A study of fracture surface of fibres in *Acacia Mangium* wood using small-angle x-ray scattering and scanning electron microscope

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Wood is a highly optimized cellular orthotropic material. Its structural properties have major influence on the fracture mechanics behavior of the material. In this study small-angle X-ray scattering (SAXS) and scanning electron microscopy were applied to study the microstructure and fracture surface of fibres in *Acacia Magnum* wood. The aim of this study was to determine the smoothness of the fibre surface as a function to the distance from pith to bark. Smoothness surface and the shape of the fibre were calculated from the scattering graph analysis. The result shows inverse relationship between the fibre smoothness and the distance from pith to bark. Scanning electron microscope (SEM) was used to determine the thickness of the cell wall from pith to bark of wood disc radius 95 mm. The cell wall thicknesses were found to be increase from 2.06 µm in the pith to 7.28 µm at the bark region.

I. INTRODUCTION

The cellular structure of wood is designed to provide optimum conditions for the tree, using a minimum of material [1]. Hardwoods such as *Acacia Mangium* meet these requirements in different ways that result in anatomical differences on the microscopic scale (cell morphology) like the shape and smoothness of the fibre and the surface roughness of the fibre [2]. Hardwoods consist mainly of two kinds of cells, wood fibers and vessels elements [3]. Wood fibers are elongated cells which are similar to tracheids except they are smaller, only 0.7 to 3 mm long and less than 20×10^{-3} mm in diameter. On the nanometer scale, elementary cellulose fibrils with a diameter in the nanometer range embedded in a hemicellulose-lignin matrix are arranged in several layers around the cell [4]. Fig. 1(a) shows a scanning electron micrograph of the general structure of the cell wall in *Acacia Mangium* wood illustrating the smoothness of the fibre cell wall. SAXS have been used widely to examine the detailed structure of wood. The angular region examined in a SAXS experiment is typically from hundredths of a degree to a few degrees 2θ. This small angle region contains information about structures having large d-spacing up to 600 Å [5]. In this paper we use small-angle x-ray scattering, (SAXS) to characterize the fibre cell wall of *Acacia Mangium* wood. In wood there is a sufficient electron density contrast between the cellulose fibrils and the surrounding hemicellulose-lignin matrix [6]. In SAXS we can consider a generalized rule that describes the behavior of scattered intensity as a function of Bragg size, r that is observed at the given scattering angle 2θ, where r is 1/q [7].

$$I(q) = N_p (1/q)\, n_e^2 (1/q) \tag{1}$$

where n_e^2 $(1/q)$, number of electrons in an atom at low angles, q is $4\pi \sin(\theta) / \lambda$, N_p $(1/q)$ is the number of scattering elements in the irradiated volume. Surface fractal law one of the general categories of power laws that are well defined in SAXS [7]. For a smooth surface $S(\mathrm{r}) = \mathrm{r}^2$, and for rough surface $S(\mathrm{r}) = \mathrm{r}^{ds}$, were ds is the surface fractal dimension that varies from 2 to 3 [8]. The scattering law in the SAXS describe two main features, Guinier's region and Porod's region that are observed in a log intensity (I) versus log scattering factor (q). Using Dimensional scattering laws, microfibrls can be described in terms of their dimension in the sense that a rod is 1-D, a disk 2-D and a sphere 3-D. Such a dimensional description implies that the mass of the object depends on the size of observation (r). Consider that scattering from a rod is observed at r = 1/q between the rod length, L, and the rod diameter, D. Then the number of scattering elements in the rod of size

$$(\mathrm{r}) = L/\mathrm{r} \text{ or } Lq. \tag{2}$$

The number of electron per scattering element is given by rD^2 that means:

$$ne^2 = \mathrm{D}^2 / q. \tag{3}$$

Using Eq. (1) we have:

$$\mathrm{I}(q) = L\mathrm{D}^4 / q. \tag{4}$$

That means the scattering is proportional to q^{-1} decay in intensity for the 1D object.

II. MATERIALS AND METHODS

Wood samples were taken from a 10-year old of *Acacia Mangium* tree grown in Sabah Forestry Development Authority Plantation (SAFODA), Sabah, Malaysia. Thin samples of thickness 200 μm×10,000 μm of *Acacia Mangium* were prepared by using sliding microtome from pith to bark of the wood section 95.0 mm ± 0.1 mm of wood disc as shown in Fig. 1(b). They were encapsulated in plastic foils in order to prevent drying and shrinkage of the wood cells in the vacuum chamber. HMBG–SWAX, SAXS of type PW 3830 X-Ray used to determine fibre shape and smoothness from pith to bark of the wood section. The samples were exposed to Cu Kα radiation of wave length λ = 0.154 nm. SAXS patterns were obtained by positional detector PSD. Data was recorded as a function of the scattering vector q with modulus $q = |q| = 4\pi / \lambda \sin(\theta)$ where 2θ is the angle between the incoming and the scattered x-ray beam. The experimental set-up consisted of 12 KV and 20 mA. The incoming x-ray beam had a circular cross-section of 0.6 mm in diameter. SAXS data for 9 samples were given from Guinier's and Porod's regions described in scattering graph. The same samples were used to determine the thickness of the cell wall as a function of the distance form pith to bark using scanning electron microscope.

