EQUILIBRIUM MOISTURE CONTENT AND MOISTURE EXCLUSION EFFICIENCY OF ACETYLATED RATTAN (*CALAMUS MANAN*)

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HISHAM HN, HALE M & NORASIKIN AL. 2014. **Equilibrium moisture content and moisture exclusion efficiency of acetylated rattan (*Calamus manan*).** The reduced equilibrium moisture content (EMC_R) and moisture exclusion efficiency (MEE) of acetylated rattan was studied in relation with age and time of acetylation. Rattans aged 10 and 13 years grown under rubber trees were reacted with acetic anhydride for 0.25 to 30 hours. Results showed that the lowest EMC_R and the highest MEE were obtained after 10 hours reaction or at the levelling-off per cent weight gain for both rattan ages. The EMC_R values at the levelling-off per cent weight gain were lower in acetylated older rattan than the younger rattan. The low EMC_R was highly correlated with the bulking coefficient.

Keywords: Acetylation, moisture adsorption, equilibrium moisture content reduced, per cent weight gain

HISHAM HN, HALE M & NORASIKIN AL. 2014. **Kandungan keseimbangan kelembapan dan kecekapan pengurangan dalam kandungan keseimbangan kelembapan oleh rotan (*Calamus manan*) terasetil.** Kandungan lembapan keseimbangan terturun (EMC_R) dan kecekapan pengurangan dalam kandungan keseimbangan kelembapan (MEE) bagi rotan terasetil dikaji berdasarkan usia serta masa pengasetilan. Rotan berusia 10 tahun dan 13 tahun yang ditanam di bawah pokok getah dibiarkan untuk bertindak balas dengan asid asetik anhidrida selama 0.25 jam sehingga 30 jam. Hasil kajian menunjukkan bahawa EMC_R yang terendah dan MEE yang tertinggi diperoleh selepas 10 jam tindak balas atau pada pengarasan peratus pertambahan berat bagi kedua-dua usia rotan. Nilai EMC_R pada pengarasan peratus pertambahan berat adalah lebih rendah pada rotan terasetil tua berbanding dengan rotan yang lebih muda. Nilai EMC_R rendah berkait rapat dengan pekali pemukalan.

INTRODUCTION

Calamus manan is the most important species for rattan industries due to its superior mechanical properties and higher dimensional stability compared with *C. ornatus* (Hisham & Uyup 2005). The stem of *C. manan* consists of vascular bundle with one metaxylem vessel, two phloem fields and protoxylem. The parenchyma and fibres are other cells encompassing the vascular bundle. Methaxylem vessel is the largest cell in *Calamus* species, with average diameter of 300 to 350 µm (Weiner & Liese 1988). During service, these voids could be easily penetrated by any surrounding water and water vapour which could alter the dimensional stability of debarked cane and any manufactured products. Dimensional instability of cane and its products will develop defects such as cracks and twists. Moisture adsorption may also lead to corrosion of nail or screw joints and colonisation of decay organisms. Dimensional stability of rattan needs to be improved for better service life. Rattan is currently used as parquet flooring, shoe platform, handbag and handles for hammer and hoe, replacing the more expensive and limited tropical timber.

Chemical modification is reported to improve dimensional stability and biological resistance of wood. This treatment is considered as non-toxic to human compared with conventional wood preservative (Rowell 1991, Hon 1996). The toxicity problem in chemically-modified wood may be shunned if non-toxic compounds

are attached into the wood structure (Thomas & Peterson 1978).

Wood treated with chemicals such as methyl ether, epoxides and aldehyde degrades its mechanical properties compared with untreated wood (Kumar 1994). However, modifying wood with acetic anhydride improves dimensional stability as well as its biological and weathering resistances but gives little effect on the strength properties of wood (Hill 2006, Rowell 2006). Several factors contribute to dimensional stabilisation of acetylated wood. The first is primarily due to the bulking effect of the acetyl groups, which causes increases in dry dimensions without increasing wet dimensions, i.e pre-swelling. The second is caused by reduction in availability of hydrogen bonding sites, mainly matrix OH groups. The third is the bulking effect or volume occupied by the bonded acyl adducts (Stamm & Tarkow 1947, Ramsden et al. 1997, Hill 2006).

