Effect of Acetylation on the Physical and Static Bending Properties of Cultivated *Rotan Manau* (*Calamus manan*) Grown in Peninsular Malaysia

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ABSTRACT

The physical and static bending properties of cultivated 'rotan manau' (*Calamus manan*) reacted with acetic anhydride at 110 °C for the time intervals of 0.25 to 30 hours were investigated. The study aimed to investigate the effects of various reaction times on the physical and static bending properties. Rattans aged 10 and 13 years were obtained from local rubber tree plantations. This study found that almost all the physical properties of acetylated rattan were not significantly different by age (except specific gravity changes) and reaction periods (except weight gain and OH substitution). The static bending properties varied by rattan age, which the modulus of rupture (MoR), modulus of elasticity (MoE) and maximum load (ML), was not significantly different by reaction period for both ages, except for the MoR of the older acetylated rattan. The acetylation of rattan, performed at 110°C for prolong reaction period, did not impair the static bending properties.

Keywords: Acetylation, cultivated rattan, physical properties, static bending

INTRODUCTION

In addition to natural beauty, rattans are renowned as a material with high flexibility. This property makes rattan easily moulded into a wide range of product applications such as furniture, temporary suspension bridges, crutches, and sport equipment. Recently, rattan has been used as an alternative for timber in new applications such as handles for hammer and hoe, mainly because of the increasing price of timber. These types of product can be classified as semi-structural or structural and require a certain mix of strength properties. Acetylation process

improves the dimensional stability and decay resistance of wood (Rowell, 2006; Hill, 2006); however, there was concern over the influences of acetylation on the strength properties of wood due to its by-product of acetic acid, which resulted either in the reduction of fibre and lignocellulose contents or a possibility of hydro-thermal effect (Bongers & Beckers, 2003; Jorrison *et al.,* 2005).

The performance of acetylated wood in terms of mechanical properties differs by species, reaction period, and mode of test. The tensile strengths of acetylated Scots pine (*Pinus sylvestris*) and lime (*Tilia vulgaris*) were not significantly different compared to the untreated specimens (Rowell & Banks, 1987). Acetylated beech wood gave a slight increase in modulus of rupture (MoR) and a small decrease in modulus of elasticity (MoE) (Militz, 1991). The MoR and MoE of acetylated pine (*Pinus sylvestris*) reduced by 6% but increased in acetylated spruce (*Picea abies*) by about 7% (Larsson & Simonson, 1994). Acetylated pine (*Pinus sylvestris*) reacted for 1 hour had a higher tensile modulus as compared to the air dried specimens; and even after 4 hours of reaction gave tensile modulus higher than the air dried specimen (Ramsden *et al.,* 1997). The MoE of acetylated radiata pine (*Pinus radiata*) in structural sizes was not significantly different but the MoR was slightly lower than the untreated specimen, although these comparisons were difficult due to high variation (Jorrisen *et al.,* 2005). In term of reaction system, the MoR of acetylated Sitka spruce (*Picea sitchensis*) increased with the increasing WPG, but it was slightly lower in the xylene system compared to the pyridine system and uncatalysed system. The MoE was not significantly different with the reaction systems (Minato *et al.,* 2003). The MoR and MoE in finger jointed acetylated beech (*Fagus sylvatica*) decreased by 141% and 63% as compared to the untreated specimen (Papadopoulos, 2008). It has been hypothesised that the variation of the mechanical properties in acetylated wood is probably caused by either the reduction of moisture content (i.e. lower EMC values), or as a result of the lowering cross sectional area or as a result of cell wall degradation that is caused by prolonged reaction times (Rowell, 1983; Hill, 2006). In contrast to wood, the acetylation of rattan has not been investigated. The acetylation of rubber wood was conducted by Karim *et al.* (2006), but the study was limited to reaction profile and dimensional stability.

This study aimed to investigate the static bending properties of rattan reacting with acetic anhydride and its relation to physical properties.

MATERIALS AND METHODS

Source of Materials

Calamus manan aged 10 and 13 years were studied. The rattans were obtained from small holders of rubber tree plantations at Felda Mempaga in Pahang (about 3° 31' N and 101° 55' E, East Peninsula Malaysia) and Baranang, Selangor (about 2⁰ 56' N 101° 52' E, West Peninsula Malaysia), respectively. The age of the rattan was estimated based on

the records of the plantation establishment obtained from the farmers. The two sites share the same climatic conditions such as the characteristic features of the Malaysian climate are uniform temperature, with high humidity and copious rainfall all year round. The type of soil is also similar, as indicated by the establishment of the rubber wood plantations at both locations.

