

## STUDY ON LIGNIN COVERAGE OF MASSON PINE FIBER

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In order to obtain the adhesion force of fiber in a paper sheet easily, the relationships between internal bonding strength (IBS) and surface lignin content of masson pine CTMP treated with peracetic acid (PAA) have been investigated with XPS technique, and the surface morphology of fibers was also imaged by AFM. The results showed that the extent of lignin covered on the fiber surface was two times as high as that of whole pulp lignin, and the IBS was inversely proportional to surface lignin. The relationship between IBS and lignin coverage was formulated based on the experimental data. The mutual adhesion forces, cellulose-to-cellulose and lignin-to-lignin, were calculated using these equations, and the results were 28.69 mN/m and 2.487mN/m, respectively.

*Keywords:* Surface lignin; Adhesion force; IBS; XPS; AFM

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## INTRODUCTION

Wood is composed mainly of cellulose, hemicelluloses, and lignin. Pulping is a process in which the wood fibers are separated from each other chemically or mechanically. Chemical pulping is a process in which wood chips are cooked with chemicals and the lignin is almost completely removed. Chemical pulp is suitable for the production of fine paper. By contrast, chemithermomechanical pulp (CTMP) is produced by a relatively mild chemical treatment followed by pressurized refining and is extensively used for the production of fluffy pulp, printing paper, hygiene products, and newsprint.

Compared with chemical pulp, CTMP has the potential to produce much less pollution, achieve a higher yield, and have better properties, such as bulk, opacity, and printability. But the internal bonding strength (IBS) of CTMP fiber is not as good as that of chemical pulp, and there is more hydrophobic lignin covering the fiber surface. It has been reported that lignin, hemicellulose, and extractives are present on the CTMP fiber surface, but it is still unclear how lignin affects the strength of a CTMP sheet (Yu et al. 2002).

The fiber surface morphology and chemical composition are different under different treatment conditions. It is very important to study the pulp in terms of surface composition, surface morphology, and chemical components distribution, because these properties affect fibers' wettability, flexibility, bonding ability, electrostatic, interactions between fibers and additives, and pulp defibration. Studies up until now of fiber surfaces have focused on chemical compositions and surface morphology by the aid of

various techniques including AFM, XPS, CLSM, tof-SIMS, and DCA (Fardim and Duran 2002; Boras and Gatenholm 1999; Koljonen et al. 2004)

Fiber and microfibrils can be imaged by atomic force microscopy (AFM) in water or in air, though the quality and ultimate resolution of AFM images may not be as good as that of electron microscopy. AFM has been extensively used to study wood, fiber, and paper because of its convenience in sample preparation and use under ambient environment (Hanley and Gary 1999). Dorris and Gray (1978) determined the chemical composition of fiber surfaces by an X-ray photoelectron spectroscopy (XPS) technique for the first time, and they estimated surface lignin on fiber surface with O/C atomic ratio.

In order to investigate the impact of lignin coverage on sheet strength, different lignin coverages on a fiber surface should be available, which could be generated by treating CTMP with cooking agents, such as NaOH, NaS, and Na<sub>2</sub>SO<sub>3</sub>, etc. Yet, because these chemicals injure cellulose and hemicellulose during delignification, PAA was chosen as a cooking agent to treat CTMP. PAA has been recognized as a powerful oxidant that oxidizes aromatics in lignin and generates dicarboxylic acids and their lactones (Teixeira et al 2000). Compared with NaOH, PAA can remove lignin effectively and has been reported as a selective agent. Most of the hemicelluloses and cellulose remained during delignification, although the microfibrils were exposed, and the fibers were slightly damaged (Zhang et al, 2008).

During delignification, different layers of cell wall can be exposed. The wood cell wall is composed of a middle lamella (ML), primary wall (P), and secondary wall layers S<sub>1</sub>, S<sub>2</sub>, and S<sub>3</sub>. Identification of different layers can be based on microfibril orientation and the amount of fibrillar and unfibrillar region (Yang 2006). The angle of microfibril orientation in the S<sub>1</sub> sublayer of masson pine fiber is about 35°, S<sub>2</sub> is in the range 25~30°, and S<sub>3</sub> has an angle of 60~80° (Chen et al. 1981).

