Influence of Microfibril Angle on Thermal and Dynamic-Mechanical Properties of *Acacia mangium* Wood Using X-Ray Diffraction and Dynamics-Mechanical Test

Tamer A. Tabet and Fauziah Haji Abdul Aziz

***Abstract*—The term micofibril angle, MFA in wood science refers to the angle between the direction of the helical windings of cellulose microfibrils in the secondary cell wall, S2 layer of fibres and tracheids and the long axis of the cell. In this study, the main MFA of the cell walls were determined for thin samples of thickness 200µm from pith and outwards, for eight ages of *Acacia mangium* wood. The determination of MFA was based on diffraction pattern arising from cellulose crystal planes of the type 002 generated by x-ray diffraction and recorded using an electronic detector. The results show an inversely relationship between MFA and age of tree in *Acacia mangium* wood. MFA decreased from 26.13° at age 3 year-old to 0.20° at tree of age 15 year-old for the pith region. . The most significant drop occurred from 16.14° at age 7 year-old to 11.30° at age 9 year-old. An inversely relationship between MFA and storage modulus *E'* was evidence in *Acacia mangium* at age 10-year-old. The results showed that about 76.22% variation of loss modulus *E"* was attributed to the MFA, while about 66.4% of the variation of glass transition T_g was explained by MFA under the same experimental condition.**

***Index Terms*—*Acacia mangium*, Microfibril angle, MFA, Glass transition, X-ray diffraction.**

I. Introduction

The orientation of the cellulose microfibrils in the S_2 layers of the cell walls of softwood has a significant influence on the mechanical properties of wood. The angle between the cellulose fibrils and the longitudinal cell axis, the microfibril angle, MFA was found to be a critical factor in determining the physical and mechanical properties of wood [8]. For this reason, considerable effort has been directed towards the measurement of the cellulose MFA. Direct measurement of MFA has been made by highlighting microfibrils in individual cell walls with iodine staining, but the most widely adopted techniques use either wide-angle X-ray diffraction or small-angle X-ray scattering [19]. The pioneering work of references [3] and [18] led to the use of the '*T*' parameter derived from the curve distribution of the intensity diffracted by the (002) planes of the cellulose fibrils.

First Author isTamer A. Tabet. Forest Plantation and Agroforestry Program, School of International Tropical Forestry, Universiti Malaysia Sabah, e.mail: tamertbt@ums.edu,my. Tel: 006-088-320000 ext:8593
Second Author: Fauziah Hj Abdul Aziz, School of Science and Technology, Universiti Malaysia Sabah e. mail: afauziah@ums.edu.my. Tel: 006-088-320000 ext: 5792.

MFA has been found to influence shrinkage of wood [15]. The MFA of the S_2 layer in the tracheid cell wall is known to be one of the main determinants of the mechanical properties of wood [4]. Reference [9] reported that the MFA also had a significant impact on paper properties. The MFA has a very strong influence on the stiffness of wood [21]. MFA in the S_2 layer of the cell wall of *Picea abies* has been to influence on cambial age and growth conditions [14].

II. Thermal and Dinamic-Mechanical Properties of Woo

Thermal analysis has been extensively applied to investigate the thermal behavior of various materials as a function of temperature [9]. A number of researches on thermal properties of wood fiber and polymer composites (WFPCs) have been reported [10]. Crystallization and morphology in WFPCs have been investigated with many thermal methods by a number of research workers [12]. Weight or volume ratios of wood fiber greatly influenced glass transition temperature and storage moduli of the resultant composites [13].

A number of different methods have been used to investigate thermal properties and vescoelastic properties of wood. One such method is dynamic mechanical thermal analysis (DMTA). This has been used to investigate wood from different trees species [9]. The specimens were tested in the shearing and bending modes in the air dried state, show a number of overlapping *tan* δ peaks which are thought to represent the glass transitions of hemicelluloses and lignin components within the wall matrix [1]. Reference [2] identified two δ peaks in temperature scan ranging from 150 to 150 Cº. However, in all cases *tan* δ never fell below 0.1 and above -50 Cº was always above 0.02. DMTA is useful tool for investigation properties of secondarily thickened cell walls, and a long with other methods, is beginning to show just how complex the wood structure architecture [16]. One of the objectives to work was to examine the influence of MFA of the cell wall on the thermal and dynamic-mechanical properties of *Acacia mangium* wood.