Determination of the Chemical Composition

One whole stem of each rattan age was used for investigating the chemical composition. They were divided into five different portions of the total stem length, namely; basal, upper basal, middle, upper middle and top. Transversely, the middle internode of each portion was divided into three different sections, namely; periphery, intermediate and centre. The rattan particles were oven dried at 60ºC for 24 hours and ground with a hammer mill. The rattan flour was equilibrated to 15% moisture content, and passed through sieves to retain the fraction of 0.4 mm to 0.1 mm. The methods used to determine the holocellulose, α-cellulose and lignin contents were in accordance to Wise *et al.* (1946), Cross-Bevan (Tappi T 9m-54; Anon, 1954) and Klason lignin (Tappi T 222 om-83; Anon, 1983), respectively. Six replicates of each chemical element were analysed for each rattan age.

Preparation of the Specimen for Acetylation

Stem from the middle and upper middle portions of both ages, having diameter more than 35 mm and classified as weak rattan (MoR below than 45 MPa), were cut into the size of 5 X 10 X 100 mm³ (r x t x l). All the specimens were marked, soxhlet extracted with toluene/methanol/acetone mixture (4:1:1) for eight hours and oven dried at 103ºC for 24 hours. The specimens were transferred into desiccators and allowed to cool at ambient temperature over silica gel, weighed to 0.1 mg and the volume measured using a digital bed micrometer (Mitotoyo: Kawasaki, Japan).

Acetylation Reaction

Rattan strips were vacuum-impregnated in acetic anhydride at 0.7 kPa for one hour and were kept submerged overnight before they were transferred into acetic anhydride at 110ºC. The reaction was continued for various time intervals (0.25, 0.5, 1, 4, 10, 15, 24 and 30 hours) to give a range of weight percentage gains. The blocks for the longest reaction time were inserted at the start while other specimens were subsequently added to give shorter reaction times. At the end of the reaction period, the reaction was quenched in ice until the liquid temperature reached 20ºC. The residue was drained-off and replaced with acetone and cooled in ice for one hour, shaken a few times, discharged, and refilled with fresh acetone. The procedures were repeated twice. The acetylated rattan was finally Soxhlet extracted with toluene/methanol/ acetone mixture (4:1:1) for eight hours and oven dried at 103ºC for 24 hours. This procedure was sufficient enough to remove all the un-reacted acetic anhydride and by-

product of acetic acid (Hill & Jones, 1999). Dry specimens were cooled, weighed and measured as above. Seven replicates for each age and reaction period were used in this study.

Determination the Physical Properties

The specific gravity prior and following modification were measured in according to ASTM D 2395 (Anon 1977). The WPG and bulking coefficient of acetylated rattan were calculated using the following formula:

$$\text{Weight gain (\%)} = [(Wm - Wum)] \times 100 \qquad [1]$$

$$\text{Bulking coefficient (\%)} = [(Vm - Vum)Vum] \times 100 \qquad [2]$$

Alternatively, the void volume changes due to acetylation were calculated as adapted from the formula by Bowyer *et al.* (2003).

$$\text{Void volume (\%)} = [(1 - (SGod/SGcw)] \times 100 \qquad [3]$$

$$\text{Void volume changes (\%)} = [(VMm - VMum)/VMum] \times 100 \qquad [4]$$

Where,
Wm = mass of modified rattan.
Wum = mass of unmodified rattan.
Vm = volume of modified rattan.
Vum = volume of unmodified rattan.
SGod = specific gravity of modified or unmodified rattan.
VMm = void volume of modified rattan.
VMum = void volume of unmodified rattan.

SGcw = specific gravity of dry cell wall rattan, 0.655 (Ashaari, 1995).

Determination the Hydroxyl Group

The OH substitution was calculated using the following formula (Hill & Jones, 1999; Hill *et al.,* 2006):

$$\text{OH groups substitution (mmol g}^{-1}) = [(Wm - Wum)/Wum](MW - 1)] \times 100 \qquad [5]$$

$$\text{Theoretical molar volumes (cm}^3/\text{mole)} = [(Vm - Vu)/M] \qquad [6]$$

Where,
MW = molecular weight of acetyl group.
M = number of moles of adduct (=[weight gain in gms] / molecular weight of adduct).