Unlike Sitka spruce, Corsican pine, rubberwood and Scots pine, the acetylation of rattan has not been investigated yet. In general comparison, acetylation of cultivated rattan is economically feasible as the cost of rattan and acetic anhydride is lower or similar to tropical heavy hardwood timber. The use of acetylated rattan as alternative to timber could reduce the demand for tropical heavy hardwood. The objectives of this study were, thus, to examine the equilibrium moisture content and moisture excluding efficiency of acetylated rattan using vapour sorption test.

MATERIALS AND METHODS

Source of material

Calamus manan, aged 10 and 13 years, were obtained from small holder rubber tree plantations at Felda Mempaga (3° 31' N, 101° 55' E) in Pahang and Baranang (20° 56' N, 101° 52' E) respectively. Climate condition and other information of both plantations are displayed in Table 1.

Determination of anatomical properties

Rattan stem of each age was divided into five different portions of its total stem length, namely, basal, upper basal, middle, upper middle and top. Transversely, each circular rattan was then cut to yield a cross-shaped piece and then these were equally divided into five different sections, namely, periphery, outer, intermediate, inner and centre. Specimens from periphery, intermediate and centre sections were only selected for anatomical investigation. They were boiled in distilled water for 1 hour and sectioned with a microtome to a thickness of 15 to 25 μm. The specimens were rinsed with distilled water three times and dried for about 2 min. Two drops of safranin-O solution (1% in ethanol) were applied to the sections and the sections were dehydrated three times for each 80, 90 and 100% ethanol series. The bottom side of the slide glass was heated for a few seconds to remove air bubbles. A total of 70 vascular bundles of each portion and section were measured for the average diameter of metaxylem vessels.

Fibre and parenchyma morphologies were prepared by mixing the rattan with 40% glacial acetic acid, 40% hydrogen peroxide and 20% distilled water followed by heating at 60 to 70 °C for 5 to 7 days. After completion or when all fibres turned to whitish colour, they were rinsed with distilled water followed by staining with 1% alcoholic safranin-O solution and mildly teased apart in glycerine on a glass slide using fine needles. A total of 70 single fibre and parenchyma cells of each portion and section were measured for length, thickness and lumen diameter using a light microscope. The objective lens of 40× was found suitable for measuring all fibre and parenchyma dimensions, except for

Table 1　Climate and other information related to the plantation sites

Climate/information	Plantation site	
	Mempaga	Baranang
Month of harvesting	June	April
Total monthly rainfall (mm)	23.8	237.7
Average daily temperature (°C)	35.1	23.7
Type of soil	Laterite	Laterite

length where a 10× lens was used. The eyepiece graticule was calibrated using a stage micrometer (0.01 mm). Conversion values of 0.025 and 0.0025 mm were used for fibre length and other dimensions respectively.

Determination of chemical composition

Rattan particles were oven dried at 60 °C for 24 hours and ground with a hammer mill. Rattan flour was equilibrated to 15% moisture content and passed through sieves to retain the fraction of 0.4 to 0.1 mm. The methods used to determine holocellulose, α-cellulose and lignin contents were in accordance to Wise et al. (1946), Anonymous (1954) and Anonymous (1983) respectively. Six replicates of each chemical element were analysed for each rattan age.