III. Materials and Methods

The wood samples used in this study were selected from 3, 5, 7, 9, 10, 11, 13 and 15 year-old of *Acacia mangium* tree from Sabah Forestry Development Authority (SAFODA) and

ISBN: 978-988-18210-7-2
ISSN: 2078-0958 (Print); ISSN: 2078-0966 (Online)

Ganui Plantations, Sabah, Malaysia. The standard methodology for the physical characterization followed the International technical standard (ISO standard 4471-1982). The samples were taken out at breast height (1.25m) on each tree. An x-ray diffractometer (Philips X-Pert PRO PW3040/60) was used to determine the average microfibril angle. A point-focused x-ray beam (Cu-Kα x-ray, beam diameter 1 mm) was applied to tangential section, 200 µm thick x 2 mm long, prepared from the pith and bark regions with a sliding microtome as shown in Figure 2b. Before proceeding further, it is necessary to define the geometry of the diffraction diagram-samples system. Geometry of diffraction-samples system as indicated in (Fig.1):

(a) The sample axis is taken parallel to the vertical Z axis of the rectangular cartesian set (X, Y, Z).

(b) The incident X-ray beam is coincident with the positive x axis.

(c) The microfibril axis direction is given by the polar or MFA, µ, the angle the microfibril makes with the z axis, and the azimuthal angle α, the angle the projection of the microfibril on to the x y plane makes with the positive x axis.

(d) The position of the reflection spot on the (002) reflection circle is given by the angle ø measured from the right hand equatorial (positive y) axis towards the upper meridional (positive z) axis.

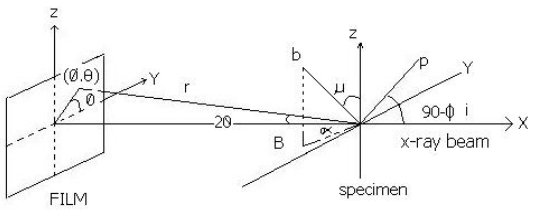

Fig.1. Geometry of the specimen and X-ray diffraction system, p is the polar of a specimen reflecting plane, θ is the Bragg angle of reflection and Φ the reflection spot angle on the X-ray diagram, µ is the microfibril angle.

The measurements were made at a speed of 6 degrees per minute, at a Bragg's angle 22.4°, using the 2 mm diverging slit and 1 mm receiving slit. A diffraction pattern is produced by the crystalline structure and recorded by an electronic detector. Parameter *T* defined by Cave, was obtained from the diffraction intensity around (002) arc [3]. Three lines were drawn to derive half the width of the curve. The first was the baseline representing the portion in the curve when the x-ray intensity was more or less minimal. Than, a tangent was drawn to divide the curve in to two equal parts. The average MFA was calculated using the following formula [13].

$$MFA = 1.575\times10^{-3}T^3 - 1.431\times10^{-1}T^2 + 4.693T - 36.19 \quad (1)$$

Radial slices 50 µm thick were cut by rotary microtome, LEICA RM from all eight ages and used for Probe microscope.

Samples for Dynamic Mechanical Analyzer DMA testing were of the impact using a table saw. They were further machined down to nominal thickness of 3.0 mm using vertical milling machine. The samples were held in place under controlled humidity and temperature. Care was taken to obtain samples from the same area of the impact region in the wood trunk. Each disc of wood was machined to produce a balance DMA samples desired thickness. The final samples dimensions were 50 mm × 13 mm × 3 mm.