Determination of the Static Bending Test

Acetylated and untreated rattans were conditioned at 20ºC and 65% relative humidity for one week and tested in the same room. The three point bending test in a span of 80 mm and a crosshead speed of 5mmmin^{-1} was set using an Instron machine. The load was positioned onto the tangential surface of the specimens, perpendicular to the grain direction. The moisture content of the specimens was measured after the mechanical testing.

RESULTS AND DISCUSSION

Variation of the Physical Properties

The results for the physical properties obtained from this study are presented in

Table 1. All the physical properties of the acetylated rattan were not significantly different by age, except for specific gravity changes. The older acetylated rattan (7.7 %) had higher specific gravity changes than younger acetylated rattan (6.0 %).

In the younger acetylated rattan (Fig.1.1 to Fig.1.10), almost all the physical properties were not significantly different by the reaction periods, i.e. ranging from 6.2 to 9.9% (bulking coefficient), 0.39 to 0.46 (specific gravity following modification), 5.2 to 6.6% (specific gravity changes), 29.5 to 41% (void volume following modification), -8.3 to -17.1% (void volume changes) and 17 to 25 cm^3mol^{-1} (molar volume). In contrast, the reaction period influenced weight gain and OH substitution for both ages. The weight gain was significantly increased from 0.25 hour (12.7%) to 10 hour (16.2%) and levelled-off up to 24 hours (16.1%) before it significantly declined at 30 hours (15.4%) reaction period. Similarly, the OH substitution was significantly increased from 0.25 hour ($9mMolg^{-1}$) to the maximum at 10 hours ($12mMolg^{-1}$) and levelled-off up to 12 hours ($12mMolg^{-1}$) before it significantly declined at 30 hours ($11 mMolg^{-1}$) reaction period.

The same trends were also observed in the older acetylated rattan (Fig.2.1 toFig. 2.10). Almost all the physical properties were also not significantly different by the reaction period ranging from 5.6 to 8.8% (bulking coefficient), 0.34 to 0.43 (specific gravity following modification), 34.1 to 48% (void volume following modification), -7.3 to -16.2% (void volume changes) and

15 to 20 cm^3mol^{-1} (molar volume). The weight gain was significantly higher at 0.25 hour (15.8%) than 0.5 hour (12.1%); the value was then increased at 10 hours (17.2%) and levelled-off up to 24 hours (18%) before it significantly declined to 30 hours (15.9%) reaction period. The OH substitution was significantly higher at 0.25 hour (11 $mMolg^{-1}$) than 0.5 hour (9 $mMolg^{-1}$); the value was then significantly increased at 10 hours (12 $mMolg^{-1}$) and levelled-off up to 24 hours (13 $mMolg^{-1}$) before it significantly declined at 30 hours (21 $mMolg^{-1}$) reaction period. The specific gravity changes were slightly lower at 0.5 hour (5.4%) compared to 0.25 hour (7.5%); the value was then stable from 1 hour to 10 hours (7.1 to 7.7%), followed by increasing to its maximum at 24 hours (9.1%) before slightly declining at 30 hours (7.7%) reaction period. This indicates that the trends of WG and OH substitutions were consistent for both ages, which were levelled-off after 10 hours. The trends of the other physical properties were almost the same for both the ages. Regardless of the reaction periods, acetylation increased the specific gravity and reduced the void volume of rattan of either age.