Different lignin coverages on the fiber surface have different impact on sheet strengths. One of the important parameters affecting sheet strength is the specific bonding strength (*SBS J/m<sup>2</sup>*), which is determined by the Scott Bond method. The overall evaluation was obtained in terms of internal bonding strength (*IBS J/m<sup>2</sup>*) and the relative bonded area (*RBA %*). The relationship among *SBS*, *IBS*, and *RBA* is shown in equation (1) (Shao and Li 2006). *RBA* is determined by *S<sub>0</sub>* and *S* as shown in equation (2), where *S* is the light scattering coefficient of the sheet, and *S<sub>0</sub>* is determined from the extrapolation of the plot of light scattering coefficient versus tensile index.

$$SBS = IBS / RBA \quad (1)$$

$$RBA = (S_0 - S) / S_0 \quad (2)$$

*IBS* is mainly ascribed to hydrogen bonds and Van Der Waals forces (Van den Akker 1959). It is an important parameter of a sheet and can be investigated by measuring the adhesion force between fiber materials with AFM. It has been found that the adhesion force between hydroxypropyl cellulose (HPC) and BKP fibres is much greater than that between HPC and CTMP fibres, and the adhesion force between HPC and HPC is about four times as large as the adhesion forces between lignin and lignin (Tan and Li 2008); additionally, the adhesion forces between two Langmuir-Blodgett

cellulose films measured with a surface force apparatus by Holmberg et al. (1997) are in a range of 500 to 1000 mN/m. But the relationships between surface properties of fiber, such as composition, morphology, and chemical components distribution with adhesion forces of fiber have not been determined effectively.

In this research, masson pine CTMP was treated with different dosage of PPA, and four different pulp samples were prepared. The fractional lignin coverage of these samples was investigated with the XPS technique in order to evaluate the impacts of lignin coverage to sheet strength. Meanwhile, images of each sample were captured by AFM to investigate the influence of surface lignin on sheet strength based on nanometer-scale observations. In order to make it simple to obtain the adhesion force of cellulose-cellulose and that of lignin-lignin, the relationship between IBS and lignin coverage was investigated.

## **EXPERIMENTAL**

### **Materials**

Unwashed masson pine (*Pinus massoniana*) CTMP was obtained from the Guangzhou Paper Mill of the People's Republic of China. Its moisture was 70%. The PAA that was used was of analytical grade with 10% concentration.

### **Procedure**

Masson pine CTMP was washed before the treatment in order to get rid of the soluble substances, improve the purity of the pulp, and reduce the chemicals consumption.

Four samples of masson pine CTMP were prepared using PAA at 80°C for 1.5h; the dosage of PAA was 10%, 20%, 30%, and 40% of pulp mass, respectively; the wood:liquid ratio was 1:8.

Klason lignin content was determined by TAPPI test method T222-om-987 (TAPPI 1998) and TAPPI useful method UM 250 was used for acid-soluble lignin (TAPPI 1991).

Handsheets were prepared with a basis weight of 60g/m<sup>2</sup>, pressed, and dried according to the corresponding ISO method, and the handsheets were conditioned at 50% RH and 23°C for at least 48h prior to strength testing.

### **Paper Physical Properties**

Tensile index, IBS, and light scattering coefficient were measured according to the standard ISO method. IBS was tested according to the Scott Bond method.

### **AFM Analysis**

Suspensions of pulp fibers with 0.1% consistency were prepared from the masson pine CTMP treated with PAA. A drop of pulp suspension was placed onto a clean, dry glass cover slip, dried on a hot plate at 70°C, and then the cover slip was glued onto a steel disc used for mounting samples onto the scanner. A Nanoscope III multimode AFM was used to generate the surface image. The image was scanned in contact mode in air

using commercial Si<sub>3</sub>N<sub>4</sub> cantilevers with drive amplification of approximate 3690mV, drive frequency of 370 kHz, amplitude setpoint of 1.4v, and scan rate of 1Hz. Each sample scanned at least 10 different fibers, and each fiber was investigated at the size ranges of both 2.5×2.5μm and 5.0×5.0μm; only representatives of the images were displayed.

### XPS Analysis

XPS measurements were performed with a Thermo ESCALAB 250-Multitechnique Surface Analysis using a monochromatic Al/K X-ray source ( $h\nu = 1486.6$  eV). The used power was 150W, the diameter of X-ray measurement area was 500×500μm, the pass energy of the energy analyzer was 20 eV, and the detective depth of XPS ranged from 5 to 7 nm. The samples were outgassed overnight under a pressure of  $1\times 10^{-9}$  Torr, and each sample was measured at three different spots.