Acetylation reaction

Rattans (> 35 mm in diameter from the middle to top portions of the stem) were used in this study. They were classified as weak rattan and had modulus of rupture (MOR) values below than 45 MPa as described by Bhat et al. (1992). They were cut into dimension of 20 mm × 20 mm × 5 mm (radius × thickness × length), Soxhlet extracted with toluene/methanol/acetone mixture (4:1:1) for 8 hours and oven dried at 103 °C for 24 hours. The Soxhlet extraction removed extractive contents in rattan. The presence of extractive in wood can have significant effect on the final per cent weight gain (Hill 2006). Specimens were transferred into desiccators and allowed to cool at ambient temperature over silica gel, weighed to 0.1 mg and the volume measured with digital bed micrometer. They were then vacuum-impregnated with acetic anhydride at room temperature followed by reaction at 110 °C. Reaction was continued for various time intervals (0.25, 0.5, 1, 4, 10, 15, 24 and 30 hours) to give a variety of per cent weight gain values. Specimens with the longest reaction time were inserted at the start followed by specimens with shorter reaction times. At the end of the reaction period, the reaction was quenched in ice until the liquid temperature reached 20 °C. 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 8 hours and oven dried at 103 °C for 24 hours. This procedure was sufficient to remove all unreacted acetic anhydride and by-product of acetic acid (Hill & Jones 1999). Dry specimens were cooled, weighed and measured as described above.

Determination of physical properties

Weight gain and bulking coefficient of acetylated rattan were calculated using the following formulae:

$$\text{Weight gain (\%)} = [(W_m - W_{um})/W_{um}] \times 100$$

$$\text{Bulking coefficient (\%)} = [(V_m - V_{um})/V_{um}] \times 100$$

where W_m = mass of modified rattan, W_{um} = mass of unmodified rattan, V_m = volume of modified rattan and V_{um} = volume of unmodified rattan.

Determination of theoretical hydroxyl group substitution

The theoretical OH substitution was calculated using the following formulae (Hill & Jones 1999):

$$\text{Theoretical OH groups substitution (mmol g}^{-1}) = [(W_m - W_{um})/W_{um}](MW\text{-}1) \times 1000$$

where MW = molecular weight of acetyl group.

Determination of reduced equilibrium moisture content

Different relative humidities were achieved using saturated salt solutions. Six types of salts were chosen in this study and each solution was prepared by stirring an excess of the salt in 100 mL of deionised water to achieve saturation. The saturated solutions and their salts were placed in glass bottles with plastic cap (48 mm and 58 mm of internal diameter and height) and stored in a controlled room set at 20 °C (variation ± 1 °C). Specimens were kept on a plastic mesh above each salt solution. When saturated solution at a controlled temperature is used, constant relative humidity is maintained (Siau 1995). The relative humidity of the atmosphere above each saturated solution at 20 °C (Kaye & Laby 1973) is displayed in Table 2.

Table 2   Saturated salt solutions and corresponding relative humidity values at 20 °C

Salt	Relative humidity (%)
Potassium nitrate (KNO_3)	93
Sodium chloride (NaCl)	76
Sodium dichromate ($Na_2Cr_2O_7$)	55
Potassium carbonate (K_2CO_3)	44
Magnesium chloride ($MgCl_2$)	33
Potassium acetate (CH_3COOK)	23

The salts were selected as they gave minimum relative humidity variation with changes in temperature (Stamm 1964). Five replicates of each age and reaction period were used. They were allowed to equilibrate over 5 to 8 weeks. Weight of specimen was measured once a week until constant weight was acheived which indicated that equilibrium moisture content (EMC) had been attained. Reduced equilibrium moisture content (EMC_R) and moisture exclusion efficiency (MEE) were calculated in accordance to Akitsu et al. (1993) and Hill (2006) respectively.

$$EMC_R \ (\%) = [(M_2 - M_1)/M_0] \times 100$$

$$MEE = [(E_u - E_m)/E_u] \times 100$$

where M_0 = oven-dry weight of specimen before modification, M_1 = oven-dry weight of specimen after modification, M_2 = weight of modified specimen at equilibrium with the atmosphere at a given relative humidity, E_u = EMC of unmodified wood and E_m = EMC of modified wood.

RESULTS AND DISCUSSION

Properties of acetylated rattan

The properties of acetylated rattan are listed in Table 3. Overall weight gain, bulking coefficient and OH substitution were not significantly different by age but they were significantly different with the reaction period. In younger acetylated rattan, the per cent weight gain was significantly increased from 5.4 (0.25 hour) to 9.7% (4 hours) and then levelled off at 13.5 to 13.8% (10 to 24 hours). Bulking coefficient significantly increased from 4.1 (0.25 hour) to 6.1% (1 hour), but it was not significantly different with 5.7% at 0.5 hour, and then levelled off at 7.7 to 8.7% (4 to 24 hours). Similarly, the theoretical OH substitution increased from

4 mmol g^{-1} (0.25 hour) to 5 and 7 mmol g^{-1} (1 and 4 hours and were significantly different), and then levelled off at 10 mmol g^{-1} (10 to 24 hours).

