The Middle Lamella Remainders on the Surface of Various Mechanical Pulp Fibres

KECHENG LI*, XUEQUAN TAN†, AND DONGBO YAN‡

*Associate Professor, †PhD Student, ‡PhD Student
*Department of Chemical Engineering and
Limerick Pulp and Paper Research and Education Centre,
University of New Brunswick, Fredericton, NB, Canada
kecheng@unb.ca

ABSTRACT

The surface of various mechanical pulp fibres including thermomechanical pulp (TMP), chemithermomechanical pulp (CTMP), and alkaline peroxide mechanical pulp (APMP) fibres, were characterized using SEM, AFM, and XPS. With SEM and AFM, middle lamella material was observed to be non-fibrillar, patch-like, while fibre secondary wall was observed to have a microfibrillar structure. It was found that after the first-stage refiner, lignin-rich middle lamella remainders are present on the fibre surface of all three pulps, although most of the fibre surfaces expose microfibrillar structure. After the final-stage refining, large amounts of granules are present on the TMP fibre surface. In contrast, most middle lamella remainders remain on the surface of CTMP fibres after final stage refining and even after peroxide bleaching. XPS results have confirmed that the non-fibrillar surface material is the lignin-rich middle lamella remainder., and the remainders of middle lamella contribute to the high surface lignin concentration.

Keywords: Thermomechanical pulp (TMP); Chemithermomechanical pulp (CTMP); Bleached CTMP; Alkaline Peroxide mechanical pulp (APMP); Scanning electron microscopy (SEM); Atomic Force Microscopy (AFM); X-ray photoelectron spectroscopy (XPS); Surface lignin; Middle lamella.

INTRODUCTION

Mechanical pulps including thermomechanical pulp (TMP) and chemi-thermomechanical pulp (CTMP) are a major furnish component for a variety of grades of paper. As more and more supercalendered paper is manufactured, the demand for high quality TMP is increasing, which is mainly for producing SC paper with high smoothness, high opacity and high physical strengths [1]. Bleached hardwood CTMP now can be used in high quality printing and wood-free paper such as writing paper and coated paper. The use of BCTMP in wood-free paper not only reduces the cost of raw material but also improves paper quality, i.e. higher bulk, higher opacity and better paper formation [2].

Recently, alkali-peroxide mechanical pulping (APMP) technology has been developed, which can produce the same quality pulp as BCTMP but with a saving of up to 40% refining energy [3,4]. Many research works have been focussed on the mechanical pulping technology in order to further improve the pulp quality and reduce production costs.

The major difference between mechanical pulp and chemical pulp is that mechanical pulp retains almost all the lignin in wood. Lignin is hydrophobic in nature. Lignin in fibres not only makes the fibres less flexible, but also impairs the interfibre hydrogen bonding, both of which will contribute negatively to the paper strength. Furthermore, the lignin present on the fibre surface will play a more important role in interfibre bonding since fibre-fibre interaction and bonding takes place on the fibre surface.

Several studies have shown that lignin is highly enriched on the fibre surface after chemical pulping [5,6,7,8]. Li and Shao [9] found that increased surface lignin concentration decreases the interfibre bonding strength. Li and Reeve [6] found that the high surface lignin concentration originates mainly from the re-adsorption of dissolved lignin in pulping liquor on the fibre surfaces, rather than from the remainder of the middle lamella material. Duchesne et al. [10] confirmed this finding and also showed that in an ITC kraft pulping, middle lamella is completely removed. However, in the mechanical pulping process, fibre separation is achieved not by dissolving lignin but by physically grinding or

rupturing wood, thus separating fibres from one another. If the separation takes place in the middle lamella region, middle lamella material will most likely attach to or remain on the surface of the adjacent fibres. Middle lamella has about 60% lignin [11]. The remainder of middle lamella on the fibre surface will definitely contribute to high surface lignin concentration.

