensional stability: The degree of dimensional stability was determined by estimating and comparing the anti-swelling efficiency (ASE), Water Absorption (WA) and Thickness Swelling (TS) of the treated and control samples of 20×20 mm dimensions. The swelling process was done through 15-min vacuum followed by soaking in distilled water for 24 h (Ashaari *et al.*, 1990a). The weights before and after swelling process were measured. The volumetric swelling coefficients (S), anti-swelling efficiency (ASE), water absorption (WA) and thickness swelling (TS) were calculated using the following equations:

$$S (\%) = 100 [(V_f - V_i) / V_i] \quad (4)$$

$$ASE (\%) = 100 [(S_c - S_t) / S_c] \quad (5)$$

$$WA (\%) = 100 [(W_f - W_o) / W_o] \quad (6)$$

$$TS (\%) = 100 [(T_f - T_o) / T_o] \quad (7)$$

where, V_f is the volume of the sample after soaking, mm^3 , V_i is the volume of oven-dried sample, mm^3 , S_i is the untreated volumetric swelling coefficient, mm^3 , S_t is the treated volumetric swelling coefficient, mm^3 , W_f is the weight of sample after soaking, g, W_o is oven-dried weight of the sample before soaking, g, T_f is the thickness of the sample after soaking, mm and T_o is the oven-dried thickness of the sample before soaking, mm.

The treated pieces were also exposed to water vapour at 95% humidity until equilibrium. Graphs of WA and TS versus days of exposure were plotted. In this study, the exposure time in water vapour was taken to be completed when the untreated samples achieved the constant weight. This took approximately 24 days.

Evaluation of mechanical properties: Static bending test was performed according to the British Standard BS 373:1957 (BSI, 1957) with a modification of specimen size (20 by 150 mm) to compare the strength and stiffness of the treated and untreated samples. The specimens were placed on a 50 kN. Instron universal testing machine, on a fixed support with a span of 120 mm and were tested under a static load with a crosshead speed of 6.4 mm min^{-1} . The mechanical properties calculated from the load deflection curves included modulus of rupture (MOR) and modulus of elasticity (MOE).

Decay resistance test: Decay resistance was tested according to the American Standard Test Method (ASTM D 1413-99) for Wood Preservatives by Laboratory Soil-Block Cultures (ASTM, 1999). The specimens

(20×20×5 mm) were first conditioned at $25 \pm 2^\circ\text{C}$ and $65 \pm 2\%$ RH until a constant weight was reached. The pre-weighed treated and untreated specimens were exposed to the white rot fungus (*Pycnoporous sanguineus*) for 12 weeks. After exposure to the fungus, the specimens were again conditioned until they reached a constant weight. The weight loss due to the decay was calculated using Eq. 8, as follows:

$$WL (\%) = 100 [(W_o - W_c) / W_o] \quad (8)$$

where, W_o is the weight of conditioned specimen before test, g and W_c is the weight of conditioned specimen after test, g.

Statistical analysis: Statistical analysis was carried out using a two-way analysis of variance (ANOVA) to analyze any changes in the property values among the treatment combinations. The mean was separated using the Duncan Multiple Range Test (DMRT) at $p \leq 0.05$.

RESULTS AND DISCUSSION

Formaldehyde emission of the Impreg: The calibration curve produced from iodometric titration is presented in Fig. 1. The graph was plotted to estimate the slope which was then used to determine formaldehyde emission. The slope obtained $9.089x$ with $R^2 = 0.997$. The results showed that the admixture of urea in the resin imparted significant reduction of the FE levels (Fig. 2). The FE values for the Impreg without urea ranged from 80.89 to 172.2 ppm and the values greatly reduced to 8.70-15.3 ppm when treated with admixed urea followed by curing for 120 min. The FE increased with resin concentration and decreased with curing time. FE was found to be positively correlated ($R = 0.897$) with polymer loading as described by WPG (Fig. 3). The results reflect that the reactive urea bonded

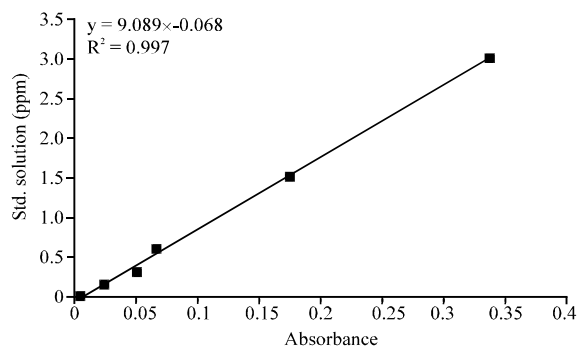


Fig. 1: Calibration curve of standard formaldehyde concentration vs. absorbance using UV-spectrophotometer