The maximum WG of the acetylated rattan strip of either age (15.7%) was lower than the acetylated wood (about 25%) as reported by Hill (2006). This was probably caused by the higher cellulose (70.1 to 73.3%) but lower hemicellulose (13.0 to 14.5%) and lignin (17.3 to 19.4%) contents in rattan than those of wood (Tables 2 and 3). In wood, lignin had the fastest
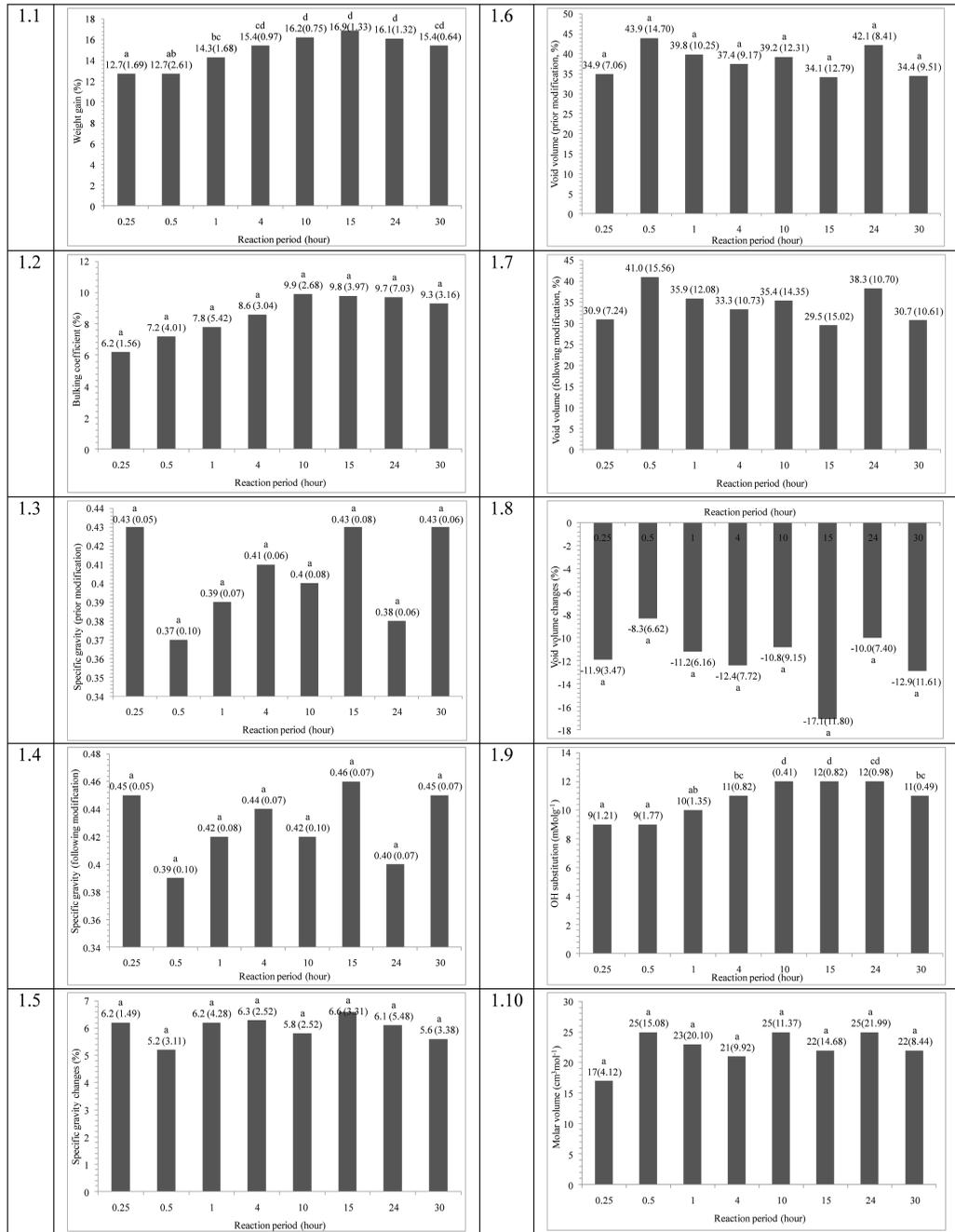
Fig. 1 The physical properties of acetylated rattan aged 10 years used for static bending test.
1.1 Weight gain 1.2 Bulking coefficient 1.3 Specific gravity (prior modification) 1.4 Specific gravity (following modification) 1.5 Specific gravity changes (%) 1.6 Void volume (prior modification) 1.7 Void volume (following modification) 1.8 Void volume changes (%) 1.9 OH substitution (mMolesg^{-1} 1.10 Molar volume (cm^{3}mol^{-1}). The parenthesis is standard deviation. Means followed by the same letter(s) in the same bar are not significantly different at the

