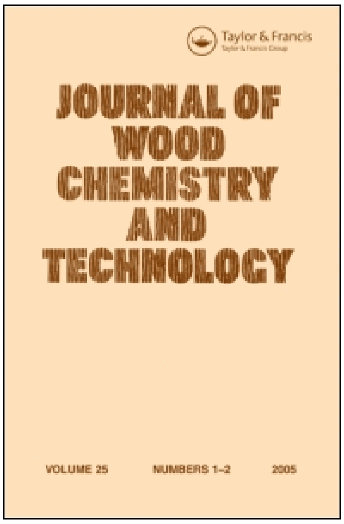

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Abstract: It has been speculated in several studies that lignin would impair interfiber bonding when present on the fiber surface due to the hydrophobic nature of lignin. Several experiments were devised in the present study to compare the internal bond strength and specific bond strength of pulp fibers with different surface lignin concentrations. The pulp samples used included unbleached and fully bleached kraft pulp, CTMP pulp with different middle lamella coverage on the fibers, and fully bleached pulp with lignin adsorbed on the fiber surface. With all of the pulp samples used, the internal bond strength was reduced markedly when lignin was present on the fiber surface; but the relative bonded area was not affected significantly.

Keywords: Interfiber bonding, specific bond strength, lignin, surface lignin, CTMP, middle lamella, surface peeling

INTRODUCTION

Interfiber bonding strength is one of the principal factors controlling the physical strength of article. This is shown in a quantitative way by Page's

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equation.[1] The paper strength is not only important for applications such as printing and coating but also for runability on the paper machine. Poor interfiber bonding strength can cause delamination and splitting of paper in the printing and coating processes. In particular, paper with low surface strength may result in linting and fiber rising in printing.

Interfiber bonding strength is conventionally considered to be mainly from the intermolecular hydrogen bonds as well as Van de Waals forces between adjacent fiber surfaces.[2,3] Therefore, the chemical composition including cellulose, hemicellulose, and lignin of the fiber surface may play an important role in interfiber bonding. It has been reported in several studies that after kraft pulping, residual lignin is highly enriched on the fiber surfaces.[4–9] Li and Reeve[10,11] found that surface lignin is mainly from the adsorption of dissolved lignin in the pulping liquor. It has been speculated in the literature that the high concentration of lignin on the fiber surface may impair interfiber bond formation during sheet forming. This is conceivable because lignin is hydrophobic by nature, or more precisely, lignin is much less hydrophilic than cellulose. Koljonen[12] et al. studied the precipitation of lignin and extractives on kraft pulp fibers under acidic conditions in the bleaching process and found that not only lignin but also, extractives and metals precipitated on the fiber surface. The strength and bonding properties of fibers were impaired slightly by the precipitated materials. However, the effect of precipitated lignin on the fiber bonding properties was not separated form other factors in their study, and little has been reported regarding the effect of fiber surface lignin on the interfiber bonding.

The interfiber bonding strength is usually evaluated in terms of the specific bond strength (SBS) and the relative bonded area (RBA). To get the SBS, the internal bond strength (IBS) which is defined as the energy or strength required for breaking bonds per unit area of the paper sheet, has to be determined. The IBS can be measured either on single fiber crossings or on paper.[13–15] Several methods have been developed for the IBS measurement such as the z-directional tensile test, the delamination test, and the Scott bond test. Although the results from different methods are different, they are highly correlated. The Scott bond test method was used in the present study.

There are also several methods for determining the RBA, which is defined as the ratio of bonded area to the total surface area of pulp fibers. These methods include microscopic observations, determination of the light scattering coefficient, and gas adsorption.[13–19] The widely used light scattering coefficient method was adopted in the present study because it is convenient to use and the results obtained from this method are linearly correlated with those from the gas absorption method.[17]

For mechanical pulp, interfiber bonding ability is even more important. Because it has better printability and relatively low cost, mechanical pulp has been used increasingly in printing paper grades. However, the interfiber bonding strength sets the limit for the proportion of mechanical pulp that can be used in various paper grades. Depending on the specific process conditions, that is, refining temperature and chemical treatment, the fiber separation line on the cell wall can be quite different, which may result in different lignin concentrations on the fiber surface. It was found in our previous study[10,20] that a mechanical peeling process can remove the lignin-rich material on the surfaces of CTMP fibers. If lignin on the fiber surface constitutes a barrier for interfiber bonding, this mechanical peeling technique may be used for modifying the fiber surface for improved interfiber bonding.