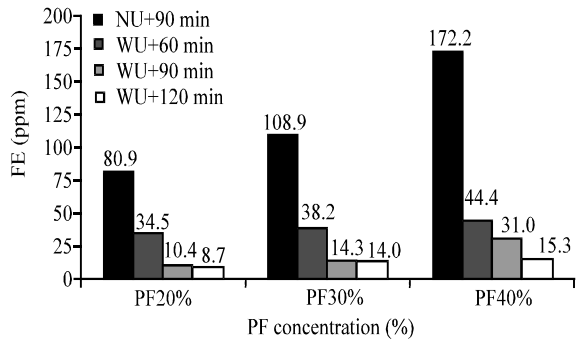


Fig. 2: FE of impreg Jelutong treated at different PF concentrations, with and without addition of urea scavenger cured at 60, 90 and 120 min, respectively (Note: NU is no urea and WU is with urea)

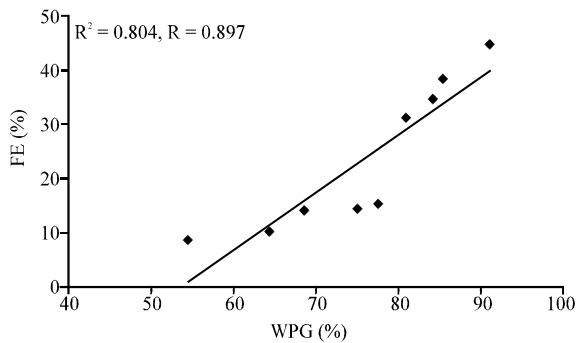


Fig. 3: The Effect of weight percent gain (WPG) on formaldehyde emission of Impreg jelutong

with the free formaldehyde released by some of the methylol groups from the resin and formed the UF cross-linked polymer. It was anticipated that if the curing time was prolonged, more polymerization of resin would occur resulting in lower FE (Amarullah *et al.*, 2010) but the mechanical properties might adversely be affected due to the hydrolyses of the cellulose chain (Ashaari *et al.*, 1990b).

The presence of cross-linked UF polymer was also apparent from the FTIR spectra of the impreg product (Fig. 4). At the spectral area of 3700-3000 cm^{-1} , it indicated the difference between the pre-polymers and cured resin (Zobra *et al.*, 2008) and the absorbance peak for the cured UF was at 3350 cm^{-1} due to the formation of bonded NH group. Our study showed that this peak was at 3339 cm^{-1} . The formation of cross-linked polymer of PF was proven by the absorbance peak of 1234 cm^{-1} . Poljansek and Krajnc (2005) found that the absorbance bands which corresponded with the PF resin was 1235 cm^{-1} for the asymmetric stretch phenolic C-C-OH. The excess free formaldehyde was still available in the treated wood as indicated by the absorbance peak of 2890 cm^{-1} and this was comparable to 2914 cm^{-1} (Poljansek and Krajnc, 2005) which is the characteristic peak of methyl group C-H.

Properties of impreg: Table 1 summarizes the analysis of variance (ANOVA) for the effect of resin concentration and curing at $p \leq 0.05$. The descriptive statistics of the