Using a curve-fitting programme, Gaussian curves were fitted for deconvolution of the carbon emission lines, and the curves were assumed to follow the shape of a Gaussian or Lorenzian relationship with a ratio (G/L) of 0.70. The chemical shifts relative to the C-C component (bonding energy 284.62 eV) used in the deconvolution were  $1.75 \pm 0.04$  eV for C-O(C<sub>2</sub>),  $3.45 \pm 0.25$  eV for O-C-O or C=O (C<sub>3</sub>), and the sensitivity factors for carbon and oxygen were 0.29 and 0.71, respectively. The constant full-width-half-maximum (FWHM) values were used in the curve fitting of each spectrum.

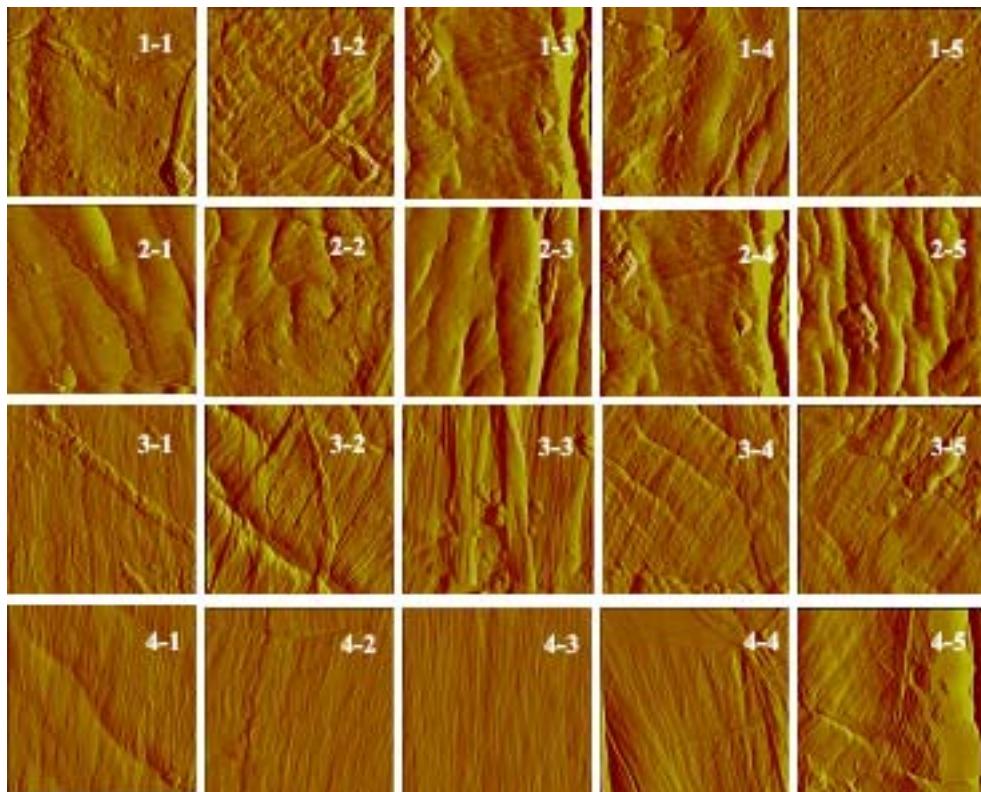
## RESULTS AND DISCUSSION

### Morphology of Fiber Surface

The fiber surface could be imaged by AFM, which made it possible to investigate fiber surface morphology. Figure 1 is representative of the masson pine fiber surface. The fiber surface is rough, and valleys and ridges are apparent in the images for 10% and 20% PAA dosage. With PAA dosage increased to 30% and 40%, more and more microfibrils can be seen on the images, with the angles between microfibril and fiber axis being different.