The objective of this study was to investigate the effect of the fiber surface lignin on interfiber bonding. Both chemical pulp and mechanical pulp were used to evaluate how the fiber surface lignin affects the specific bond strength and the relative bonded area. It was hoped that this investigation would improve the fundamental understanding of interfiber bonding and provide guidance for producing mechanical pulp fibers with surfaces favorable to improved interfiber bonding.

EXPERIMENTAL

Materials

Unbleached and fully bleached kraft pulp from mixed softwood chips was obtained from a commercial production line. The lignin content of the unbleached pulp was 3.2% (Klason lignin). Aspen CTMP was provided by Tembec Inc. Kraft lignin, *Indulin AT*, used for lignin adsorption experiments, was provided by Westvaco Co.

Mechanical Peeling of CTMP Fiber Surface Material

A PFI mill was used at 30% consistency instead of the standard 10%. The bar-bar clearance was adjusted to 0.35 mm and the standard weight (pressure) applied to the roll was removed. Under these conditions, the abrasive and surface peeling actions are expected to prevail.[10,20]

Lignin Adsorption on Pulp Fibers

Kraft lignin was dissolved in 10 g/L aqueous sodium hydroxide to prepare a 20 g/L lignin solution. The initial pH was adjusted to 12.0 with 0.5 M H_2SO_4. The fully bleached kraft pulp and the alkaline lignin solution were mixed together with a pulp/liquor ratio of 1:20. The adsorption was carried out at 80°C for 2 h. The detailed procedure has been described previously.[11] Three replicate adsorption experiments were conducted and average results are reported. The pH after adsorption was 11.5–12.0.

Surface Lignin Determination by X-ray Photoelectron Spectroscopy (XPS)

The fiber surface lignin concentration was determined by XPS. XPS was performed on a Leybold max200 X-ray photoelectron spectrometer. The fibers were made into sheet form and dried in air. The analyzed area was 1 × 7 mm. Atomic ratios of oxygen to carbon (O/C) were determined by operating in a low-resolution mode with a pass energy of 192 eV. The sensitivities for carbon and oxygen were 0.32 and 0.75, respectively. The lignin concentration calculation was based on the O/C ratios of the samples and average molecular formulae according to a method described in details in the literature.[4,21]

SEM Observation of Fiber Surfaces

CTMP fiber samples were dehydrated with ethanol and dried with critical point drying method. The samples were mounted on a sample holder and coated with gold. SEM imaging was performed with a JSM-6400 scanning electron microscope with a digital imaging system at 5 kv.

Handsheet Testing

Handsheets were prepared with a nominal basis weight of 80 g/m^2 on a British standard handsheet mold and then pressed and dried according to TAPPI standard method T205 om-88.[21] Handsheets were conditioned at 50% RH and 23°C for at least 48 h prior to testing. Measurement of basis weight, thickness, and tensile strength were performed according to TAPPI standard test methods.

Determination of the Specific Bond Strength (SBS) and the Relative Bonded Area (RBA)

The internal bond strength (IBS) of sample sheets was tested by the Scott Bond method using a Monitor/Internal Bond Test Station manufactured by TMI Test Machines Inc.[22] Samples were prepared on the I-Bond Sample Prep Station accompanying the internal bond testing station. Ten measurements were made on each sample sheet and the average result was reported.