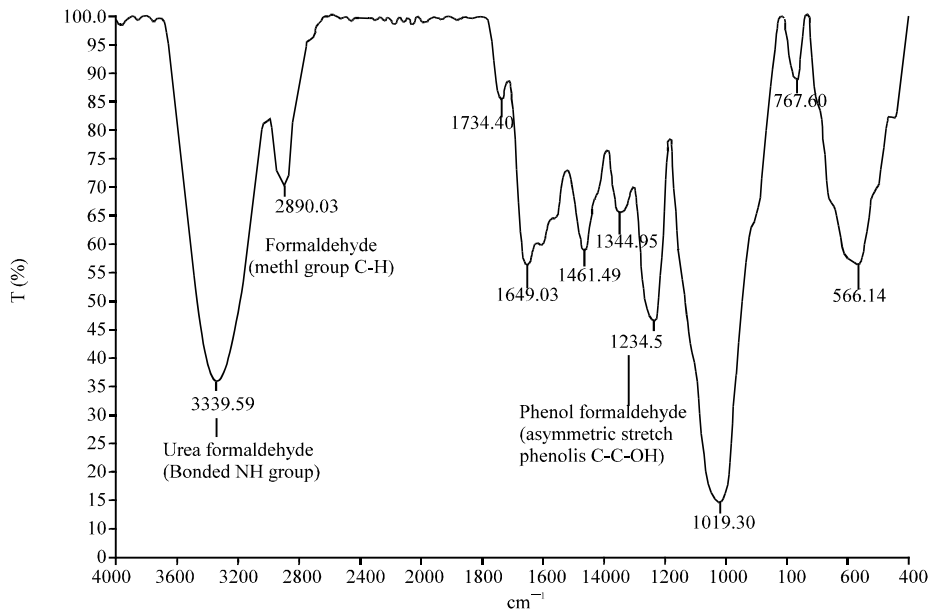


Fig. 4: FTIR spectra of impregjelutong treated with 30% LmwPF admixed with urea and cured for 90 min

Table 1: A summary of the analysis of variance ($p \leq 0.05$) for the effects of concentration of LmwPF and curing time

Variables	df	p-value						
		Density	WPG	MOR	MOE	ASE	WA	TS
Concentration (C)	2	0.000	0.576	0.173	0.028	0.582	0.000	0.000
Curing time (CT)	2	0.001	0.007	0.677	0.126	0.551	0.000	0.002
C x CT	4	0.747	0.913	0.173	0.333	0.719	0.124	0.075

Table 2: Mean on density, weight percent gain, and mechanical properties (\pm std deviation) of Impreg jelutong at different treatment combinations

PF con. (%)	Curing time (min)	Density (kg m^{-3})	WPG (%)	MOR (Nmm^{-2})	MOE (Nmm^{-2})	ASE (%)	WA (%)	TS (%)
20	60	630 \pm 127.0 ^{CD}	84.13 \pm 9.81 ^{AB}	79.40 \pm 7.65 ^A	8255 \pm 345 ^{BC}	28.26 \pm 5.85 ^{AB}	43.56 \pm 2.19 ^B	2.00 \pm 0.80 ^{B^{CD}E}
	90	594 \pm 21.47 ^D	64.31 \pm 25.3 ^{BC}	80.70 \pm 16.00 ^A	9579 \pm 2038 ^{AB}	27.67 \pm 10.92 ^{AB}	38.19 \pm 2.13 ^C	1.07 \pm 0.72 ^{EF}
30	120	583 \pm 46.83 ^D	54.55 \pm 18.44 ^C	77.73 \pm 8.38 ^{AB}	8903 \pm 447 ^{ABC}	35.39 \pm 10.65 ^A	36.82 \pm 2.65 ^{CD}	0.82 \pm 0.45 ^F
	60	744 \pm 55.87 ^{AB}	85.31 \pm 10.5 ^{AB}	70.31 \pm 17.55 ^{AB}	7752 \pm 2372 ^C	14.92 \pm 3.27 ^C	35.88 \pm 2.38 ^{CD}	3.01 \pm 0.38 ^B
	90	690 \pm 24.04 ^{BC}	75.03 \pm 4.6 ^{BC}	80.90 \pm 12.60 ^A	8744 \pm 686 ^{ABC}	15.84 \pm 10.50 ^{BC}	33.57 \pm 1.48 ^D	2.71 \pm 0.33 ^{BC}
40	120	649 \pm 28.40 ^{CD}	68.55 \pm 2.47 ^{BC}	69.98 \pm 9.04 ^{AB}	8442 \pm 919 ^{BC}	14.02 \pm 6.79 ^{BC}	26.64 \pm 1.01 ^E	1.80 \pm 0.56 ^{C^{DE}F}
	60	797 \pm 13.94 ^A	91.06 \pm 11.08 ^A	80.71 \pm 6.36 ^A	9192 \pm 834 ^{ABC}	17.94 \pm 15.95 ^{BC}	24.72 \pm 3.70 ^E	2.85 \pm 1.05 ^B
	90	759 \pm 32.18 ^{AB}	80.93 \pm 6.8 ^{AB}	78.57 \pm 7.04 ^A	8985 \pm 859 ^{ABC}	25.98 \pm 14.19 ^{AB}	20.55 \pm 4.67 ^F	1.52 \pm 0.66 ^{DEF}
120	698 \pm 34.10 ^{BC}	77.51 \pm 4.29 ^{BC}	83.92 \pm 12.22 ^A	10432 \pm 1145 ^A	14.41 \pm 10.60 ^{BC}	19.91 \pm 3.02 ^F	2.51 \pm 0.65 ^{BCD}	
Untreated solid wood	90	396 \pm 32.02 ^E	-	62.95 \pm 4.79 ^B	6124 \pm 486 ^D	-	63.17 \pm 3.54 ^A	4.18 \pm 1.15 ^A

Group means with the same letter are not significantly different at $p \leq 0.05$

properties are shown in Table 2. The results showed that the concentration (C) of resin significantly affected the density, MOE, WA and TS, while curing time (CT) affected only the density, WPG, WA and TS. The interaction between C and CT showed no significant difference for all properties tested.