Various surface analysis techniques are available for fibre surface characterization, both physical and chemical, which include scanning electron microscopy (SEM), field-emission SEM (FE-SEM), atomic force microscopy (AFM) and x-ray photoelectron spectroscopy (XPS) [12,13,14,15,16,17,18,19,20]. SEM has been used widely for analysis of the fibre morphology and fibre surface structure and their changes in the pulping and refining processes [12]. If an appropriate drying method is used, such as critical point drying (CPD) or freeze drying (FD), the surface structure of the entire surface area of a fibre width can be clearly observed at various magnification levels. This is important since the result from a larger view can be more representative. In addition, once the entire fibre width is in the same view, it is easier to identify the location of certain features on the fibre and to distinguish different structures by comparing them to the neighbouring structures. In contrast, AFM provides a much better resolution, allowing the fine structure of the cellulose microfibrils to be observed on a nanometer scale [14,15,16]. Therefore, the physical structure of the fibre surface can be detected with high accuracy. Different from SEM and AFM, XPS can provide elemental or chemical information of the fibre surface. Therefore, by using XPS, fibre surface concentration of different substances such as lignin and cellulose can be determined [6,7,8,19].

The objective of the present investigation is to characterize the surface structure of various mechanical pulp fibres with regard to the presence of middle lamella remainders. It is expected that the knowledge obtained from this work can shed light on elucidating the fibre surface property, in particular, the surface lignin in relation to the mechanical pulping process, which will then provide guidance on producing desirable fibre surface property by adjusting or modifying the refining process.

EXPERIMENTAL

Pulp Samples

Aspen CTMP and BCTMP were obtained from a commercial production line. The production line uses a two-stage refining system and a hydrogen peroxide bleaching process. Black spruce TMP samples were obtained from an SC paper production line. The process uses a three-stage pressurized refining system. Eucalyptus Grandis APMP samples were obtained from a commercial production line.

PFI Treatment

CTMP fibres were treated in a PFI mill in order to peel off the surface materials. 30 grams (o.d.) of pulp was used for each run and the pulp consistency was 30%. The clearance between the roll and the housing was set to be 0.35 mm, and the additional weight for refining pressure was removed.

SEM Observation

Pulp fibres were dehydrated through a graded series of ethanol from 30% to 100 % and then dried in a Balzers CPD030 Critical Point Drier. The dried samples were mounted on aluminium stubs and coated with gold in a Polaron S150A Sputter Coater. The samples were examined with a JEOL JSM-6400 Scanning Electron Microscope at 10 kV, and digital images were recorded.

AFM Observation

A single wet fibre was carefully placed on the AFM sample holding stub and then fixed with double adhesive tape. AFM observations were performed in open air at room temperature immediately after the sample was prepared. A multimode AFM (Digital Instruments) with NanoScope IIIa controller and J type Scanner was used. The samples were scanned with a silicon nitride tip mounted on a V-shaped cantilever (Veeco NP-S20, spring constant of 0.06 N/m). The tip radius is 10nm. Surface height and deflection images were obtained by scanning samples in contact mode, at a scan rate of 1.5Hz. Deflection or error signal image, which shows more detailed surface structure than the height image were used in this study.

XPS Analysis

X-ray photoelectron spectroscopy (XPS) was performed on a Leybold Max200 X-ray photoelectron spectrometer. Samples were mounted on a sample holder and the analysis was performed under a vacuum less than 10^{-9} torr. The analyzed area was 1x7 mm. Monochromated Al Kα sources were used and operated under 12 or 15 kV and at 15-25 mA. The XPS spectra were obtained with a photoelectron take-off angle of 90° relative to the sample surface.

Fibre samples were prepared in sheet form. After drying, all samples were subjected to Soxhlet extraction with acetone and distilled water in series, four hours for each solvent. The samples were then placed on clean glass slides, pressed lightly, and then dried in an oven at 60°C. The glass-smooth side of the sample was used to provide a smooth, dry surface for the XPS measurement

[6].