The older acetylated rattan behaved differently. The per cent weight gain fluctuated from 6.6 to 11.1% (0.25 to 1 hour) but levelled off at 10.3 to 10.9% (10 to 24 hours). Bulking coefficient was not significantly different with reaction period and ranged from 6.2 to 7.2%. The theoretical OH substitution also fluctuated between 2 and 8 mmol g^{-1} (0.25 and 14 hours) but gave a maximum of 15 mmol g^{-1} (15 hours).

Maximum per cent weight gain of acetylated rattan, i.e. 13.8%, was lower than acetylated wood (~25%) (Hill 2006). This is probably caused by 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 as obtained in the experiment than those of wood (Tables 4 and 5). Lignin had the fastest reaction with acetic anhydride followed by hemicellulose and lastly, the cellulose (Rowell 1983). The time for levelling-off per cent weight gain of rattan of either age, which was 10 hours, was faster compared with that of hinoki wood (uncatalysed, about 25 hours, 125 °C) (Li et al. 2000) but not as fast as Sitka spruce (uncatalysed, about 2.5 hours, 120 °C) (Minato & Ogura 2003). This can be explained by the kinetic perspective resulted from different modification procedures. Kinetics of the reaction depends on the access of reagent to the reaction site and the real chemical reaction (Minato & Ogura 2003). Maximum theoretical OH substitutions of rattan (10 and 15 mmol g^{-1} for 10 and 13 years old respectively) were higher than that of accessible OH group in Corsican wood (5.7 mmol g^{-1}) as reported by Hill and Jones (1999).

Reduced equilibrium moisture content of acetylated rattan

Table 6 shows the mean values of EMC_R at each relative humidity for untreated and acetylated rattan at both ages. Moisture contents increased from the lowest to the highest relative humidity. Overall, untreated younger rattan adsorbed more moisture than untreated older rattan which indicated its higher hygroscopicity. This was probably caused by higher hemicellulose content in the younger rattan (14.5%) compared with the older rattan (13.0%). Hemicellulose has the highest sorption capacity followed by cellulose

Table 3　　Mean values of the properties of acetylated rattan used for equilibrium moisture content reduced test

Property	Reaction (hours)	Age (years)		DF	F	Significance
		10	13			
Weight gain (%)	0.25	5.4 a	9.0 ab			
	0.5	5.4 a	6.6 a			
	1	7.0 b	11.1 b			
	4	9.7 c	9.4 ab			
	10	13.5 d	10.3 ab			
	15	13.8 d	10.7 b			
	24	13.7 d	10.9 b			
	Average	9.9	9.6	1	0.13	0.71 ns
Bulking coefficient (%)	0.25	4.1 a	6.6 a			
	0.5	5.7 b	6.3 a			
	1	6.1 b	6.2 a			
	4	7.7 c	6.2 a			
	10	8.7 c	7.2 a			
	15	8.3 c	6.9 a			
	24	8.3 c	6.5 a			
	Average	7.1	6.6	1	1.58	0.21 ns
OH substitution (mmol g^{-1})	0.25	4 a	6 a			
	0.5	4 a	2 b			
	1	5 b	8 b			
	4	7 c	7 b			
	10	10 d	7 b			
	15	10 d	15 c			
	24	10 d	8 b			
	Average	7	7	1	0.00	0.99 ns

Means followed by the same letter(s) in the same column are not significantly different at the 0.05 probability level, ns = not significant, DF = degree of freedom, F = F distribution