Light scattering coefficients (S) of the handsheets were determined according to TAPPI standard method T425om-91.[23] The light scattering coefficient for the unbonded sheet (S_o) was obtained by extrapolation of the plot of the scattering coefficient against tensile strength.[24] The specific bond strength and the relative bonded area were calculated as follows:

$$\mathrm{SBS} = \mathrm{IBS}/\mathrm{RBA} \tag{1}$$

$$\mathrm{RBA} = (\mathrm{S_o} - \mathrm{S})/\mathrm{S_o} \tag{2}$$

RESULTS AND DISCUSSION

Interfiber Bond Strength of Unbleached and Bleached Kraft Pulp

Unbleached kraft pulp has been reported to have a surface lignin concentration several times higher than the corresponding bulk lignin concentration. Therefore, by comparing the interfiber bond strength of unbleached pulp fibers and fully bleached pulp fibers, the influence of surface lignin on interfiber bonding can be revealed. As listed in Table 1, the internal bond strengths of the unbleached fibers and the fully bleached fibers were 137.2 and 188.6 $\mathrm{J/m^2}$, respectively. This means that in a unit sheet area, the bond strength of fully bleached pulp fibers, which is assumed to have virtually no lignin, is about 44% higher than that of unbleached fibers. The lower internal bond strength of unbleached fibers can be ascribed to the presence of lignin on the fiber surface. However, it should be noted that the difference in the amount of hemicellulose and the DP of cellulose between bleached and unbleached pulps may also affect interfiber bonding. In the present study, the same bleached and unbleached pulps were used in all the experiments, so the two factors can be considered to be approximately constant.

Lignin is also present in the bulk of the unbleached pulp fibers, and the lignin content of the unbleached kraft pulp used in the present study was 3.2% (Klason lignin). If the amount of residual lignin is high, it may result in a difference in fiber flexibility and collapsibility between unbleached pulp fibers and bleached pulp fibers. If this is the case, the bonded area or contact area between fibers would be different for the unbleached fibers and

Table 1. Internal bond strength of a mixture of unbleached and fully bleached kraft softwood pulps

Percentage of unbleached pulp	0%	50%	67%	75%	100%
Light scattering coefficient ($\mathrm{m^2/kg}$)	30.25	27.72	27.05	27.52	29.84
RBA (%)	23.59	29.98	31.67	30.49	24.63
IBS ($\mathrm{J/m^2}$)	188.6	182.0	174.4	168.6	137.2
SBS ($\mathrm{J/m^2}$)	799.5	607.1	550.7	612.7	557.0

[a] $\mathrm{S_o} = 39.59\ \mathrm{m^2/kg}$.

the fully bleached fibers. Therefore, the lower IBS of unbleached fibers might not be only from the interference of surface lignin with the formation of hydrogen bonds between cellulose molecules. However, the RBAs determined for the unbleached and the fully bleached pulp were virtually the same, which means that lignin on the fiber surface or inside the fiber wall did not affect the bonding area, or that the difference caused by lignin in RBA was not significant enough to be detected by light scattering analysis.

It is clear that the lowered internal bond strength for unbleached fibers is mainly due to the reduced specific bond strength. Specific bond strength can be interpreted in terms of the strength of single bonds or the number of the bonds in the bonded area. Due to the hydrophobic nature of lignin, the presence of lignin in the bonded area may constitute a barrier for the formation of hydrogen bonds between cellulose molecules. Because the distance between two optical contact surfaces could be in the order of a few hundred angstroms and the hydrogen bonding and Van der Waal forces operate over a distance of a few angstroms, it is not possible to distinguish between the reduction of the number of bonds or the reduction of the strength of the bonds caused by lignin. To get a more quantitative sense of the influence of lignin on interfiber bonding, the unbleached pulp and the fully bleached pulp were mixed in various ratios and paper sheets were made from the mixed pulps to determine the IBS and RBA. The basic idea was that when unbleached pulp is mixed with fully bleached pulp, a lignin-rich surface will be introduced in the contact area between fibers in a sheet of paper, and increasing the proportion of unbleached pulp, the lignin-rich surface area will be increased. It can be seen from Figure 1 that the SBS