IV. RESULTS

A. *Determining of MFA*

Figure 2 shows typically diffraction pattern arising from the region of *Acacia mangium* wood as a slice samples of thickness 200µm from growth age 3 year-old. The MFA is determined from the intensity that has strongest peak [20]. In this work, the intensity peak at $2\theta \approx 22°$ has been used to calculate the parameter *T* for all growth ages because the peak of the diffraction intensity gives the best Full Width Half Maximum, FWHM value in this case. In case of wood sample from 3- year- old tree, the peak at $2\theta = 22.4973°$ was used to calculate the MFA. Fig. 2 shows that angle $2\theta = 22.4973°$ gives the FWHM about 0.5196° for wood sample of 3-year-old.

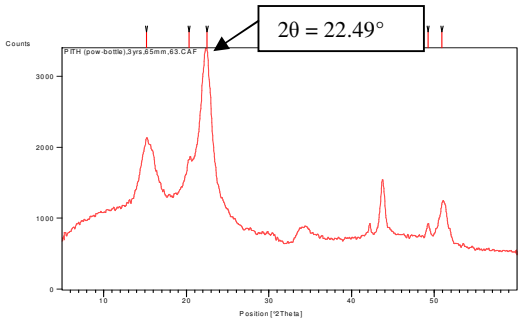

Fig. 2. Typical diffracting pattern arising from pith region of *Acacia mangium* sample at 3-year-old.

The parameter *T* was used as an indicator to MFA. *T* was calculated using Cave method [4]. Mean MFA was calculated using the formula developed by Yamamoto *[22]*. Three lines were drawn to derive half the width of the curve. The first was the baseline representing the portion in the curve where the X-ray intensity was more or less minimal. Than a tangent were drawn through the infection point on one side of the curve. Finally a vertical line was drawn to divide the curve in to two equal parts as shown in "Fig. 3". The results shows that the MFA ranged from 26.13° at the pith region of *Acacia mangium* wood at age 3 year-old, and decreases to about 0.20° ± 0.01° at tree age 15 year-old. It was found that the mean MFA at the bark region of *Acacia mangium* wood behaves the same way. MFA and standard deviation for *Acacia mangium* from 3 year-old at bark region was calculated using the polynomial relationship of Yamamoto [22].

$$MFA = 1.575\times10^{-3}T^3 - 1.431\times10^{-1}T^2 + 4.693T - 36.19$$

$T = 2.3° \pm 0.1°$ for growth age of 3-year-old as shown in Fig.3

$$MFA = 1.575\times10^{-3}(2.3)^3 - 1.431\times10^{-1}(2.3)^2 + 4.693(2.3) - 36.19$$
$$MFA = 0.0191 - 0.7569 + 10.7939 - 36.19 = -26.13^o$$
$$MFA = -26.13^o$$

ISBN: 978-988-18210-7-2
ISSN: 2078-0958 (Print); ISSN: 2078-0966 (Online)

The (-) signal mean that the microfibrils orient in the back cell wall.

The parameter *T* given by Cave (1966) by the formula:

$$T = MFA + 2\sigma \quad (2)$$

σ = 14.21°

Were σ = 14.21° represent the standard deviation of the intensity distribution arising from the fibril orientation about the mean value.

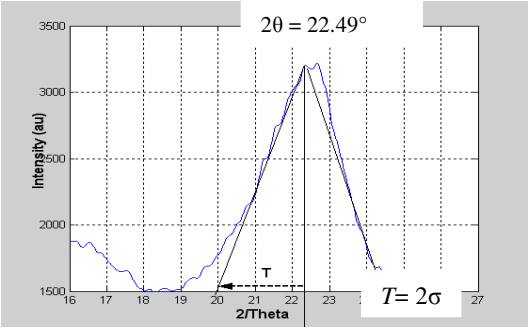

Fig. 3. X-ray diffraction intensity used to measurement of *T* in wood model of 3 year-old.

The results of MFA for pith and bark region of the tree under investigation are summarized in Table 1 and 2 respectively. Overall MFA was found to decrease when the growth age increased in all discs. It was found that MFA decreases from 26.13º at growth age about 3 year-old to 0.20º at the pith center of age 15 year-old (Table 1). In the bark region, MFA ranged from 31.26º at age 3 year-old to 0.47 at age 15 year-old.