RESULTS AND DISCUSSION

Middle Lamella Remainders on the Surface of Fibres from the First-Stage Refiner

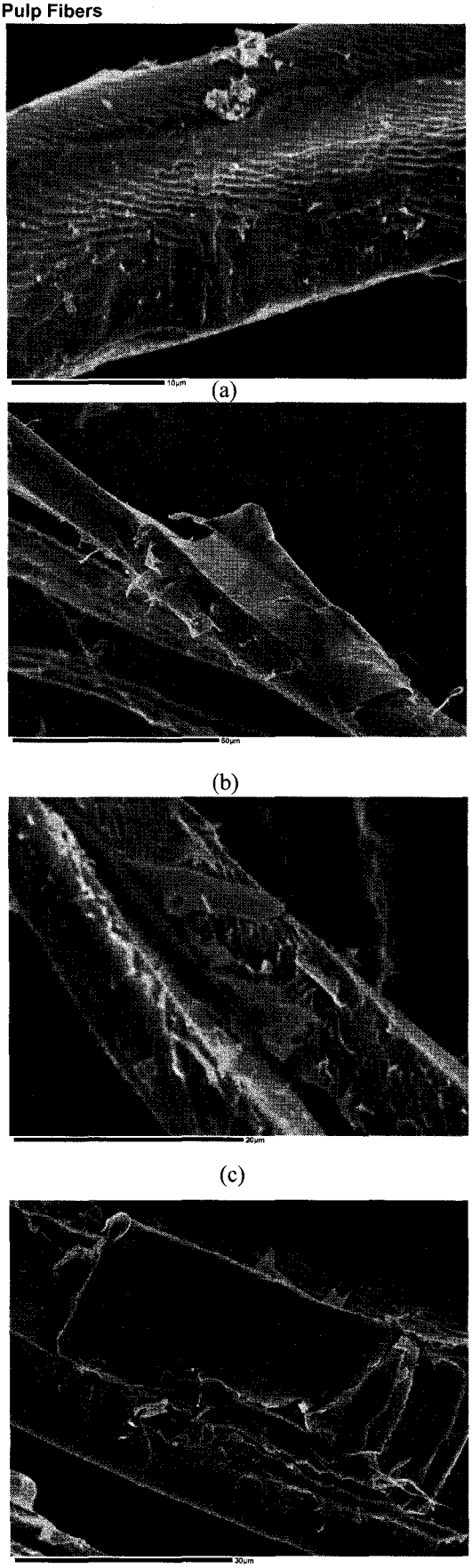
Defibrization or fibre separation takes place mostly in the first stage refiner. In the subsequent refiner stages, fibres are further treated for strength development, and shives and fibre bundles are reduced. Therefore, by examining the surface structure of fibres from the first-stage refiner, the fibre separation lines can be identified. In addition, since some of the fibres are not completely separated from each other, the middle lamella material can be more easily identified by its location and the morphological features.

Figure 1 shows some typical CTMP fibres from the first-stage refiner. It can be seen from Figure 1(a) that the fibre surface has a clear microfibrillar structure, which means that the fibre separation occurs mostly in the secondary wall. However, as shown in Figure 1(b), a large patch of non-fibrillar material attaches onto the microfibril structure of the fibre outer surface. Figure 1(c) shows the similar non-fibrillar patches being present between the two adjacent fibres. This is the location where middle lamella is present in wood gluing the two fibres together. A more distinct non-fibrillar, patch-like feature is shown in Figure 1(d). The location is the cell corner when the fibre is in wood.

All the patch-like materials on the fibres show no microfibril structure, which is in agreement with the material structure of the middle lamella. It is well known that middle lamella consists mainly of lignin, hemicellulose and pectin. There is little cellulose fibrillar structure in it, except that when it approaches the primary wall, some middle lamella material will penetrate into the crossed fibrillar structure of the primary wall [11]. Therefore, from the morphological feature and the location, it can be identified that the smooth, patch-like material on the fibre surface as observed is the middle lamella material remaining on the fibre surface after fibre separation.

In fact, Börås and Gatenholm [17] proposed a model for the presence of lignin, extractives and carbohydrates on the fibre surface based on their experimental results, in which, lignin is proposed to be present on the fibre surface in patches. From the hysteresis between advancing contact angle and receding contact angle measured on single CTMP fibres after removal of extractives, they conclude that lignin is not homogeneously distributed on the fibre surface but is present as patches.

Figure 2(a,b,c) show the typical surface structure of the APMP pulp fibres. Similar to the CTMP fibres, the middle lamella remainder can be easily identified.