III. RESULTS

Fig. 2 shows typical SAX-Scattering graph obtained from *Acacia Mangium* wood samples at distance 90 mm from the pith of the wood section. The scattering law in the SAXS describe two main features, Guinier's region and Porod's region that are observed in a log intensity I versus log scattering factor q plot (Fig. 3), where q is the wave vector of the scattered beam ($Å^{-1}$) [7]. The shape of scattering graph at Porod's region, is use to obtain information on the smoothness of the fibre surface [9]. Here the fibers are smooth at distance 10 mm from the pith to 40 mm. This is shown in the slope as declare statement from −4.97 to −3.62 as shown in Table I. Fractal surface analysis in Porod's region showed that fibre surface was rough at the bark region from 50 mm to 90 mm from the pith. This is shown in the Porod's slope from −2.48 to −0.32.

Surface structure analysis from Guinier's region of scattering graph using Guinier analysis [7] showed a rod shape of fibre in *Acacia Mangium* samples as shown in Fig. 4. This is shown in the slope value from −4.97 to −3.62 (Table I). The thickness of the cell wall, *T* for the same samples was measured using scanning electron microscope (SEM) (Fig. 5). The results showed that *T* clearly increased with increasing the distance from pith to bark with slightly deflection at distance from 60 mm to 70 mm as shown in Fig. 6. *T* was found to range from 2.23 μm at 20 mm from the pith to 7.28 at 90 mm from the pith region.

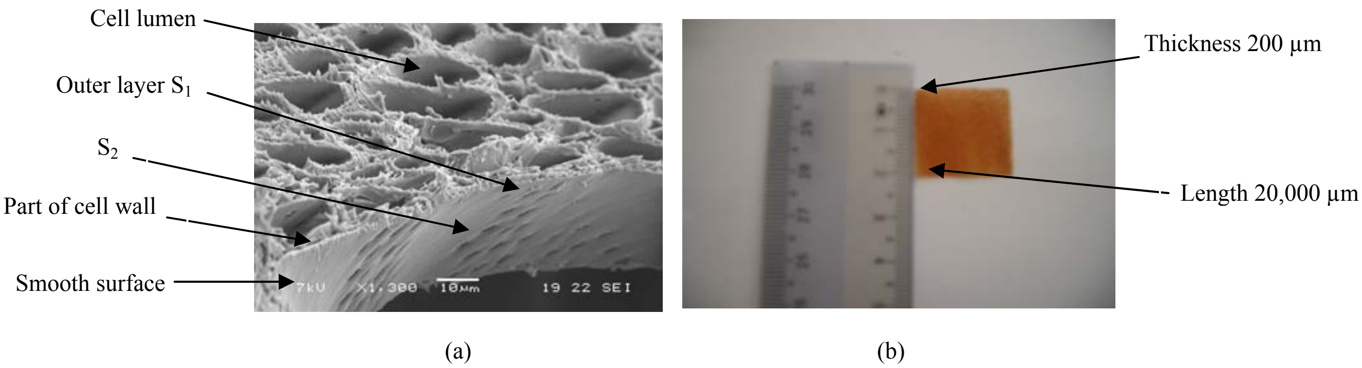


FIG. 1. (a) Scanning electron micrograph of the general structure of the cell wall in *Acacia Mangium* wood illustrating the smoothness of the fibre cell wall. (b) Thin sample 200.0 μm thick × 10,000.0 μm long.