Figures 2 (5.0μm×5.0μm) and 3 (2.5μm×2.5μm) are AFM images chosen from Fig. 1. From Figs. 2 and 3 it can be seen that the fiber surface was rough and different layers of cell wall were exposed. Figures 2(a) and 3(a) can be interpreted as middle lamella. Figures 2(b) and 3(b) are the AFM images of CTMP treated with 20% PAA. The rough area is interpreted as middle lamella, which accounted for 50% of the area. The remainder was primary wall covered with a thin layer lignin, indicating that part of the middle lamella was removed when CTMP was treated with 20% PAA. Figures 2(c) and 3(c) are the images of CTMP treated with 30% PAA, and they show that there were two different groups of microfibrils. The angles between fiber main axis and the microfibrils on one group was about 35° (S<sub>1</sub> feature) and another was 25°~30° (S<sub>2</sub> feature). These microfibrils paralleled each other, which indicates that the middle lamella and primary wall were removed and S<sub>1</sub> and S<sub>2</sub> were exposed when CTMP was treated with 30% PAA. Figures 2(d) and 3(d) are the images of CTMP treated with 40% PAA. It can be seen that the fiber surface was very smooth, the microfibrils were parallel to each other, and the

angle between microfibrils and fiber axis was approximately 25°. It can be deduced that the S<sub>2</sub> layer was exposed when 40% PAA was used. In addition, from Figs. 2(c) and 3(c) it was found that there were some uneven granules with sizes of around 220nm to 463nm on the fiber surface. A few granules were represented in Figs. 2(d) and 3(d) also, but their diameters were smaller. These granules can be interpreted as being comprised of lignin, hemicellulose, and extractives, or alternatively as being a complex of lignin and hemicellulose (Gustafsson et al. 2003) that precipitated on the fiber surface from the liquid phase during the treatment (Koljonen et al. 2005).

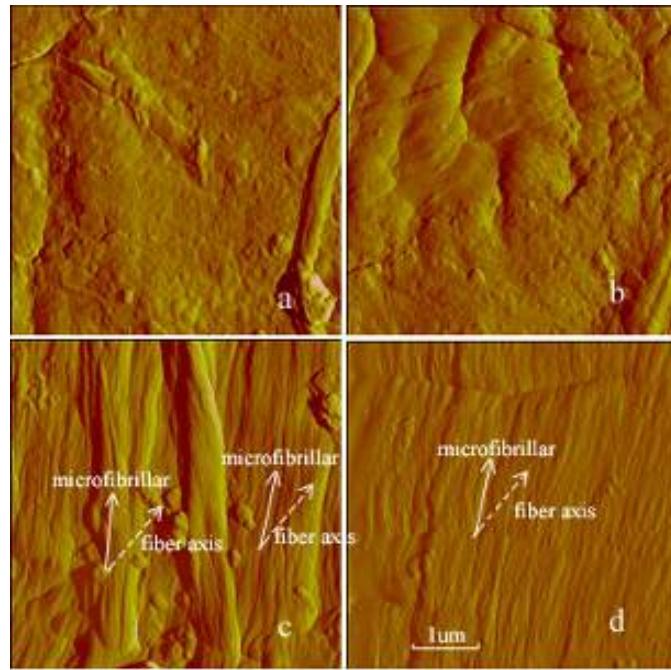


**Fig.1.** AFM amplitude images of masson pine CTMP ( $5\mu\text{m} \times 5\mu\text{m}$ ) treated with different dosage of PAA. (1-1~1-5) 10% PAA, (2-1~2-5) 20% PAA, (3-1~3-5) 30% PAA, (4-1~4-5) 40% PAA.