MFA decreased as the tree age increase. Most significant drop occurring from 21.45º at age 5 year-old to 16.14º at age 7 year-old, and from 9.80º at 10 year old 4.96º at 11 year-old at the pith region. The smallest value of MFA was found in the pith center, MFA = 0.20º ± 0.01º. An inverse relationship between MFA and tree age was evident in this study within the pith region "Fig. 4". The MFA of *Acacia mangium* in the bark region behaves the same way. The highest rates of decreasing of MFA occur between ages 5 to 7 year-old within the bark region. MFA was found to drop from 27.36º at 5 year old to 17.83º ± 0.01º at age 7 year old. An inverse relationship between tree age and MFA was found within the bark region "Fig. 5". A strong positive direct relationship ($R^2 = 0.98$) has been found between MFA and the distance from pith to bark in wood model at age 10 year old "Fig. 6".

Table 1: The value of MFA and standard deviation σ in the pith region for each age of *Acacia mangium* tree.

Sample No.	Tree age (year)	MFA ± 0.01°	σ (°)	Microfibrils orientation
1	3	26.13	14.21	Back cell wall
2	5	21.45	8.97	Back cell wall
3	7	16.14	5.47	Back cell wall
4	9	11.30	2.40	Back cell wall
5	10	9.80	1.40	Back cell wall
6	11	4.96	4.27	Front cell wall
7	13	0.26	0.08	Front cell wall
8	15	0.20	0.07	Front cell wall
9	15 (Pith center)	0.20	0.07	Front cell wall
		Mean= 10.05	Mean= 4.10	
		Std. Dev= 9.64	Std. Dev= 4.83	

Table 2: The value of the MFA and the standard deviation in the bark region for each age of *Acacia mangium* tree.

Sample No.	Tree age (year)	MFA ± 0.01°	σ (°)	Microfibrils orientation
1	3	31.62	15.13	Back cell wall
2	5	27.36	12.68	Back cell wall
3	7	17.83	6.60	Back cell wall
4	9	14.44	4.47	Front cell wall
5	10	9.87	1.43	Front cell wall
6	11	5.67	1.42	Front cell wall
7	13	3.17	3.16	Back cell wall
8	15	0.47	0.15	Back cell wall
		Mean=13.80	Mean= 5.63	
		Std. Dev=11.27	Std. Dev= 5.53	

ISBN: 978-988-18210-7-2
ISSN: 2078-0958 (Print); ISSN: 2078-0966 (Online)

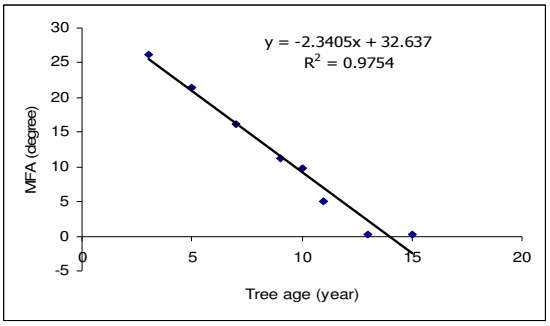

Fig. 4. The relationship between MFA and the tree age in the pith region of *Acacia mangium* wood.

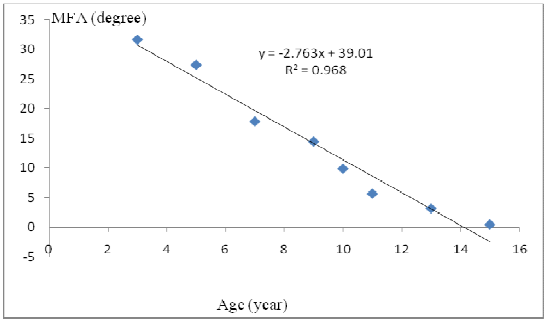

Fig. 5. The relationship between MFA versus tree age in the bark region of *Acacia mangium* wood.