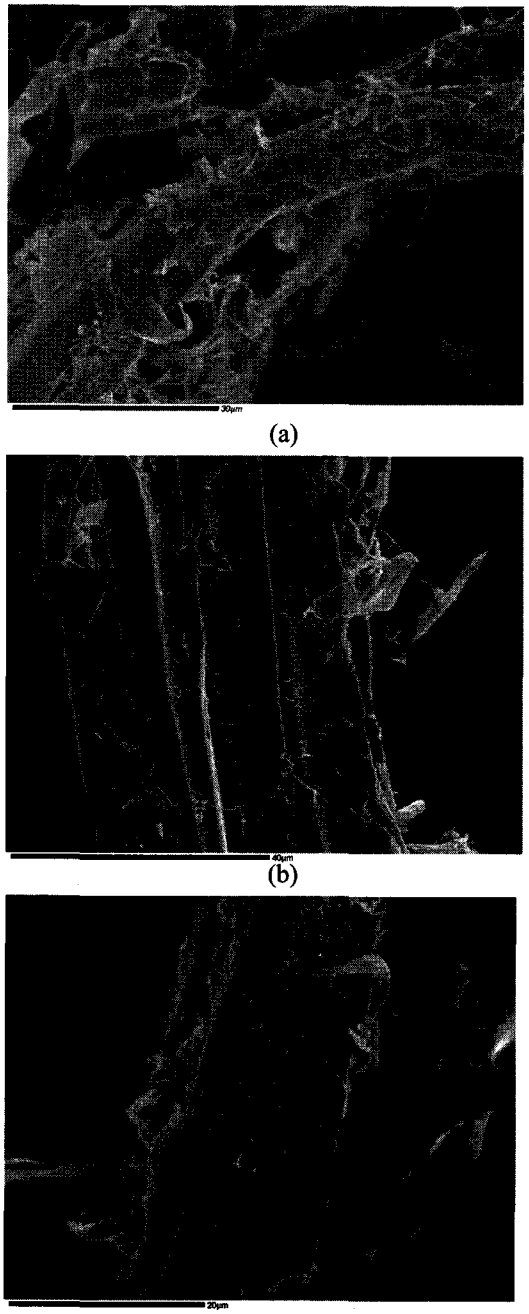


(a) (b) (c) (d)

Fig. 1 Surface Morphology of CTMP Fibres from the First-Stage Refiner

Figure 2(a) shows a fibre which is still attached to several

other fibres, i.e. a fibre bundle. It can be seen that a large amount of patches are present on the fibre surface. Figure 2(b) shows the presence of non-fibrillar material between two fibres, in contrast to the microfibrillar structure on surface of the three fibres. The exposure of the secondary wall microfibrils on the fibre surface can also be clearly identified as shown in Figure 2(c).



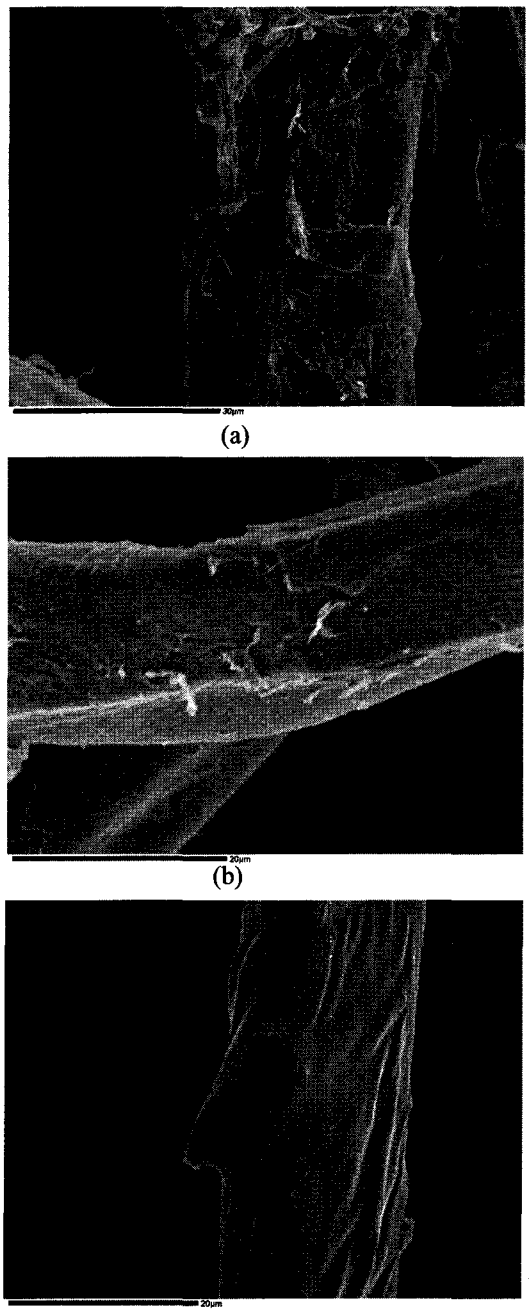
(a)