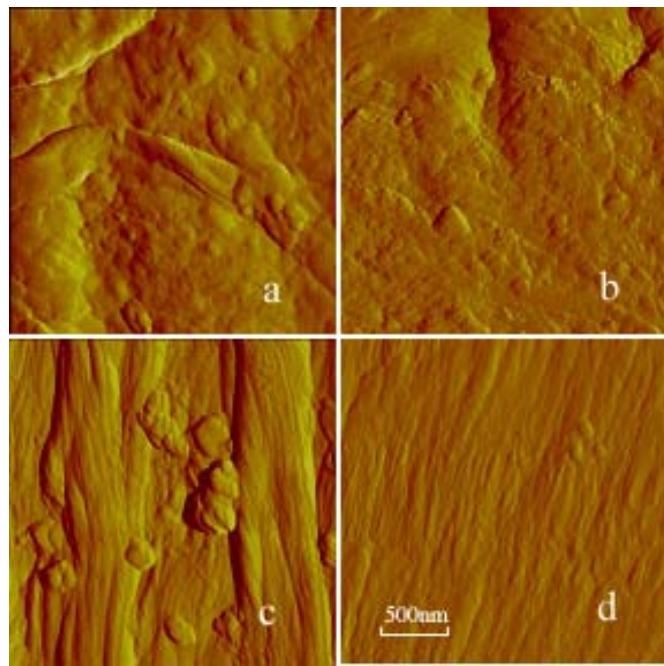
### XPS Analysis

An XPS spectrum of masson pine CTMP treated with 20% PAA is shown in Fig. 4. It can be seen from the spectrum that two kinds of elements, oxygen and carbon, were present on the fiber surface (peaks located at 532.97eV and 285eV respectively). In addition, a low amount of sodium retained on the fiber surface was also revealed. Thus, it was conjectured that the sodium originated from Na<sub>2</sub>SO<sub>3</sub> used for pre-treatment of masson pine chips prior to the grinding of the chips into pulp.

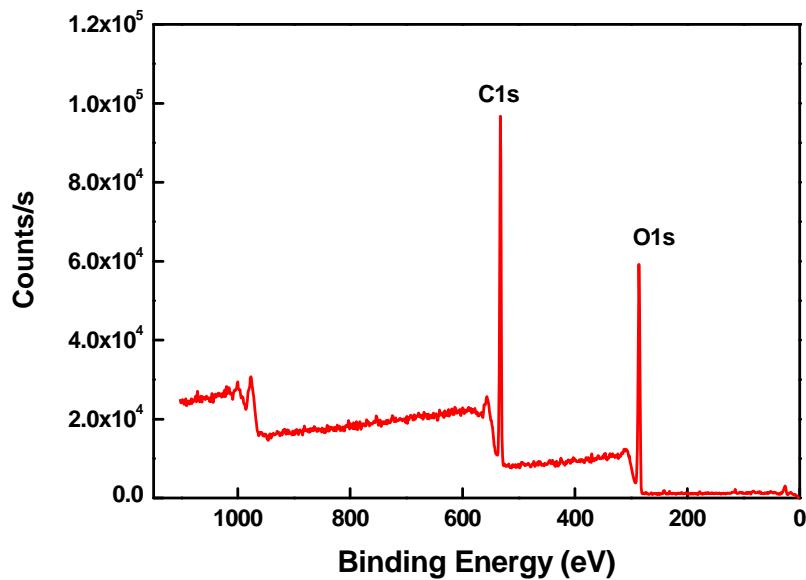
A C<sub>1s</sub> Gaussian curve was fitted for the deconvolution with a curve-fitting program, and the results are shown in Fig. 5. The C<sub>1s</sub> peak was divided into contributing peaks for C<sub>1</sub>, C<sub>2</sub> and C<sub>3</sub>. The binding energy of C<sub>1</sub> (C-C and C-H) was 284.62eV; the chemical shifts relative to C<sub>1</sub> were 1.77eV for C<sub>2</sub> (C-OH) and 3.89eV for C<sub>3</sub> (C = O).



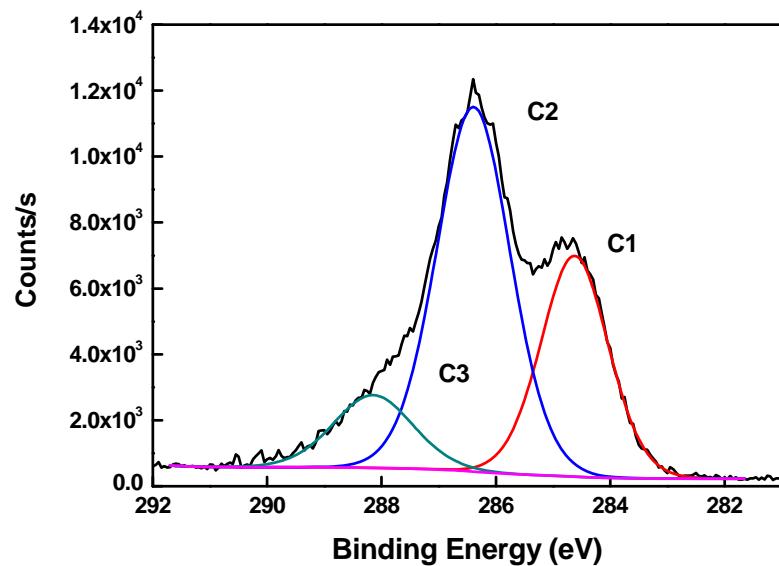
**Fig. 2.** AFM amplitude images of masson pine CTMP ( $5\mu\text{m} \times 5\mu\text{m}$ ) treated with different dosage of PAA. (a) 10% PAA (b) 20% PAA (c) 30% PAA (d) 40% PAA