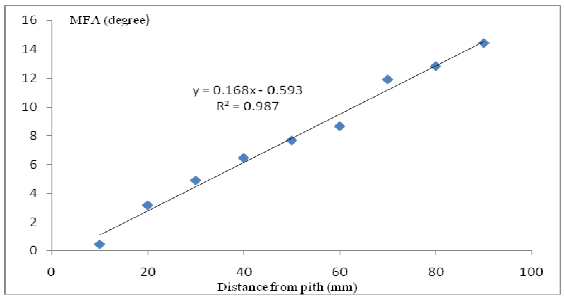

Fig. 6. The relationship between MFA and the distance from the pith in *Acacia mangium* wood at age 10 year- old

B. The Effect of Microfibril angle on the Thermal and Dynamic- Mechanical Properties of Acacia mangium Wood

In this study, the DMTA technique is used in glass transition calculation. Method was based on the temperature at which mechanical properties began to be compromised, that is the maximum damping ratio. This method is used to present the glass transition of *Acacia mangium* wood as a function to the MFA, because this is the method commonly found in the literature. The thermal and mechanical properties of *Acacia mangium* wood under analysis are presented in Table 3. The values of T_g were calculated based on the maximum damping ratio.

Table 3: Thermal and mechanical properties of *Acacia mangium* wood with respect to the MFA. The values of T_g were calculated based on the *tanδ.*

MFA (°)	*E'* (GPa)	*E"* (GPa)	Glass transition (°C)	*tanδ*
0.46	9.00×10^8	55129999	102.583	0.175
3.17	7.79×10^8	46028906	103.213	0.145
4.90	7.16×10^8	48354672	92.694	0.166
6.46	7.13×10^8	45717485	88.899	0.165
7.68	7.14×10^8	44826412	99.669	0.151
8.66	6.47×10^8	45953213	84.633	0.165
11.91	6.24×10^8	42198028	84.113	0.153
12.84	6.71×10^8	37456490	83.688	0.172
14.44	4.76×10^8	31983774	83.895	0.147

The effect of MFA on the storage modulus (*E'*), loss modulus (*E"*) and *tanδ* in the pith center for MFA about 0.46º is presented as shown from "Fig. 7, 8 and 9". A general declining trend for all curves of *E"* test is observed when the wood samples go through higher temperatures. The only noticeable transition can be detected at around 99.66 ºC as shown in Table 3. As it seen in "Fig. 7", wood sample at MFA 0.46º have higher T_g about 77.64 ºC based on the maximum of *E"*. No significant difference between the temperatures based on the change of storage modulus is observed. Result show also that, in the case of the temperature based on the *tanδ*, a significant different due to the increase of MFA can easily be seen (Table 3). It was found that T_g ranging between 102.583 ºC at MFA about 0.46º "Fig. 9", and 83.895 ºC at MFA about 14.44º as shown in Table 3. An inversely relationship has been established between MFA and *E"* for wood sample at age 10 year-old "Fig. 11".

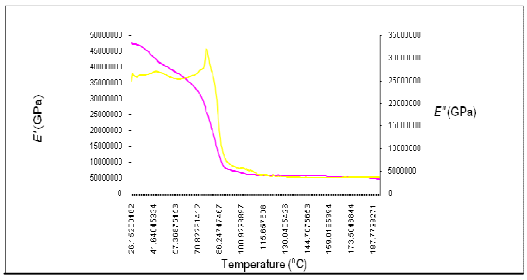

Fig.7. T_g determined by the onset of the *E'* change and the maximum of *E"* at MFA = 0.46°. The T_g value based on the change of *E'* = 26.072 °C while T_g based on the maximum of *E"* = 77.647 °C. The Frequency = 1 Hz.