(b)

(c)

Fig. 2 Surface Morphology of APMP Fibres from the First-Stage Refiner

Compared with the CTMP and APMP fibres, the TMP fibres have lower surface coverage of middle lamella material as shown in Figure 3 (a,b). The microfibrillar pattern of the TMP fibres is clearer than that of CTMP. This is due to the fact that, in the CTMP process, lignin in the middle lamella region is softened by the chemical treatment and thus fibre separation occurs more in the middle lamella region or middle lamella and primary wall region.



(a)

(b)

(c)

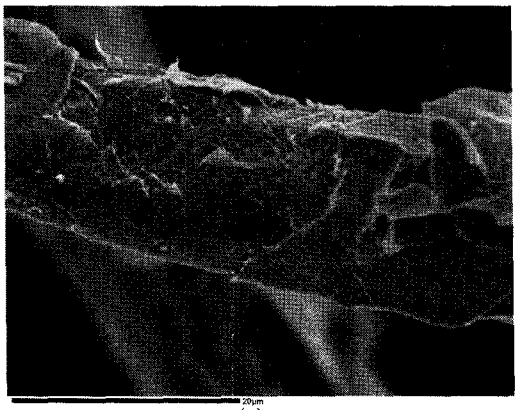
Fig. 3 Surface Morphology of TMP Fibres from the First-Stage Refiner

A more interesting finding is shown in Figure 3(c), i.e., a very smooth, smelt-like layer is present on the fibre surface, and beneath it is shown some particle-like features. It is well understood that at higher

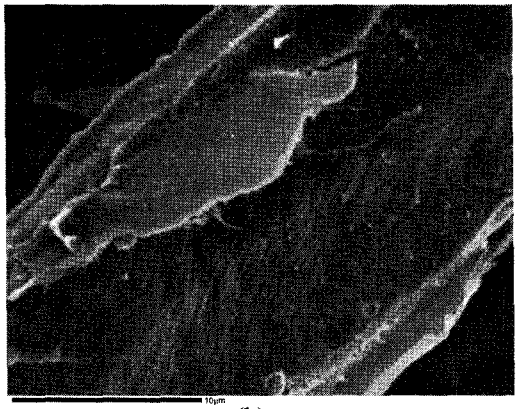
temperature in the reforming zone, lignin would melt. If we compare it to the well-characterized fibre infrastructure, i.e. non-fibrillar middle lamella, crossed microfibril structure in the primary wall, and parallel mircofibril structure in the secondary wall [11], this smooth layer is most likely to be the melted lignin on the fibre surface. It is reasonable to assume that at high temperature, lignin, most probably from the middle lamella remainder in this case, melts and then spreads over the nearby fibre surface area and forms a coating on it when the temperature goes down as the pulp fibres exit the refining zone. The particles covered beneath the melted lignin are likely to be the middle lamella remainder which does not melt.

Middle Lamella Remainders on the Surface of Fibres from the Final-Stage Refiner

The middle lamella remainder on the fibre surface will definitely increase the overall surface lignin concentration since the lignin content of middle lamella material is about 60%, in comparison with that of the secondary wall which is only about 20 - 30% [11].