Table 4　　Mean values of chemical composition in rattan

Component	Age (years)		DF	F	Significance
	10	13			
Holocellulose (%)	83.95	86.26	1	5.63	0.02[*]
α-cellulose (%)	70.09	73.29	1	5.62	0.02[**]
Hemicellulose (%)	14.52	12.98	1	1.33	ns
Lignin (%)	19.42	17.29	1	2.18	0.1[*]

*significant at $p < 0.05$, **significant at $p < 0.01$, ns = not significant, DF = degree of freedom, F = F distribution

Table 5　　Mean values of chemical composition in wood

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

[1]Dinwoodie (2000), [2]Kollmann and Fengel (1965), [3]Kurschner and Melcerova (1965)

Table 6　Mean values of reduced equilibrium moisture content of *Calamus manan* at various relative humidity values

Age (years)	Reaction (hours)	Relative humidity (%)					
		23	33	44	55	76	93
10	0	14.01	13.34	14.94	17.03	26.26	52.22
	0.25	7.62	7.18	8.24	9.91	15.17	44.42
	0.5	6.21	6.53	7.66	9.00	14.06	29.46
	1	2.60	2.50	3.82	4.17	7.48	25.69
	4	9.23	8.33	9.45	10.75	15.38	34.30
	10	1.45	1.82	2.04	2.74	5.43	12.61
	15	4.44	4.15	5.11	5.76	10.14	22.36
	24	2.90	3.49	3.81	4.71	9.52	17.16
13	0	4.83	10.17	10.61	13.30	18.40	31.52
	0.25	1.90	1.87	2.69	4.13	6.48	11.21
	0.5	2.68	2.36	3.27	4.12	6.53	11.29
	1	1.70	1.79	2.59	3.49	6.14	11.11
	4	1.64	1.68	2.36	3.46	5.45	9.15
	10	1.12	1.07	1.70	2.83	4.58	7.54
	15	0.99	1.16	1.76	2.83	4.19	8.31
	24	1.91	1.61	2.20	3.39	5.23	9.46

and lignin (Runkel & Luthgens 1956, Christenses & Kelsey 1959). Anatomical properties might have also influenced the sorption capacity as the untreated younger rattan had larger metaxylem vessel (349.1 µm) as well as fibre (11.23 µm) and parenchyma lumen (31.1 µm) diameters which could provide more space for moisture accommodation. In contrast, the untreated older rattan had average metaxylem vessel, fibre and parenchyma lumen diameters of 249.2 µm, 10.09 µm and 28.8 µm respectively (Table 7).

Moisture content was highest at 4 hours (except at 93% relative humidity) and was lowest at 10 hours reaction period in the younger acetylated rattan. Meanwhile, moisture content was highest at 0.5 hour and was lowest at 10 hours (except at 23 and 76% relative humidity) reaction periods in the older acetylated rattan. However, the values of EMC_R at 23 and 76% relative humidity were not much different between 10 and 15 hours reaction periods in the older acetylated rattan. This indicated that the EMC_R was lowest at the levelling-off per cent weight gain for both ages (13.5 and 10.3% weight gain, reacted for 10 hours, Table 5). Overall, older acetylated rattan had lower EMC_R values than the younger acetylated rattan at the levelling-off per cent weight gain,

except at 55% relative humidity. Thus, the acetylated older rattan had better dimensional stability than the younger acetylated rattan. The lowest EMC_R at the levelling-off per cent weight gain was probably due to the OH groups in rattan being substituted by acetyl groups. Acetylation is a single site reaction which means that one acetyl group is substituted with one hydroxyl group with no polymerisation (Rowell 1983). Thus, acetylation changed the hydrophilic nature of untreated rattan to more hydrophobic property of acetylated rattan. In acetylated Corsican wood, the lowest EMC at all relative humidity levels was obtained at 22.5% weight gain (Papadopoulos & Hill 2003).

Linear regression analyses were performed between the highest values of EMC_R at 93% relative humidity and per cent weight gain, bulking coefficient and OH substitution. Results showed that bulking coefficient had the highest correlation with EMC_R for both ages, where the younger rattan gave higher correlation ($r^2 = 0.71$) than the older rattan ($r^2 = 0.55$) (Table 8). This finding was similar to acetylated Corsican pine (Papadopoulos & Hill 2003) and acetylated elm and maple (Papadopoulos 2005). Other physical properties had lower correlation with EMC_R.