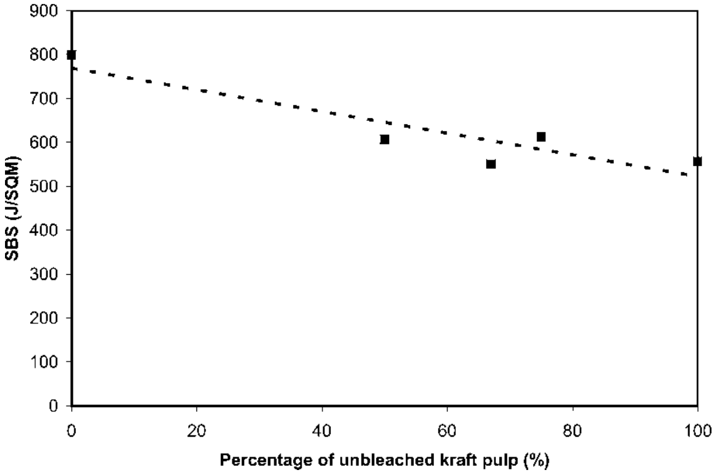

Figure 1. Specific bond strength changes with the percentage of unbleached kraft pulp in the mixed pulp.

decreased with increasing the fraction of unbleached pulp. The more area covered by lignin, the lower bond strength. This is expected because increasing the amount of lignin present in the contact areas will block more interfiber bonds.

Figure 2 is a plot of light scattering coefficient versus tensile strength for extrapolating to obtain S_o and the total surface area of the unbleached pulp according to Ingmanson and Thode.[24] It was used for calculating the RBA of sheets made from the unbleached pulp, the bleached pulp, and the mixtures of unbleached pulp and bleached pulp. This approximation is reasonable given the fact that in the extrapolation method, S_o is calculated by assuming that it is unchanged by refining the pulp to various levels.

Interfiber Bond Strength of Fully Bleached Pulp Fibers with Lignin Adsorbed on the Fiber Surface

Lignin can be sorbed on fully bleached pulp fibers, with adsorption on the surface being the main deposition.[11,12] By adsorbing lignin on the fiber surface, a lignin-enriched sample can be created, which can be used for comparing internal bond strength with fully bleached pulp. This may be a more direct way to investigate the effect of surface lignin on interfiber bonding. Table 2 lists the results from the two samples. After the lignin adsorption, the surface lignin concentration on the fully bleached fiber surface was about 16–18% as determined by XPS, whereas the overall lignin concentration in the pulp was only 0.8–1.0% as determined by kappa number. The result of IBS appears unexpected since after lignin adsorption on the fiber surface, the IBS even increased compared with the fully bleached pulp. However, the RBA of the sample after lignin adsorption also increased, so the SBS of the sample after lignin adsorption actually

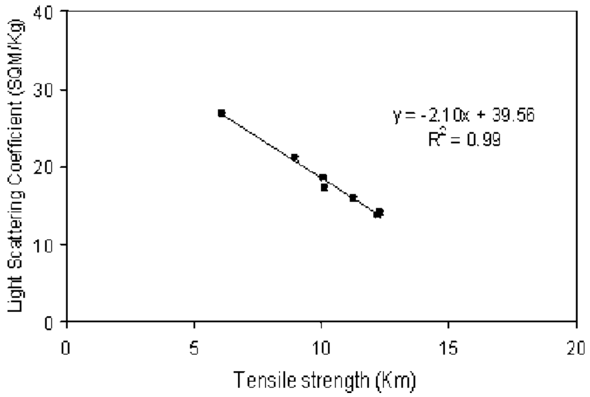

Figure 2. Extrapolation of S_o for unbleached kraft softwood pulp.

Table 2. Internal bond strength of fully bleached kraft pine pulp before and after lignin adsorption

Pulp samples	Before lignin adsorption	After lignin adsorption	Control experiment
Surface lignin concentration (%)	0.5%	16–18%	0.5%
Light scattering coefficient (m^2/kg)	31.64	29.4	29.9
RBA (%)	20.1	25.7	24.4
IBS (J/m^2)	204.1	243.9	244.7
SBS (J/m^2)	1020	943	1005

$^a S_o = 39.59\ m^2/kg$.

decreased. This is in agreement with the foregoing discussion that the surface lignin reduces the interfiber bond strength.