TABLE 1
The mean physical properties of the acetylated rattan strip used for static bending test

| Properties | Rattan age (years) | | DF | F | Significance |
	10	13			(Rattan age)
Weight gain (%)	15.0 (2.07)	15.7(2.27)	1	2.94	0.09[Ns]
Bulking coefficient (%)	8.6(4.15)	7.5(2.95)	1	2.62	0.11[Ns]
Specific gravity (prior to modification)	0.40(0.07)	0.37(0.06)	1	5.62	0.02[*]
Specific gravity (following modification)	0.43(0.08)	0.40(0.07)	1	3.35	0.07[Ns]
Specific gravity changes (%)	6.0(3.27)	7.7(2.78)	1	8.67	0.004[**]
Void volume (prior to modification, %)	38.4(10.65)	42.9(9.17)	1	5.61	0.02[*]
Void volume (following modification, %)	34.5(12.10)	38.4(10.27)	1	3.35	0.07[Ns]
Void volume changes (%)	-11.8(8.20)	-11.3(6.32)	1	0.13	0.72[Ns]
OH substitution (mMolesg^{-1})	11(1.47)	11(1.61)	1	3.06	0.08[Ns]
Molar volume (cm^{3}mol^{-1})	23(13.71)	20(8.67)	1	1.60	0.21[Ns]
Final moisture content (%)	4.4(0.61)	4.1(0.62)	1	9.16	0.003[*]

[*]is significant at $P<0.05$. [**] is significant at $P<0.01$ probability levels. Ns-is not significant. The parenthesis is standard deviation. DF is degree of freedom, F is F distribution.

TABLE 2
The mean values of the chemical composition in rattan

| Chemical composition | Rattan age (years) | | DF | F | Significance |
	10	13			(Rattan age)
Holocellulose (%)	84.0(5.03)	86.3(1.79)	1	5.63	0.02[*]
α-cellulose (%)	70.1(5.71)	73.3(4.67)	1	5.62	0.02[**]
Hemicellulose (%)	14.5(6.12)	13.0(4.07)	1	1.33	Ns
Lignin (%)	19.4(5.86)	17.3(5.32)	1	2.18	0.1[*]

[*]is significant at $P<0.05$, [**] is significant at $P<0.01$ probability levels. Ns-is not significant. The parenthesis is standard deviation. DF is degree of freedom, F is F distribution.

TABLE 3
The mean values of the chemical composition in wood

| Chemical composition | Types of wood | | | |
	Hardwood[1]	Softwood[1]	European beech[2]	Pine[3]
α-cellulose (%)	42±2	45±2	49.1	52.2
Hemicellulose (%)	27±2	30±5	36.5	22.1
Lignin (%)	20±4	28±3	23.8	26.3