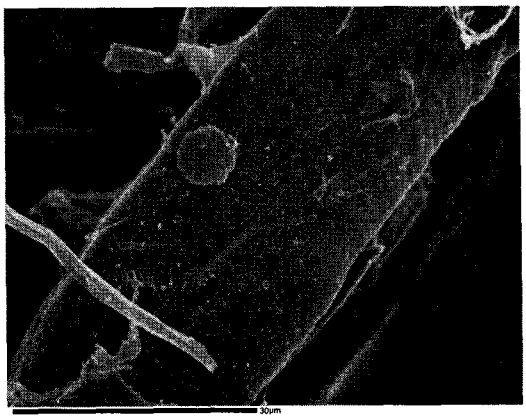
(a)



(b)

Fig. 4 Surface Morphology of CTMP Fibres from the Third-Stage Refiner

High surface lignin will not only affect the interfibre bonding strength, it may also affect the flexibility and



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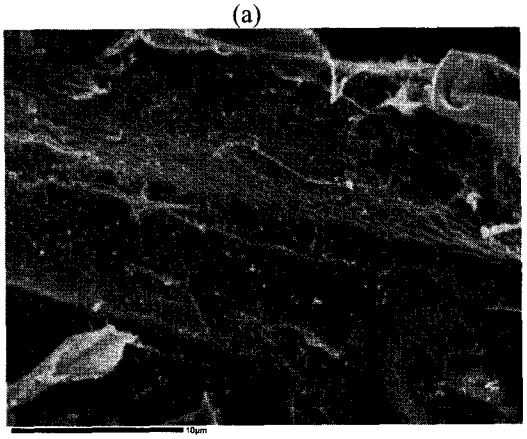

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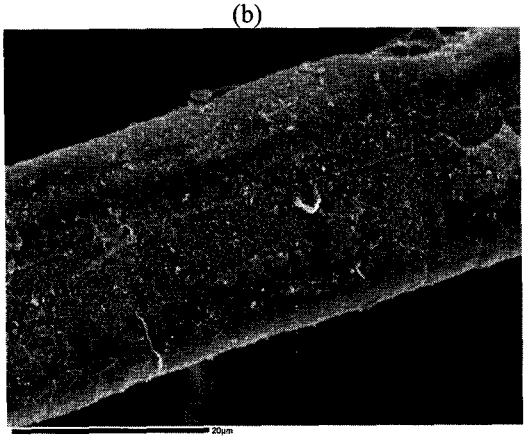

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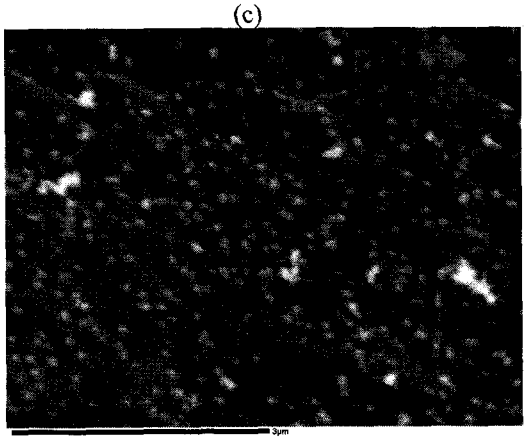


(d)

Fig. 5. Surface Morphology of TMP Fibres from the Third-Stage Refiner

collapsibility of an individual fibre, particularly when the melted lignin coats the fibres as shown in Figure 3(c). In addition, the layer of lignin coating on the fibre surface may also affect the fibre development, i.e. fibre fibrillation, in the subsequent refining process.

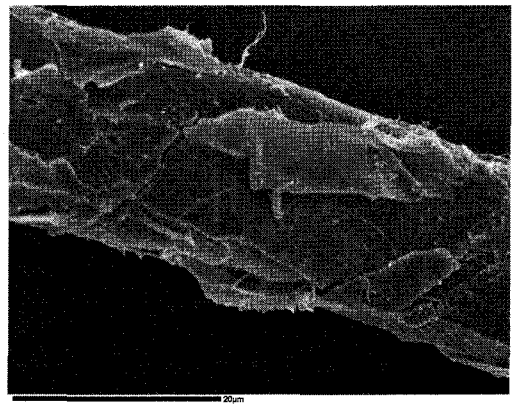
It can be seen from Figure 4 that even after the final refiner stage, the middle lamella remainders are still clearly seen for CTMP fibres although much reduced. The surface morphology of these fibres is quite heterogeneous with different microfibrillar patterns exposed and with some areas covered with middle lamella remainders. From the different microfibril angles, it can be interpreted that different lamellae of the secondary wall are exposed on the surface of a fibre. This indicates that although the further refining modifies significantly the fibre surface structure, the original features generated in the first-stage refining still remains to some extent.