**Fig. 3.** AFM amplitude images of masson pine CTMP ( $2.5\mu\text{m} \times 2.5\mu\text{m}$ ) treated with different dosage of PAA (a) 10% PAA (b) 20% PAA (c) 30% PAA (d) 40% PAA



**Fig. 4.** XPS spectrum of masson pine CTMP treated with 20 % PAA



**Fig. 5.** C1s spectrum of masson pine CTMP treated with 20% PAA

The data regarding the deconvolution are listed in Table 1. The areas of C<sub>1</sub>, C<sub>2</sub>, C<sub>3</sub>, and O<sub>1s</sub> contributions were 19.7, 35.61, 7.66, and 37.04, respectively. The C<sub>1s</sub> area was 62.94, which is equal to the sum of C<sub>1</sub>, C<sub>2</sub>, and C<sub>3</sub> areas.

**Table 1.** XPS Data of Masson Pine CTMP Treated with 20% PAA

C	Lowest binding energy	peak	highest binding energy	CPS	FWHM eV	area (P) CPS.eV	area(N) KE <sup>1.0</sup>	area. %
C <sub>1s</sub> (-CH)	291.7	284.62	281.65	6695.41	1.44	10064.16	0.0544	19.7
C <sub>1s</sub> (-OH)	291.7	286.39	281.65	11061.53	1.58	18176.49	0.0983	35.61
C <sub>1s</sub> (=O)	291.7	288.15	281.65	2204.53	1.7	3905.05	0.0211	7.66
O <sub>1s</sub>	536.6	532.97	529.5	21280.43	1.85	44210.14	0.1023	37.04

Surface lignin coverage could be estimated by use of Equation (3). The (O/C)<sub>sample</sub> ratio was determined from the ratio of the O and C<sub>1s</sub> areas in the plot. The theoretical value of (O/C)<sub>lignin</sub> is 0.33 and that of (O/C)<sub>cellulose</sub> 0.82 (Dorris and Gary 1978). In contrast to Equation (3), surface lignin coverage can also be calculated according to Equation (4). C<sub>1</sub> is the relative amount of C<sub>1</sub> in the C<sub>1s</sub> spectrum, and  $\alpha$  refers to the contribution to the C<sub>1</sub> peak from surface contamination. The value used for  $\alpha$  is 2. Table 2 summarizes the estimated surface lignin coverage calculated from Equations (3) and (4):

$$\text{lignin\%} = \frac{(O/C)_{\text{sample}} - (O/C)_{\text{cellulose}}}{(O/C)_{\text{lignin}} - (O/C)_{\text{cellulose}}} \quad (3)$$

$$\text{lignin\%} = 100 \times (C_1 - \alpha) / 49 \quad (4)$$

**Table 2.** Lignin Coverage on CTMP Fiber Surface Estimated by Equation (3) and Equation (4)

PAA %	O %	C <sub>1s</sub> %	O/C	lignin*(%)	C <sub>1</sub> (%)	lignin#(%)	klason lignin and acid soluble lignin
10	34	66	0.515	63.0	39.3	76.1	28.9
20	36.6	63.4	0.577	50.5	33.9	65.1	25.8
30	37.6	62.4	0.603	45.5	30.3	57.8	23.1
40	38.2	61.8	0.618	42.4	29.8	56.7	19.4

\*consequence by equation (3)   #consequences by equation (4)