[1]Dinwoodie (2000), [2]Kurschner and Melcerova (1965), [3]Kollmann and Fengel (1965)
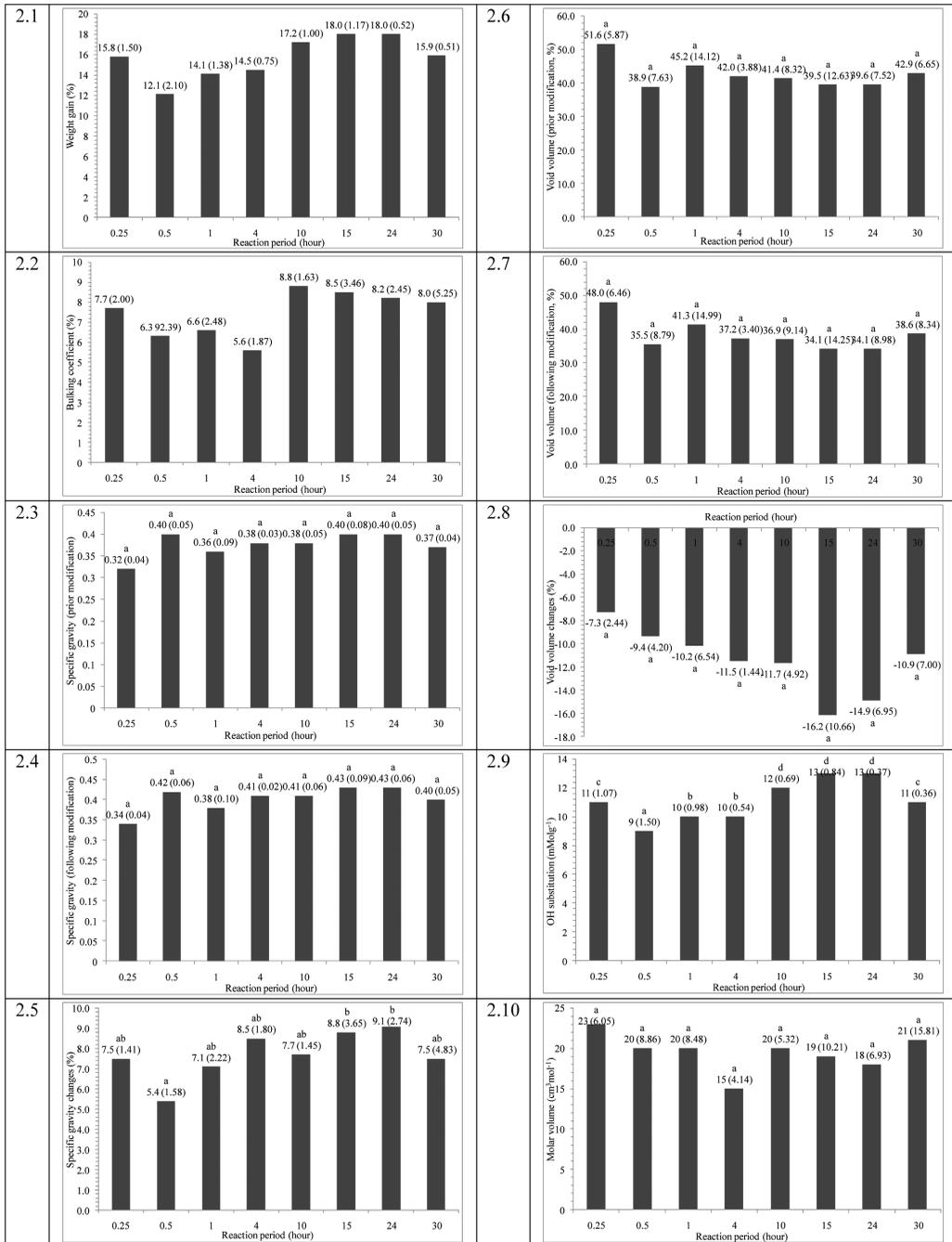
Fig. 2 The physical properties of acetylated rattan aged 13 years used for static bending test.
2.1 Weight gain 2.2 Bulking coefficient 2.3 Specific gravity (prior modification) 2.4 Specific gravity (following modification) 2.5 Specific gravity changes (%) 2.6 Void volume (prior modification) 2.7 Void volume (following modification) 2.8 Void volume changes (%) 2.9 OH substitution (mMolesg^{-1} 2.10 Molar volume (cm^{3}mol^{-1}). The parenthesis is standard deviation. Means followed by the same letter(s) in the same bar are not significantly different at the

reaction with acetic anhydride, followed by hemicellulose and lastly the cellulose (Rowell, 1983). As much of the cellulose is likely to be crystalline, it is inaccessible within the crystalline core for reaction. The time for the levelling-off WG of rattan of either age (10 hours) was faster than in the case of hinoki wood (uncatalysed, about 25 hours) reacted at 125ºC (Li *et al.,* 2000), but not as fast as Sitka spruce (uncatalysed, about 2.5 hours) reacted at 120ºC (Minato & Ogura, 2003). The differences can be explained by the kinetic perspective resulted from different modification procedures. The kinetic of the reaction depends on the access of the reagent to the reaction site and the real chemical reaction (Minato & Ogura, 2003). The difference of the maximum WG between rattan and wood certainly causes the different values of their physical properties.

Variation of the Static Bending Properties

The MoR and MoE varied by age and reaction period (Table 4). In the younger acetylated rattan, the MoR and MoE were not significantly different by reaction period. They ranged from 34.87 MPa to 45.88 MPa and from 1091 MPa to 1452 MPa, respectively.

In the older acetylated rattan, the MoR values (but not for the MoE) were significantly different with reaction period. The MoR was significantly lower after 0.25 hour reaction (20.49 MPa) as compared to the untreated (23.00 MPa) but the value successively increased to the maximum (34.02 MPa) up to four hours of reaction,

and then steadily declined with longer reaction times, i.e. 28.35 MPa after 30 hours of reaction. This gave the maximum MoR improvement of 48% (4 hours of reaction) in the older acetylated rattan. In most cases, the MoR and MoE were higher in the younger rattan than the older rattan, regardless of the reaction period. This might be a feature of the higher initial MoR and MoE in the untreated younger rattan. The untreated younger rattan had average MoR and MoE values of 40.94 MPa and 1353.14 MPa, while these were 23.00 MPa and 909.29 MPa in the older untreated rattan, respectively.