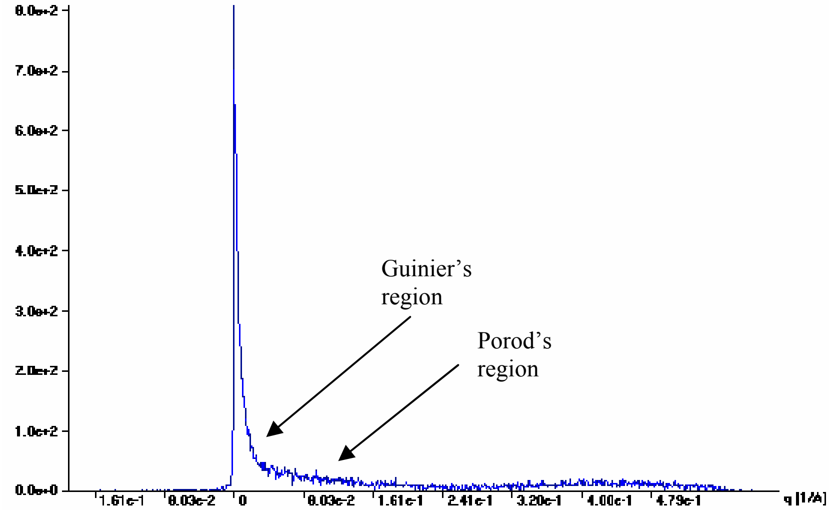
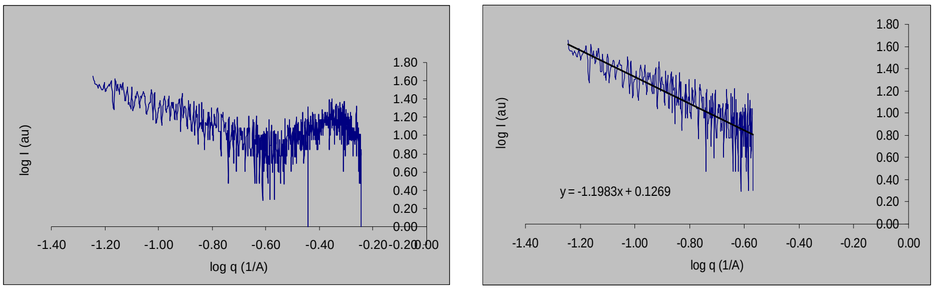
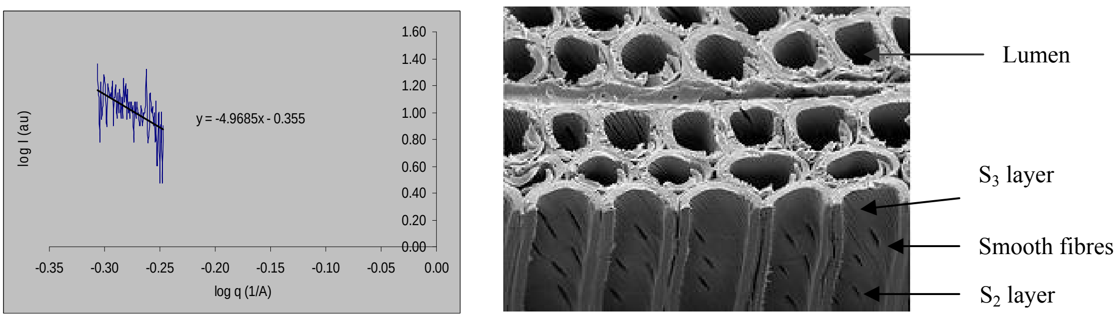


FIG. 2. Small-angle X-ray scattering graph obtained from *Acacia Mangium* wood at distance 40 mm from the pith.



(a) (b)

FIG. 3. (a) Guinier's region and porod's region of scattering graph with arbitrary unit. (b) Guinier analysis of *Acacia Mangium* sample of thickness of 200 μm.

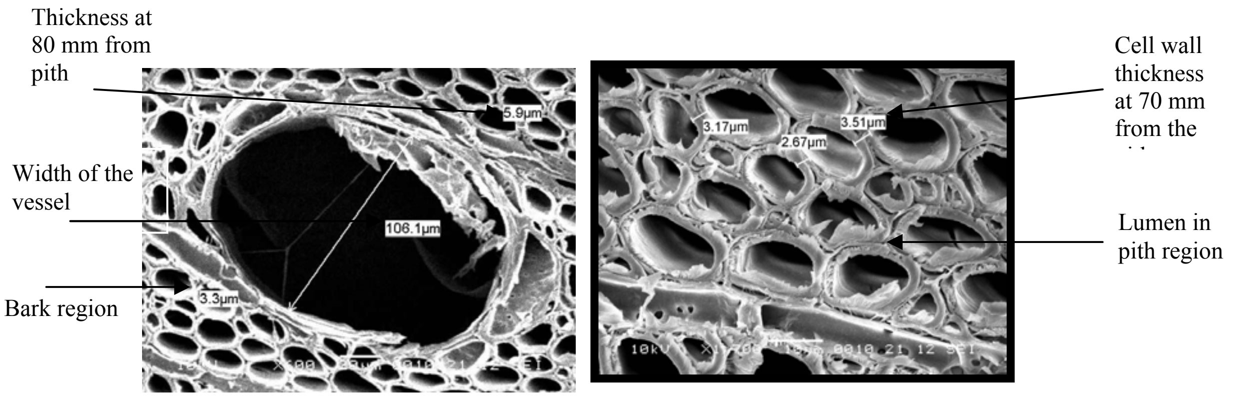


(a) (b)

FIG. 4. (a) Porod analysis of *Acacia Mangiun* Sample of thickness 200 μm. (b) Scanning electron micrograph of *Acacia Mangium* wood illustrating the shape of the fibre in the cell wall.