As displayed in Table 2, the properties of the impreg were superior than the untreated wood indicating that the treatments had enhanced the wood properties. The density of the Impreg increased significantly by 50-100% to about 583-797 kg m^{-3} from the original density of 396 kg m^{-3} and it was positively correlated with resin concentration and inversely correlated with curing time. WPG increased with the concentration and decreased as the curing time increased. The WPG for those treated with 20% PF was between 54.55-84.13%, whereas for those treated with 40% PF, the values ranged from 77.51% to 91.06%. The adverse effect of WPG with the curing time may probably due to insufficient time to fully cure the resin near the inner zone of the sample. The partly cure resin may consist of some water molecule which would add to the weight of the treated product. As the curing time was lengthened, more resin was expected to cure and form the cross-linked polymer. A positive correlation ($R = 0.83$) between WPG and density was also noted, as shown in Fig. 5.

The MOR values among the treatment combinations did not show any significant difference at $p < 0.05$. The values were in the range of 69.98-83.92 N mm^{-2} , which were higher than the untreated wood (62.95 N mm^{-2}). For the MOE, there was no specific trend, but the value was significantly higher compared with the untreated wood, that was 7752-10,432 N mm^{-2} versus 6124 N mm^{-2} . It is believed that the resin filled in the lumen and wall of the cell and formed a rigid cross-linked polymer resulting in

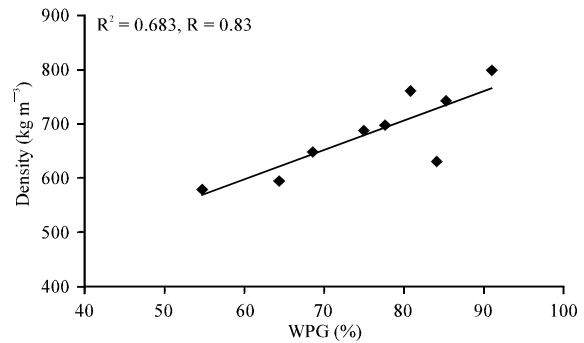


Fig. 5: The effect of weight percent gain (WPG) on the density of impreg jelutong

the increase in strength and stiffness. This phenomenon has also been reported by many researchers (Deka *et al.*, 2000; Kajita and Imamura, 1991).

Dimensional stability of impreg: The dimensional stabilization of the impreg was quantified by analyzing the anti-swelling efficiency (ASE), water absorption (WA) and thickness swelling (TS). The untreated samples were used for comparison purposes. The ASE values were 27.7-35.4% for the wood treated with 20% resin, 14.0-15.8% for the treated wood with 30% resin and 14.4-26.0% for those treated with 40% resin. The ASE values indicated that some of the resin had successfully penetrated into the cell wall and upon heating, it formed an insoluble polymer which bulked the cell wall (Ohmae *et al.*, 2002). At lower PF concentrations, more resin would penetrate into the cell wall and as a result, higher ASE values would be achieved compared with those treated at higher concentrations. The ASE for the impreg was markedly lower compared with the wood

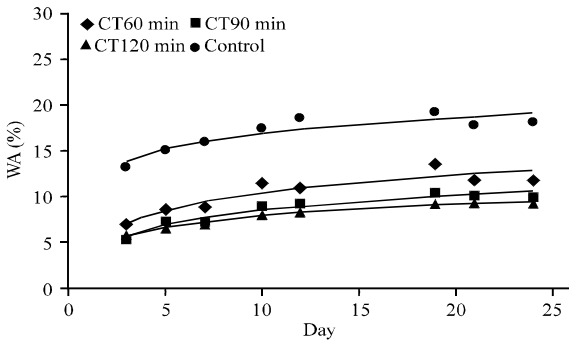


Fig. 6: Water absorption of the impreg produced from 30% resin + 30% urea and untreated wood exposed to 95±2% humidity