ISBN: 978-988-18210-7-2
ISSN: 2078-0958 (Print); ISSN: 2078-0966 (Online)

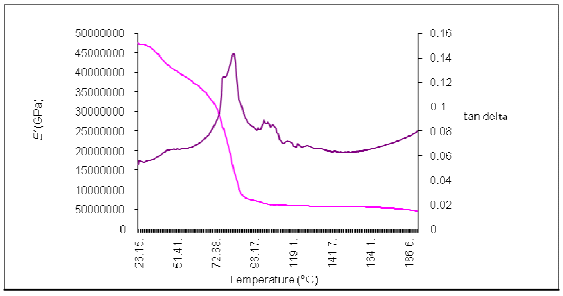

Fig. 8. The T_g value based on the change of E' = 26.072 °C while T_g based on the maximum damping ratio, $tan\delta$ = 102.593 °C, at MFA = 0.46°. The Frequency = 1 Hz.

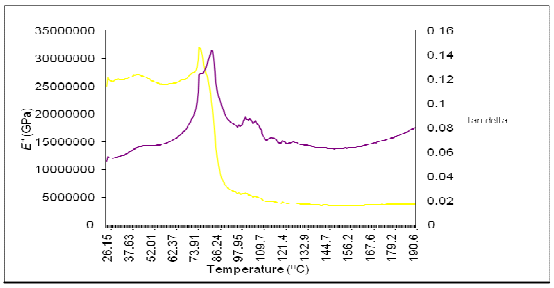

Fig. 9. The T_g value based on the maximum of E'' = 77.647 °C while T_g based on the maximum damping ratio, $tan\delta$ = 102.593 °C, at MFA = 0.46°. The Frequency = 1 Hz.

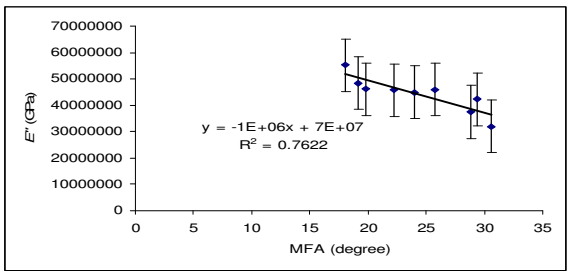

Fig. 10. The relationship between MFA and E'' in *Acacia mangium* wood of 10-year-old.

V. CONCLUSION

In this work, the variations of MFA with the tree ages were studied for the real wood cell wall structure of *Acacia mangium* using X-ray diffraction. The results of MFA measurements from the eight *Acacia mangium* trees, 3, 5, 7, 9, 10, 11, 13 and 15-year-old examined in this study showed that differences in MFAs between tree ages were significant. The general trend was for the MFA to be greatest in the young wood of age 3-year-old and decrease gradually with increasing the tree age. This trend has been found for both pith and bark region of the wood samples. It was found that for eight ages of trees examined, the MFA was at its greatest in the sample of 3-year-old, where angle as high as 26.13° were recorded. The lowest for all eight cases were taken from the pith region were found in tree age 15-year-old where the angle varied from 1.99° to 0.20° in the front cell wall direction of the microfibrils. For all cases of the wood samples were taken from the bark region of the eight trees, the results shows that the MFA was its greatest in tree age 3-year-old, were angle as high as 31.62° were recorded. Tables are numbered with Roman numerals. The lowest MFAs for the same samples were found in 15-year-old of the trees were the angle was 0.47° in the back cell wall direction of the microfibrils.

Results shows that MFA in *Acacia mangium* wood increases rapidly as a function of the distance from pith towards the bark in wood models were taken from 10 year-old. The general trend was for the MFA to be lowest near the pith and then to increase gradually towards the cambium. It was found that for the samples taken from 10 year-old trunk, the MFA was at its lowest at a distance 10.0 mm from the pith center, where angle as 0.46° was recorded. The highest MFA for all samples in the trunk was found at the distance 90.0 mm from the pith where the angle was 14.44° (Table 3). Obviously, the MFA has a strong correlation with the wood quality as well as with the physiomechanical function of tree [5].