The correlation test (Table 5) was performed in order to determine the relationship between the static bending and physical properties of acetylated rattan. The results showed that improvements of MoR and MoE in acetylated rattan were highly correlated with specific gravity of the specimens following modification, as depicted by the highest R^2 values. In contrast, the factor which was the most inversely related to MoR and MoE of the acetylated rattan was percent void volume following modification. However, the final moisture content did not influence the MoR and MoE of the acetylated rattan, despite being significantly higher in the untreated rattan. These trends were consistent for both ages.

The work to the maximum load was not significantly different by reaction period for either rattan age. Generally, younger rattan had a significantly higher work to the maximum load than the older rattan,

TABLE 4
The mean values of the static bending properties

| Properties | Reaction (h) | Rattan age (years) | | DF | F | Significance |
		10	13			(Rattan age)
Static bending	0	40.94[a]	23.00[ab]			
(Modulus of rupture, MPa)	0.25	45.88[a]	20.49[a]			
	0.5	37.41[a]	30.07[bc]			
	1	38.75[a]	31.01[bc]			
	4	37.88[a]	34.02[c]			
	10	36.40[a]	31.32[bc]			
	15	43.51[a]	28.85[abc]			
	24	34.87[a]	28.55[abc]			
	30	40.28[a]	28.35[abc]			
	Average	39.27	28.93	1	42.60	0.00[*]
Static bending	0	1353.14[a]	909.29[a]			
(Modulus of elasticity, MPa)	0.25	1452.00[a]	833.14[a]			
	0.5	1120.00[a]	958.00[a]			
	1	1114.71[a]	1015.29[a]			
	4	1168.29[a]	1043.71[a]			
	10	1131.00[a]	973.86[a]			
	15	1287.86[a]	928.57[a]			
	24	1091.00[a]	870.86[a]			
	30	1139.57[a]	897.71[a]			
	Average	1186.09	938.22	1	37.34	0.00[*]
Static bending	0	0.07[a]	0.05[a]			
(Maximum load, kN)	0.25	0.11[a]	0.04[a]			
	0.5	0.09[a]	0.07[a]			
	1	0.11[a]	0.07[a]			
	4	0.09[a]	0.08[a]			
	10	0.09[a]	0.08[a]			
	15	0.12[a]	0.07[a]			
	24	0.09[a]	0.08[a]			
	30	0.01[a]	0.06[a]			
	Average	0.10	0.07	1	29.00	0.00[*]

[*] is significant at 0.05 probability level. Means followed by the same letter(s) in the same column are not significantly different at the 0.05 probability level. DF is degree of freedom. F is F distribution.

TABLE 5
The correlation tests on the static bending of acetylated rattan

Properties	Modulus of rupture		Modulus of elasticity	
	10 years	13 years	10 years	13 years
Specific gravity (prior to modification)	0.89**	0.82**	0.86**	0.77**
Specific gravity (following modification)	0.90**	0.83**	0.88**	0.77**
Specific gravity changes	0.51**	0.13[Ns]	0.56**	0.30*
Void volume (prior to modification)	-0.89**	-0.82**	-0.86**	-0.77**
Void volume (following modification)	-0.90**	-0.83**	-0.88**	-0.77**
Void volume changes	-0.80**	-0.61**	-0.77**	-0.65**
Percent weight gain	-0.30*	-0.17[Ns]	-0.33*	-0.26*
Bulking coefficient	-0.58**	-0.25[Ns]	-0.64**	-0.50**
OH substitution	-0.29*	-0.17[Ns]	-0.32.	-0.26*
Molar volume	-0.71**	-0.51**	-0.75**	-0.67**
Final moisture content (following test)	0.17[Ns]	-0.23[Ns]	0.32*	-0.01[Ns]

* correlation is significant at 0.05 probability level, **is significant at 0.01 probability level.

regardless of the reaction period. The averages were 0.10 kN and 0.07 kN in 10 and 13 years old rattan, respectively.

Overall, this indicates that the acetylation of rattan conducted in either short or prolong reaction periods did not significantly degrade the static bending properties, which probably because the reaction was performed at below 120°C. In the acetylation of solid wood, Rowell (1983) mentioned that the upper limit of 120°C was commonly used to minimise wood degradation.

CONCLUSION

Acetylation alters the physical properties of rattan. The trends of the physical properties were found to be almost identical for the acetylated rattan of both ages. Meanwhile, the reaction period only influenced the weight gain and OH substitution of rattan of both ages. The acetylation of rattan at the temperature of 110°C does not significantly impair the static bending properties even though the reaction period is extended up to 30 hours.

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