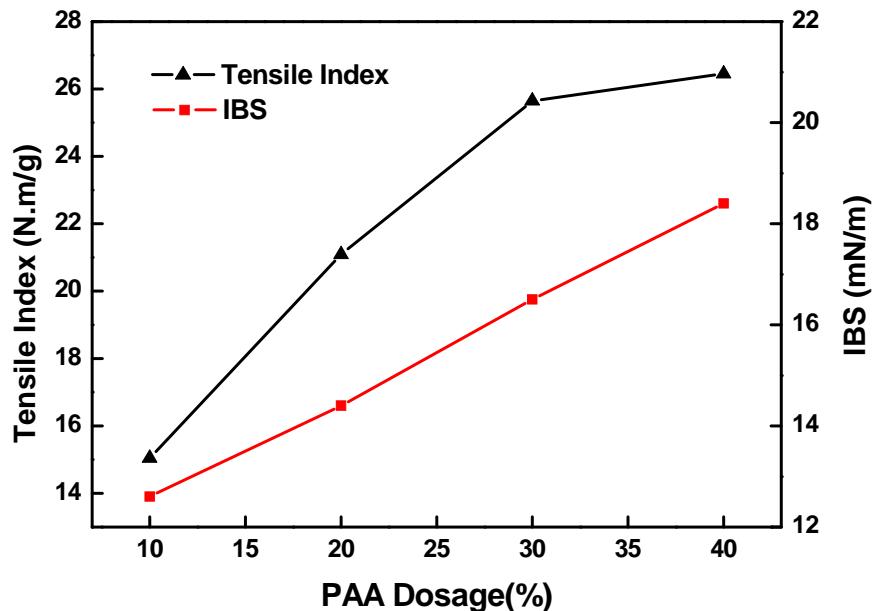
Table 2 indicates that surface lignin decreased from 63% to 42.4% with the dosage of PAA increasing from 10% to 40%. This conclusion is in agreement with the morphology, as determined by AFM. It is well known that lignin is enriched in the middle lamella. The concentration of lignin in different layers falls in the order of ML>P>S<sub>1</sub>/S<sub>2</sub>. As described previously, with the increase of PAA dosage, ML and P layers were removed, and S<sub>1</sub> and S<sub>2</sub> sublayers were exposed, so the amount of surface lignin was reduced. It can also be seen from Table 2 that surface lignin estimated with Equation (4) was higher than that estimated with Equation (3). This is attributed to the possibility of masson pine CTMP absorbing moisture during storage, which would have resulted in a slight increase in oxygen content and a lower amount of lignin when calculated with Equation (3). In addition, compared with the amount of lignin in the

whole pulp, the amount of surface lignin would be approximately 2 times as high as that of lignin in the bulk.

### Paper Physical Properties

In order to evaluate the impact of surface lignin on bonding strength, physical properties including tensile index, internal bonding strength (Scott Bond method) as well as light scattering coefficient were measured.

Sheet strength results are provided in Table 3. The plot (Fig. 6) of tensile index and *IBS* versus PAA dosage indicated that the tensile index and *IBS* increased with increasing PAA dosage. As described earlier, due to the delignification effect of PAA, the increase of PAA dosage resulted in the removal of ML and P and the exposure of S<sub>1</sub> and S<sub>2</sub>, meaning that surface lignin was reduced and more cellulose was exposed. The number of hydrogen bonds generated in the interfacial bonding areas of the sheet were greatly increased, and conversely, less lignin interfered with the internal bonding; therefore, tensile index and *IBS* increased.



**Fig. 6.** plot of tensile index and IBS versus PAA dosage

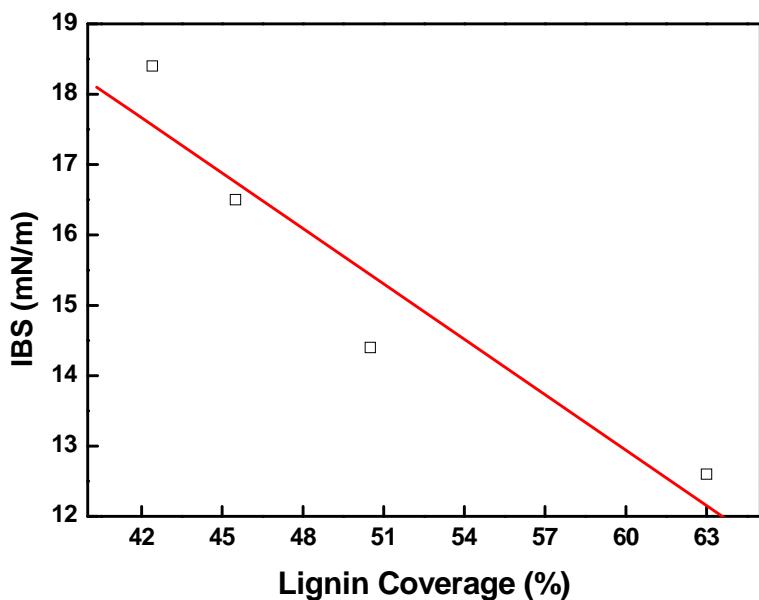
It can be seen in Table 3 that the difference between light scattering coefficients of the samples was slight, so the light scattering coefficients could be considered to be constant, which means that the internal bonded area in all samples was the same and surface lignin coverage had no significant effect on the bonding area. Equation (1) shows that *SBS* increased with the decrease of surface lignin, because *IBS* increased and the *RBA* remained constant.