However, in the case of the TMP fibres, after the final-stage refining, most of the middle lamella remainders has disappeared. Instead, a large amount of granular material is present on the fibre surface, as shown in Figure 5. The size of the granules is in the range of 50-300 nm as shown in Figure 5(d). Gustafsson et al. [14] also observed using AFM the granular structures on the surface of Finnish TMP fibres and the size of the granules was found to be in the range of 15-400nm. Simola et al. [15] studied the morphology of the surface lignin in the early stage of kraft pulping, i.e., after 10 min cooking, and after a complete pulping process, i.e. after 220min cooking. They found with AFM that lignin appears granular on the surface of fibres in the early stage of pulping. The granular lignin disappears gradually with cooking and more and more microfibrillar structure exposures on the fibre surface. They also examined the isolated lignin with AFM and found that the isolated kraft lignin also appears granular.

In fact, if large pieces of lignin-rich middle lamella material are present on the fibre surface after the first-refiner stage, further refining may grind them into small particles. Therefore, the present observation is in agreement with the studies reported in the literature. Since the granules are scattered quite evenly on the entire fibre surface, it is plausible to assume that some broken pieces of the middle lamella in the system may re-adsorb on the fibre surface during refining.

Middle Lamella Remainders on the Surface of BCTMP Fibres

Figure 6 shows the surface morphology of CTMP fibres after peroxide bleaching. It can be seen that most of the middle lamella remainders still look the same as they do before bleaching. However, some new features were found as shown in Figure 6(b). It looks like that part of some middle lamella remainders is dissolved or melted, similar appearance to what is observed on the TMP fibre as shown in Figure 3(c). This is probably due to the partial dissolution of some low molecular mass pectin, or hemicellulose, or sulphonated lignin in bleaching, or due to the melting of lignin at high temperature already happened in the preceding refining process.



(a)

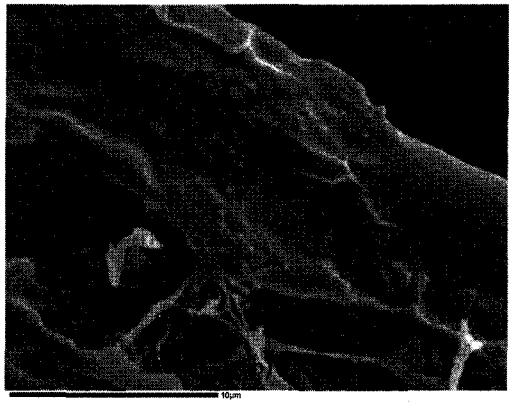


Fig. 6 Surface Morphology of BCTMP Fibres

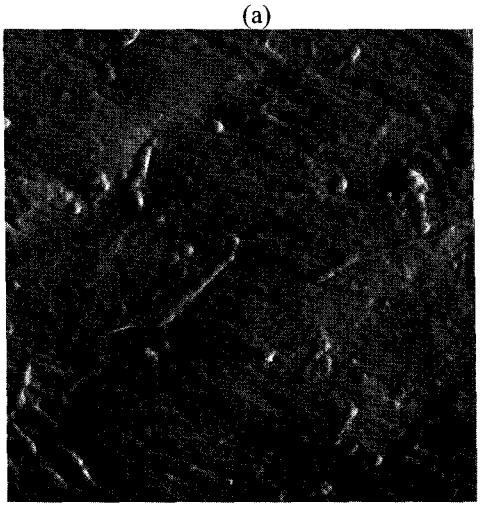
Nevertheless, the lignin present on the BCTMP fibre as such will affect the mechanical property and interfibre bonding ability of individual fibres. The observation is in agreement with a previous report that peroxide bleaching does not change the chemical composition of the CTMP fibres in terms of the atomic ratio of oxygen to carbon [17], neither does it change the distribution of lignin on the TMP fibre surface [21]. However, this does not exclude that peroxide bleaching can alter the fibre properties as reported in [22].

Fibre Surface Lignin Characterized by AFM and XPS

The identification of the middle lamella remainders is not only based on their location on the fibre surface, i.e., their presence between two adjacent fibres and their presence on the top of the microfibril structure, it is also based on the appearance of the texture in comparison

with the cellulose microfibril texture of the secondary wall. Since the ultrastructure of wood fibre walls is well understood, it is quite convincing that the smooth, non-fibrillar patches on the fibre surface of various mechanical pulp fibres observed with SEM are middle lamella remainders. In order to further confirm this conclusion, AFM and XPS were used to characterize the fibre surface structure and
determine fibre surface lignin concentration, respectively.