Table 7   Mean values of the anatomical properties of *Calamus manan*

Property/component	Age (years)		DF	F	Significance
	10	13			
Metaxylem vessel diameter (µm)	349.1	249.2	1	50.89	0.00*
Fibre					
Length (mm)	1.05	1.23	1	31.62	0.00*
Diameter (µm)	15.82	15.32	1	1.63	0.20 ns
Wall thickness (µm)	4.84	5.32	1	9.53	0.00*
Lumen diameter (µm)	11.23	10.09	1	12.49	0.00*
Parenchyma					
Length (µm)	768.9	680.3	1	11.15	0.00*
Diameter (µm)	35.8	33.4	1	7.67	0.01*
Wall thickness (µm)	4.7	4.6	1	0.04	0.84 ns
Lumen diameter (µm)	31.1	28.8	1	8.05	0.01*

*Significant at 0.01 probability level, ns = not significant, DF = degree of freedom, F = F distribution

Table 8   Regression coefficient of equilibrium moisture content

Property	Age (years)	Regression	
		r^2	p
Weight gain	10	0.63439	0.0321
Bulking coefficient		0.71377	0.0167
OH substitution		0.63436	0.0321
Weight gain	13	0.22099	0.2871
Bulking coefficient		0.55293	0.0554
OH substitution		0.24196	0.2622

r^2 = coefficient of determination, p = probability

Moisture exclusion efficiency of acetylated rattan

MEE of acetylated rattan decreased with increasing relative humidity levels (Table 9). On average, younger rattan had higher MEE than older rattan at the lowest relative humidity levels (23%). In contrast, older rattan had higher MEE than the younger rattan at relative humidity level of ≥ 33%. Generally, rattan at 24 hours reaction period had slightly higher MEE than those at 15 and 10 hours, except for values at 23% relative humidity levels (both ages). However, MEE values were not much different between those reacted for 10, 15 and 24 hours, except at 23% relative humidity. This indicated that optimum MEE at all relative humidity levels was achieved at the levelling-off per cent weight gain (13.54 and 10.26% for 10 hours reaction for 10 and 13 years respectively). Maximum values of MEE obtained at levelling-off per cent weight gain ranged from 73.54 to 84.83% and from 74.28 to 89.15% for both ages respectively.

CONCLUSIONS

Acetylation reduced equilibrium moisture content and increased moisture exclusion efficiency of cultivated rattan. Lowest equilibrium moisture content and highest moisture exclusion efficiency were obtained at the levelling-off per cent weight gain, which was higher in the younger than the older rattan. The reduction of equilibrium moisture content of acetylated rattan was correlated with bulking coefficient.

Table 9　Mean values of moisture exclusion efficiency (MEE)

Age (years)	Reaction (hours)	MEE (%)					
		23	33	44	55	76	93
10	0.25	29.04	31.42	31.56	41.48	31.89	28.66
	0.5	66.50	66.17	64.65	63.08	61.88	59.19
	1	74.27	74.75	70.49	67.69	61.97	69.18
	4	70.19	74.21	71.23	69.78	67.11	60.23
	10	84.38	80.95	81.29	79.08	73.89	73.54
	15	80.64	78.47	75.51	76.85	72.97	71.90
	24	89.84	82.12	81.98	79.65	70.01	75.02
	Average	73.17	70.11	68.60	69.06	63.10	63.15
13	0.25	56.00	80.14	72.83	68.27	64.29	65.23
	0.5	38.08	78.09	70.27	70.47	66.02	65.40
	1	67.67	80.40	74.44	73.62	67.35	66.50
	4	66.42	83.01	77.81	74.73	71.64	72.27
	10	74.28	89.15	83.76	79.40	76.24	77.26
	15	77.58	89.06	84.06	80.76	76.01	76.25
	24	59.87	91.21	88.39	83.25	77.14	76.11
	Average	63.33	84.49	78.82	75.85	71.40	71.48

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