When a tree is young it needs to be elastic in order to move in the wind. After some decades, however the cells produced by matured trunk cambium have a smaller MFA for stiffness and keeping the trunk upright. By contrast, young woods have to be rather elastic, allowing them to bend. Therefore, MFA need to be larger. The MFA is thus critical to the total mechanical balance of the tree, correct MFAs are essential for its survival. However, the results clearly show that greater variation exists across the distance from pith to bark and between the tree ages.

Effect of MFA on the storage modulus, loss modulus and glass transition of *Acacia mangium* wood is discussed in this study. Results show a good inversely relationship between MFA and the E' was evidence in *Acacia mangium* wood of 10-year-old. A general declining trend for all curves of E' test is observed when the wood samples go through higher MFA. The only noticeable variation can be detected in the case of MFA about 6.46° (Table 3). Thus, as the MFA increases the E' decreases. It was found that for a decrease in MFA from 14.44° to 0.46°, the E' increased from 4.76 $\times 10^8$ GPa to 9.00 $\times 10^8$ GPa.

The results also showed that E'' was most strongly influenced by MFA under the same experimental conditions. The model suggests that a similar reduction in MFA as outlined above, causes E'' to increase about 23,146,225.00 GPa. These results supports those of Cave and Walker [7] who found that wood stiffness and bending strength are negatively affected by large microfibril angle. [6] and [17]. reported that the tensile strength of the tracheid decreasing with increasing MFA in the cell wall. This behave of dynamic-mechanical properties of wood with MFA may be attributed to the effect of MFA on the density of wood. Fig. 1 meant 16000 A/m or 0.016 A/m. The density of wood impacts the pulping process, energy consumption and is important for pulp yield [9]. High density had a positive correlation with tear strength but the effects diminished when position in the tree was taken in to account indicating that it is not the density in itself that is important but ruther the properties of the fibres which are mirrored by the density [6]. Farther more has been reported that these two properties have been successfully

ISBN: 978-988-18210-7-2
ISSN: 2078-0958 (Print); ISSN: 2078-0966 (Online)

combined to predict the dynamic-mechanical properties of small specimens [5].

As a result the mechanical properties of *Acacia mangium* wood can be expected to weaker with high MFA. Glass transition in highly crystalline polymers is difficult to identify [21]. This is true because in such cases T_g is a minor event, masked by crystallinity, and because crystalline polymers frequently have multiple transitions arising from relaxations associated with amorphous phase, or both. The effect of MFA on the glass transition was discussed in this study. T_g was calculated based on the temperature at which mechanical properties began to be compromised, that is the maximum damping ratio. An inversely correlation was evident between T_g and MFA in *Acacia mangium* wood. The regression analysis of MFA and T_g data shows that about 71.94% of the variation in T_g was explained by MFA.

ACKNOWLEDGMENT

We greatly appreciate the professional co-operation and assistance of Universiti Malaysia Sabah. We also wish to thank Universiti Putra Malaysia in accessing and using the X-Ray diffraction equipment. Finally, we would like to thank fully Malaysian German Forestry Education Project for the financial support.

REFERENCES

[1] C. Birkinshaw, M. Buggy and C.C. Heen. "Dynamic Mechanical Analysis of Wood," *Journal of Material Science.* vol. 5, Aug. 1986, pp. 89–94.

[2] C. Birkinshaw, M. Buggy and A. Carew "Thermomechanical Behaviour of Wood and Wood Product" *Journal of Material Science"* .vol.3. 1993, pp. 359–362.

[3] I.D. Cave, X-ray measurement of microfibril angle. *For. Prod. J*. Vo. 4. Jul., 1966, pp. 37- 44.

[4] I.D. Cave, "The anisotropic elasticity of the plant cell wall" *Journal of Wood Science and Technology*, vol. 2, Oct. 1968. pp. 268-278.

[5] I.D. Cave, "The Longitudinal Young's Modulus of Pinus radita," *J.Wod Sci. Technol.* vol. 3, June. 1969, pp. 40-48.