The reason for the increased RBA after lignin adsorption is probably because of the high pH used in the adsorption experiment. During the adsorption experiment at pH above 12, the fiber wall is expected to swell significantly. The swelling of the fiber wall will make fibers more flexible and more collapsible, thus resulting in more bonded area in the sheet of paper. To confirm this, a control experiment was performed to determine the RBA change of the samples due to swelling, in which the adsorption experiment was conducted without adding lignin in the system. It can be seen from Table 2 that the RBA of 24.4% of the control sample is very close to the RBA of pulp after lignin adsorption, in comparison with the RBA of the original pulp. This indicates that the alkaline condition used in the adsorption experiment promotes fiber swelling, which increases the RBA of the pulp samples.

Interfiber Bond Strength of CTMP Pulp Fibers with Different Coverage of Middle Lamella Remainders

Depending on the chemical pretreatment and refining conditions, fiber separation takes place more or less in the middle lamella region or the cell wall area. A substantial amount of middle lamella material can be retained on the surface of CTMP fibers. Middle lamella material is highly rich in lignin, the concentration of lignin being about 70–80%.[25] The middle lamella remainder on the fiber surface is expected to cause deteriorated bonding ability for the fibers due to its high lignin concentration. In our previous studies,[10,20] a mechanical peeling technique was developed that can specifically remove the fiber surface material of CTMP fibers leaving the rest of the fibers almost intact. The basic idea of the mechanical peeling technique is to use high pulp consistency (30%) and large bar-bar clearance (0.35 mm) in the PFI operation so the abrasion and brushing actions in refining are enhanced and the cutting and splitting actions are essentially eliminated. This kind of PFI condition will facilitate rubbing off or peeling off the fiber surface material.

Using this technique, CTMP fibers with different middle lamella coverage were produced analysis of their interfiber bonding ability. Figures 3–5 show the middle lamella material remaining on the fiber surface and the removal of these materials by the mechanical peeling process.

It can be seen from Table 3, that the CTMP fibers used in the present study had the average middle lamella coverage of about 15–20% estimated by SEM

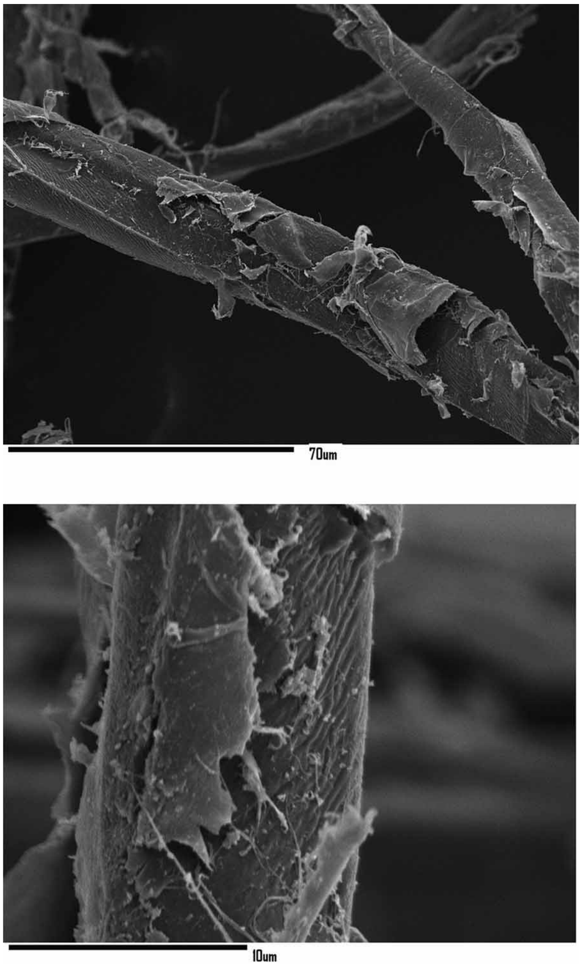

Figure 3. Middle lamella material on the CTMP fiber surfaces.