(a)



(b)

Fig. 7 Surface Morphology of CTMP Fibres before (a) and after (b) surface peeling. Contact AFM mode, deflection images. Scan size (a) 5x5 μm, (b) 2x2 μm.

Figure 7(a) shows the surface structure of a CTMP fibre. It can be seen that a large non-fibrillar patch is present on the fibre surface next to an area of microfibril structure. With several similar images, the diameter of the microfibrils was observed to be in the range of 20-60 nanometres. The diameter of the microfibrils observed is in agreement with the observation of the cellulose microfibrils reported in the literature [14,15]. Compared with SEM, the resolution of AFM is much high. As the microfibrils are observed to be on a diameter in nanometre scale, the structure of the patches still appears non-fibrillar. This further corroborates that the patches on the fibres surface are non-cellulose material.

In addition, a surface peeling technique was used to peel off the surface material of the CTMP fibres. Basically, a laboratory PFI mill is used and a special refining condition is set to enhance the rubbing-off effect between fibres so that mostly the surface material only is peeled off during the PFI treatment. Our previous study shows [6] that most of the non-fibrillar patched on the fibre surface can be removed by this process. It can be seen from Figure 7(b) that after this surface peeling process, fibre surface exposes mostly the microfills of the secondary wall. XPS analysis of the surface lignin concentration was performed on both original CTMP fibres and the PFI-peeled CTMP fibres. It can be seen from Table 1 that before surface peeling, the CTMP has a surface lignin concentration of 41.6 %, which is much higher than the total lignin concentration of the CTMP pulp, 16.8%, measured as Klason lignin. This is probably due to the remainders of the lignin-rich meddle lamella material present on the fibre surface. The contribution of middle lamella remainder to the surface lignin of pulp fibres from the early stage of kraft pulping was found in our previous study [6]. The gradual dissolution of middle lamella material in the early stage of kraft pulping was also imaged with FE-SEM by Duchesne and Daniel [18].

Table 1 Surface Lignin and Total Lignin Concentrations of the CTMP Fibres

	Surface lignin (XPS)	Total lignin (Klason)
CTMP fibres	41.6%	16.8%
CTMP fibres after peeling	16.3%	16.5%

After the surface peeling with a PFI, surface lignin concentration is reduced to the level of the total lignin concentration in the CTMP pulp. This proves that the peeled patch-like surface material is rich in lignin.

XPS analysis is based on the elemental composition of the substance detected so reveals the chemistry aspect of the substance, while SEM and AFM mainly reveal the physical feature of the material. Therefore, from both the chemistry and the physical or structural feature, the non-fibrillar, patch-like material on the fibre surface can be identified as the remainders of middle lamella.

CONCLUSIONS

With SEM, the surface morphology of various mechanical wood pulp fibres can be characterized. Middle lamella materials appear smooth and patch-like while fibre secondary wall shows clear microfibrillar structures. It has been found that middle lamella remainders are present on the surfaces of TMP, CTMP, and APMP fibres after the first-stage refining. However,

the surfaces of these fibres expose mostly the microfibrillar structure of secondary layers. Fibre surface structure appears quite heterogeneous, which indicates that the fibre separation or cell wall rupture can occur at various locations even on a fibre during refining.

After the final-stage refining and even after peroxide bleaching, the patch-like middle lamella material remains on the CTMP fibre surface. This lignin-rich material on the fibre surface will not only affect the interfibre bonding, but probably also affect the flexibility and collapsability of individual fibres. However, after final-stage refining, the patch-like middle lamella material is reduced substantially on the TMP fibre surface. Instead, a large amount of granule-like material is present on the fibre surface. These small particles are expected to be the broken pieces of the middle lamella material.

The melting of lignin at high temperature during TMP refining has also been observed. Lignin appears like a smooth film coating on the fibre surface. Some unmelted lignin particles are covered under the coating layer.

AFM and XPS analysis in conjunction with a surface peeling process have confirmed that the non-fibrillar surface material is the lignin-rich middle lamella remainder.

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