[6] I.D. Cave, "Modeling the structure of the softwood cell wall for computation of mechanical properties" *J. Wood science and technology.* vol. 10, Sept. 1976, pp. 19-28.

[7] I.D. Cave and J.F.C. Walker. "Stiffness of Wood In Farown Plantation Softwood: The Influence of Microfibril angle" *Forest Product Journal* .vol. 5, Jul. 1994, pp. 43-51.

[8] I.D. Cave. "Theory of X-Ray Measurement of Microfibril Angle in Wood. Part 1. The Condition for Reflection. X-Ray Diffraction by Materials with Fibre Type Symmetry" *J. Wood Science and Technology.* vol. 3, Feb. 1997, pp. 143–152.

[9] L.A. Donaldson. "Variation in Microfibril Angle among Three Genetic Groups of *Pinus radiate*." *Journal of Forestry Science.* vol. 2, Apr. 1993. pp. 164-175.

[10] R. Evans, M. Hughes and D. Menz. ".Microfibril Angle Variation by Scanning X-Ray Diffractometry" *Appita Journal*. vol. 7, 1999. pp. 261-267.

[11] J.A. Evertsen ".Determination of Wood Quality in *Sitka spruce picea sitchensis* (Bong) carr by Destructive and Non-Destructive method" PhD thesis, University College Dublin. 1988. pp. 67-75.

[12] J.M. Felix and P. Gatenhom. "Effect of Transcrystalline Morphology on Interfacial Adhesion in Cellulose/ Polypropylene Composites". *Journal of Material Science,* vol. 29. pp. 304-313.

[13] P. Gatenholm, H. Bertilsson and A. Matheson. "The Effect of Chemical Composition of Interphase on dispersion of Cellulose Fibres in Polymers. *Journal of Applied Polymer Science.* vol. 21, 1993. pp. 197-208.

[14] H. Hakan, W. Lindsrom Jemes, W. Evans and P. Steven. "Influence of Cambial Age and Growth Conditions on Microfibril Angle in Young Norway Spruce (*Picea abies*)." *Holzforschung*. J. vol. 17, 1998. pp. 173-181.

[15] J.M. Harris and B.A. Meylan. "The Influence of Microfibril Angle on Longitudinal and Tangential Shrinkage in *Pinus radiate*" *Holzforschung J.* vol. 19, Jul. 1965. pp. 144-153.

[16] A. Tamer Tabet, Fauziya Aziz and Shahidan Radiman. " Nano-structural Study of Microfibrils in *Acacia mangium* Wood Using Small-Angle X-Ray Scattering. *Journal of Nuclear and Related Technology*. Vol. 4,March, 207. pp. 209 -216.

[17] T. Mary Treacy., Jos Evertsen and Áine Ní Dhubháin. "A Comparision of Mechanical and Physical Wood Properties of a Range of *Sitka spruce* Provenances. Processing Products No. 1. Extract from Coford final report publication Coford, Dublin, Ireland. 2001.pp. 112-115.

[18] B.A. Meylan. . "Measurement of Microfibril Angle by X-Ray Diffraction" *Forest Prod. J.* vol. 17, 1967, p.p. 15 -58.

[19] J.F. Senft and B. Bendetsen. "Measuring Microfibrillar Angles Using Light Microscopy". *J. Wood and Fibre Science.* vol. 15, Jul. 1985, p.p. 364-367.

[20] S. Andersson. "A Study of the Nanostructure of the Cell Wall of the Tracheids of Conifer Xylem by X-Ray Scattering. *University of Helsinki" Report Series in Physics*. 2006, p.p. 18-25.

[21] J.F.C. Walker and B.G. Butterfield. "*The Importance of the Microfibril Angle for the Processing Industries" New Zealand Forestry J*. vol. 9, Feb. 1996. pp. 34-40.

[22] H. Yamamoto, T. Okuyama and M. Yoshida. "Method of Determining the Mean Microfibril Angle of Wood over a Wide Range by the Improved Cave's Method. *J Mokuzai agkkaishi*. vol. 11. Sept. 1993.pp. 39-45.