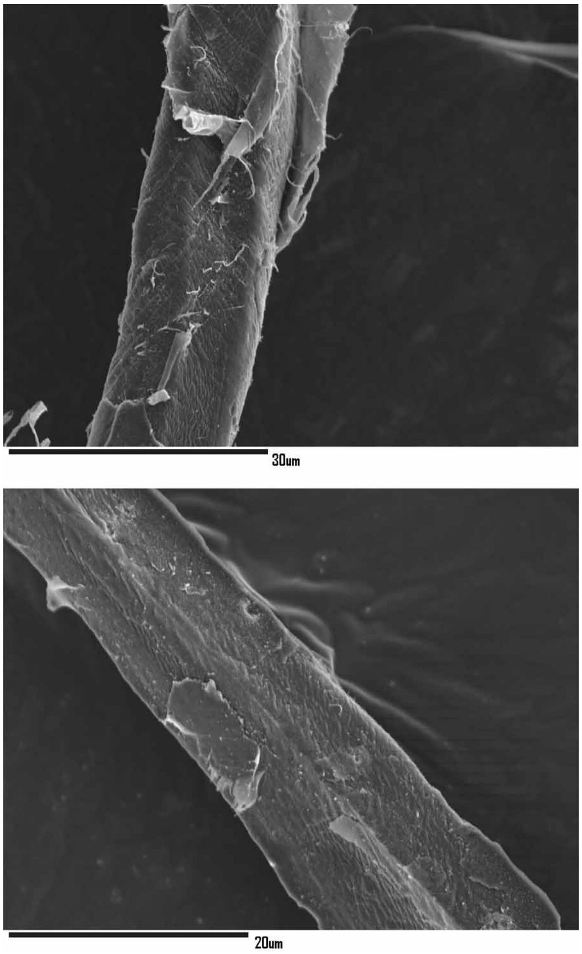

Figure 4. Middle lamella material on the CTMP fiber surface after 12,000 PFI revolutions.

observation. After the mechanical peeling treatment with 12,000 PFI revolutions, the middle lamella coverage was reduced to less than 5%. The IBS for the two pulp samples were 16.29 and 22.71 J/m^2, respectively. This shows that after the removal of middle lamella material from the fiber surface, the bond strength increased by almost 40%.

Table 3 also lists the RBA and the IBS of the pulp samples after different PFI revolutions. It can be seen that the IBS increased with an increase in the PFI revolutions used. This is understandable because the longer the PFI peeling treatment, the more middle lamella material will be removed from the fiber surface. However, it can be seen from the SEM images that after

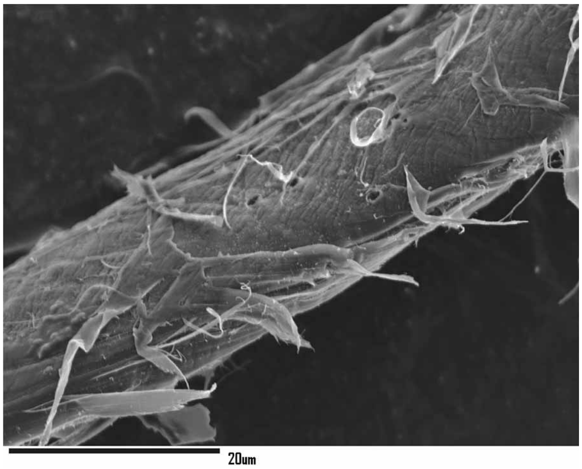

Figure 5. Surface fibrillation of a CTMP fiber after 24,000 PFI revolutions.

12,000 PFI revolutions the middle lamella material was almost completely removed. When the PFI treatment was continued to 24,000 revolutions, external fibrillation occurred. The same information can be obtained from the plot of the IBS versus PFI revolutions in Figure 6. The IBS increased slowly from 0 to 12,000 PFI revolutions, and then increased rapidly with 24,000 PFI revolutions. In fact, at from 6000 to 12,000 PFI revolutions, the IBS leveled off. This implies that up to 6000 PFI revolutions, the increase of the IBS resulted mainly from removal of middle lamella material. From 6000 to 12,000 PFI revolutions the middle lamella coverage did not change much because it was already reduced to a very low level, so the IBS did not improve significantly. When the PFI treatment proceeded to 24,000 revolutions, the increase in the IBS could be attributed to external fibrillation. Therefore, it is clear that in order to remove only the middle lamella material, the PFI revolutions must be kept below a certain level. This is