**Table 3.** The Relationship between Paper Strength and PAA dosage

PAA (%)	10	20	30	40
Lignin coverage (%)	63.0	50.5	45.5	42.4
Tensile strength index(N·m/g)	15.04	21.09	25.65	26.45
IBS( $J/m^2 \times 10^{-3}$ )	12.6	14.4	16.5	18.4
Light scattering coefficient( $m^2/kg$ )	2.69	2.65	2.71	2.68

### Adhesion Force of Cellulose-cellulose and Lignin-lignin

As mentioned above, *SBS* is specific bonding strength. If the fiber surface were to be completely covered by lignin, then *SBS* would be the adhesion force of lignin-lignin, but if the fiber surface were to be completely covered by cellulose, then *SBS* would be the adhesion force of cellulose-cellulose. Here *SBS* was substituted by *IBS* because *RBA* is difficult to calculate for mechanical pulp. Figure 7 is a plot of *IBS* versus surface lignin coverage.

**Fig. 7.** plot of IBS versus surface lignin on fiber surface

Linear regression was performed with Origin 7.5 software, and the regression formula is given by Equation (4), which shows that  $y$  is equal to 28.687 if  $x = 0$ , and  $y$  is 2.487 if  $x = 100$ . This means that if there is no lignin on the fiber surface or if the fiber surface were almost completely covered by cellulose, then the adhesion force between fibers would be approximately 28.687 mN/m. If the fiber surface were totally covered by lignin, then the adhesion force between fibers would be 2.487 mN/m; that is to say that the adhesion force due to the cellulose-cellulose interaction was 28.687 mN/m and that of the lignin-lignin interaction was 2.487 mN/m. The adhesion force for cellulose-to-cellulose was 11 times greater than that of lignin-to-lignin.

$$y = 28.687 - 0.262x \quad (4)$$

Compared with the reported value of 500 to 1000 mN/m of adhesion forces between two Langmuir-Blodgett cellulose films, measured with a surface force apparatus by Holmberg et al. (1997), 28.687mN/m of adhesion force is much smaller. This difference may be explained as follows: First, *SBS* rather than *IBS* ought to be used to calculate the adhesion force, because the light scattering coefficient method determining *RBA* has been shown to be inaccurate for mechanical pulp (He et al. 2007). Because it is difficult to determine the *RBA* of the sheet, the results would be larger if *SBS* was used for the determination of adhesion force. Secondly, the components of fiber surface include cellulose, hemicellulose, lignin, and extractives. Provided that PAA is able to remove lignin completely, in addition to the cellulose retained on the fiber surface, hemicellulose and extractives would be present on the fiber surface also. Therefore, the result should be attributed to the net adhesion force of cellulose, hemicelluloses, and a small contribution due to extractives, rather than just that of cellulose. Thirdly, PAA treatment changed the single fiber properties a little, which had an impact on *IBS*. Even though the result was much lower than the theoretical value, the value of this work is that a way was found to investigate the adhesion force of carbohydrate and that of lignin.

## CONCLUSIONS

1. Two surface testing techniques, XPS and AFM, were found to compliment each other very well. XPS gives quantitative information on the composition of fiber surface, while AFM provides images of the fiber surface and helps to explain the coverage of surface components.
2. Lignin content on untreated fiber surfaces was two times as high as that of lignin in the bulk; surface lignin had a significant impact on sheet strength properties such as specific bonding strength and tensile index and had some impact on bonding area
3. The relationship between *IBS* and surface lignin coverage was investigated. It was found that *IBS* was inversely proportional to surface lignin coverage, and an equation describing the relationship between *IBS* and surface lignin coverage was suggested. Adhesion forces of cellulose-to-cellulose and of lignin-to-lignin calculated using this equation were 28.687 mN/m and 2.487mN/m, respectively. The adhesion force of cellulose-to-cellulose was 11 times greater than that of lignin-to-lignin.

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