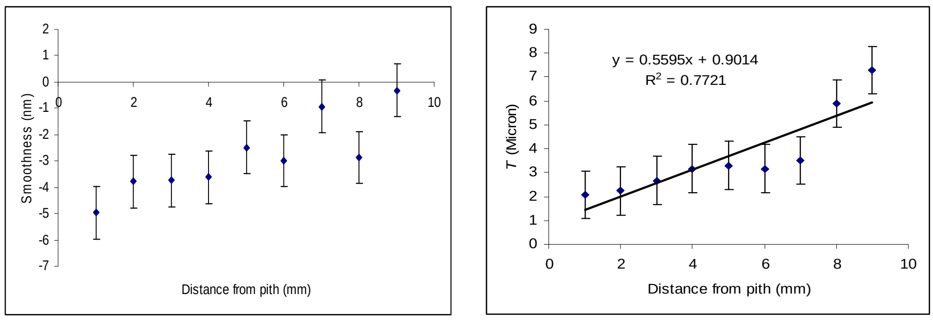
TABLE I. The results of SAXS analysis for *Acacia Mangium* wood.

Distance from pith (mm)	Sample region	Porod's slope	Guinier's slope	Cell wall thickness, T (μm)
10	Pith	-4.97	-1.15	2.06
20	Pith	-3.78	-1.20	2.23
30	Pith	-3.74	-1.05	2.67
40	Bark	-3.62	-0.96	3.17
50	Bark	-2.48	-0.94	3.30
60	Bark	-2.99	-0.99	3.17
70	Bark	-0.94	-0.78	3.51
8 0	Bark	-2.87	-0.82	5.9
90	Bark	-0.32	-0.91	7.28
		Std Dev=1.4555	Std Dev=0.39084	Std Dev= 1.74
		Mean= - 2.856	Mean = - 0.977	Mean= 3.69
				Maxi. =7.28
				Min. = 2.06



(a) (b)

FIG. 5. SEM micrograph shows the thickness of cell wall in *Acacia Mangium* wood. (a) The bark region at 60 mm from pith. (b) At distance 20 mm from the pith.



(a) (b)

FIG. 6. (a) The error bars of the graph. (b) The relationship between thickness of the cell wall and the distance from pith to bark in *Acacia Mangium* wood.

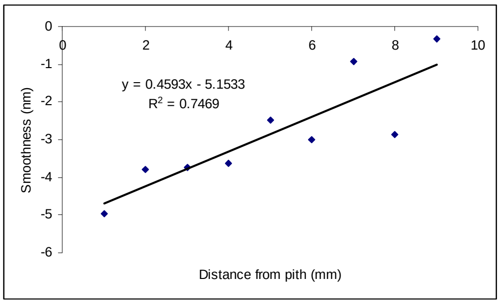


FIG. 7. The relationship between fibre smoothness and the distance from pith to bark in *Acacia Mangium* wood.

IV. DISCUSSION AND CONCLUSION

In this study small-angle x-ray scattering and scanning electron microscope techniques were used to determine the fracture surface of thin section in *Acacia Mangium* wood. The surface structure of the fibres in cell wall and the shape of the fibre were carried out based on the surface fractal law of scattering graph [7], and yielded two interesting results: (1) SAXS analysis showed the smoothness of the fibre varied from pith to bark of the wood trunk.. Surface is smooth at pith region from 10 mm to 40 mm, this is shown in the slope value from −4.97 to −3.62. Smoothness was found to decrease near the bark at the value of slope. This is shown in the slope as declare statement from −2.48 to −0.32 as shown in Fig. 7. (2) Guinier analysis of the scattering graphs showed that a rod shape of fibre in the pith region from distance 10 mm to 60 mm in the bark region. This results give a reasonable explanation for the inversely relationship between fibre length and the distance from pith to bark that has been recently obtained. As the fibre length increase in the pith region, the fibre will take a rod shape than the shape will be irregular rod shape in the bark region. Cell wall thickness was found to increase fro pith towards the bark s shown in Fig. 6. The highest increase was found from 5.9 to 7.28 μm at the end of the bark from 80 to 90 mm from the pith. The reason has been recently suggested by Fratzl [10], is that the latewood is denser than the earlywood because the cell walls and fibres are thicker.

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