Table 3. Internal bond strength of Aspen CTMP after surface peeling treatment with a PFI

PFI (revs)	0	3000	6000	12,000	24,000
Middle lamella coverage (%)	15–20	—	—	<5	—
Light scattering coefficient (m^2/kg)	42.48	41.83	40.51	40.48	42.25
IBS (J/m^2)	16.3	19.5	22.1	22.7	36.7
Tensile strength (km)	0.22	0.56	0.72	0.88	1.46

[a] Middle lamella coverage estimated by SEM observation.

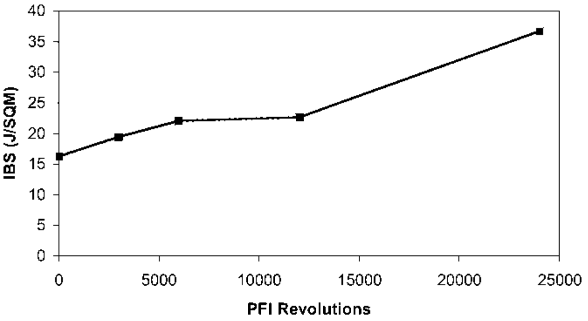

Figure 6. The increase of internal bond strength of CTMP fibers with the surface peeling treatment.

consistent with the original idea for the mechanical peeling technique, that is, the peeling action should be gentle enough so that only the lignin-rich surface material is removed without significant loss of pulp yield.

The RBA of the samples decreased only slightly as PFI peeling proceeded. It is apparent that the extrapolation to obtain S_o and the total unbonded area used by Ingmanson and Thode[24] cannot be applied. Swanson and Steber[17] mentioned that the extrapolation to obtain S_o by plotting tensile strength against light scattering coefficient may not be appropriate for mechanical pulp because the total unbonded surface area may change after refining. Nevertheless, with the mechanical peeling treatment in the present investigation, the light scattering coefficient did not change much for different PFI revolutions, that is, the RBA remained literally the same. Therefore, even though the RBA can not be calculated because of the lack of the S_o, the influence of surface lignin on interfiber bonding strength can be accounted for directly from the IBS result.

It can also be seen from the results of this study that with a simple mechanical peeling treatment, the internal bond strength of CTMP fibers can be increased by 40%. If a similar technology can be incorporated in the existing industrial refiner process, the quality of CTMP fibers can be greatly improved. Further investigation in this direction is ongoing in our laboratory.

SUMMARY

The results from the present investigation show that if lignin is present on the fiber surface of either kraft pulp or CTMP, the internal bond strength of the pulp fibers will be reduced. Because the relative bonded area determined for the pulps with surface lignin and without surface lignin did not differ much, the reduced internal bond strength is ascribed to the reduced specific bond strength, and in turn, to the interference of lignin with the formation of hydrogen bonds between cellulose molecules in the bonded area.

It is conventionally believed that lignin present on the fiber surface will block the formation of hydrogen bonds between cellulose molecules on the fiber surface due to the hydrophobic nature of lignin. However, the result from the present work is rather preliminary and no direct evidence can be provided to support this notion. More fundamental understanding of the interaction between lignin and cellulose may be obtained by atomic force microscopy analysis, and this will be carried out in the future work. In addition, there are several different techniques for measuring the internal bond strength and relative bonded area. Further investigation is needed in using an appropriate technique that can reveal the true value of the specific bond strength. Nevertheless, the present investigation was directed toward a relative comparison between fibers with surface lignin and fibers without surface lignin, and the results shed light on the effect of surface lignin on interfiber bonding.

Lignin-rich surface material on CTMP fibers was found to reduce the internal bond strength of the fibers. More interestingly, it was found that by removing the surface material, the internal bond strength of the CTMP fibers was improved by 40%. If this technique can be further incorporated in the industrial process, the interfiber bonding ability of commercial CTMP would be greatly improved.

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