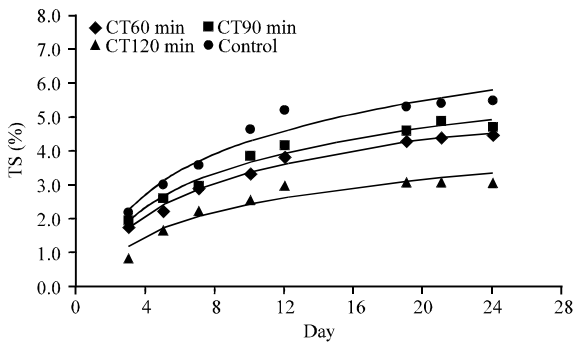


Fig. 7: Thickness swelling of the impreg produced from 30% resin + 30% urea and untreated wood exposed to 95±2% humidity

treated without the admixture of urea reported in the previous study. Aikfei (2010) found that *mahang* (*Macaranga* spp.) impregnated with the same resin without the formaldehyde scavenger had an ASE value of more than 60%. Rowell (2005) revealed that the molecular weight distribution of the resins significantly influenced the viscosity and the ability of the resin to penetrate the cell wall. The presence of urea would have increased the molecular weight of the resin system, thus limit the penetration into the cell wall of the treated wood.

Lower WA (19.91-43.6%) was also found for the impreg compared with the untreated wood (63.2%). Among the treatment combinations, regardless of the curing time, the impreg produced from 40% resin had the lowest WA. This showed that at this concentration, the treating solution had a higher molecular weight distribution, thus limited the penetration into the cell wall and in turn, higher amounts of resin filled up the lumen and formed internal coating which reduced water absorption.

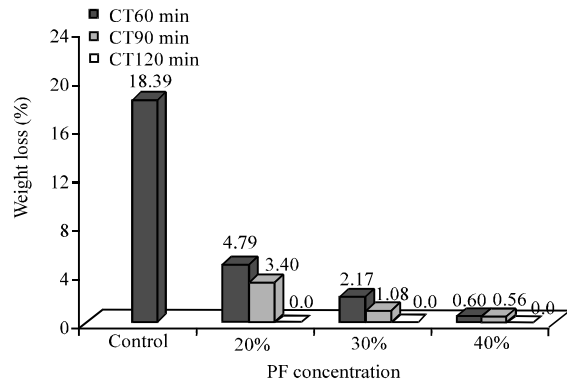


Fig. 8: WL of impreg and untreated wood at different treatment combinations after the exposure to *P. sanguineus* for 12 weeks

The behaviour of the impreg and the untreated wood (control) over a period of exposure to humidity (95±2%) is illustrated in Fig. 6. The trends showed that the moisture uptake for the control was higher than the impreg. Among the impreg products, the one with the longest curing time (120 min) had the least moisture uptake whereas those with the shortest curing time (60 min) had the most. The mean WA at the end of the exposure period (24th day) was 18% for the control treatment and between 8-11% for the impreg. The plausible reason was that, at a shorter curing time, incomplete polymerization of the resin occurred in the lumen, thus leaving more air spaces for moisture uptake. In terms of TS (Fig. 7), the trends over period of exposure to humidity were similar to WA where the impreg with the longest curing time had the lowest trend with a maximum TS of 3.10% while the control treatment had the highest trend with a maximum TS of 5.50%.

Resistance of impreg against fungal decay: The weight loss (WL) among the impreg after 12 weeks of exposure to the white rot fungus (*Pycnoporous sanguineus*) can be seen in Fig. 8. The impreg experienced lower WL compared with the untreated wood, that was 0 to 4.8% for the impreg and 18.4% for the untreated wood. It was also found that WL decreased with the increase of concentration and curing time. The findings showed that the impregnation treatment with the presence of urea had successfully increased the resistance of *jelutong* impreg against fungal decay by 80-100%. Earlier research revealed that polymer loading of 30% was enough to protect *Macaranga* sp. impreg from white rot (Aikfei, 2010) and at resin weight gain of 10-20% was enough to suppress the growth of decay fungi (Furuno *et al.*, 2004).

CONCLUSIONS

Impregnation of low density wood *Dyera costulata* with admixture of urea (30% based on solid PF resin) was able to reduce formaldehyde emission from the products. The FTIR spectra analyses showed that urea had reacted with some free formaldehyde and formed a cross-linked UF polymer. Regardless of PF concentration, the formaldehyde emission decreased as the curing time increased. The lowest formaldehyde emission was found on the impreg cured at 150°C for 120 min. The impreg had higher MOR, MOE and dimensional stability compared to the untreated solid wood. The analysis of variance showed that the concentration of resin (C) significantly affected the density, MOE, WA and TS while curing time (CT) affected the density, WPG, WA and TS. There was no interaction between C and CT for all properties tested. The treatment rendered the impreg products with approximately 20% in ASE. Besides, the moisture uptake and thickness swelling of the impreg were lower compared to the untreated wood when exposed to high humidity over a period of time. The treatment had also changed the wood into highly resistant to fungal decay.

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