

Improving the Mechanical Properties of Ultra-Low Density Plant Fiber Composite (ULD_PFC) by Refining Treatment

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To improve the mechanical properties of ultra-low density plant fiber composite (ULD_PFC), a suitable beating process to improve the fibrillation of cellulose fibers and maintain their length was investigated. The physical properties of cellulose fibers and papers, surface chemical bonds, and internal bond strength (IB) of ULD_PFCs were analyzed. The results showed that the beating degrees, degree of fibrillation, and fiber fines increased with the decreasing of beating gap, except for the fiber weight-average length, width, kink index, and curl index. The tensile index and burst index of paper showed an increasing trend with an increase in beating degree, while the tear index showed a decreasing trend. FTIR results showed that intermolecular and intramolecular hydrogen bonds in ULDF were broken. A suitable beating gap of 30 μm with a beating degree of 35 °SR was obtained. The corresponding IB was 50.9 kPa, which represented an increase of 73.1% over fibers with a beating degree of 13 °SR.

Keywords: Beating; Ultra-low density; Mechanical properties; Microstructural characterization; Papermaking

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INTRODUCTION

Ultra-low density plant fiber composite (ULD_PFC) is an environmentally friendly material that is derived from renewable plant fibers. ULD_PFC partially solves the problems of lacking resources and pollution, as it can serve in applications such as building insulation material and packaging buffer material as a substitute for petroleum-based polymers (Trevors 2010; Xie *et al.* 2011). ULD_PFC is produced using a liquid frothing process. During this process, approximately 5 wt.% dry plant fibers, 5 wt.% chemical agents, and 90 wt.% water—the liquid medium—are mixed together. When liquid is frothed, air is introduced into the foaming system to create the interspaces (bubbles). As water is drained out, a network structure is formed by the fibers, which play a vital role in the frame material. In this process, fibers adhere through water bridging and a little starch glue without heating and pressing. The poriferous network structure can endow ULD_PFC with ultra-low densities (ranging from 10 to 90 $\text{kg}\cdot\text{m}^{-3}$) and many advantages, such as low thermal conductivity and high coefficient of sound absorption (Xie *et al.* 2004; 2008a,b; 2011). However, the ultra-low density and bonding without or a little adhesive correspond to their low mechanical properties, which limit their application. Previous studies showed that the mechanical properties of ULD_PFC are significantly improved by chemical agents, such as water, glass, silicon, and aluminum compounds (Xie *et al.* 2012; Chen *et al.* 2015a, b). However, additives absorbed on fibers through hydrogen bonds are easily removed during dehydration and drying, which wastes chemical additives and decreases the

mechanical properties of ULD_PFC. The absorption capacity of fibers is directly proportional to their surface area, which can be changed by the mechanical treatment of beating/refining. Therefore, it is important to open up the fiber surface to improve the bonding among fibers (Bhardwaj *et al.* 2004, 2007; Kamel *et al.* 2004; Mutje *et al.* 2006; Gulsoy 2014).

Mechanical beating/refining mainly causes mechanical shear by friction among fibers. It is a complex process involving many physical changes in fibers, such as cutting, external fibrosis, and curling (Hou *et al.* 2011; Gao *et al.* 2012). The strength of a handsheet is mainly affected by the quality and strength of the fibers (*i.e.*, fiber length) and the extent of bonding between the fibers (Bhardwaj *et al.* 2004). This theory may be useful for the preparation of ULD_PFC. Therefore, a suitable refining approach is essential for developing ULD_PFC with the desired mechanical properties. In the medium or high consistency refining process, friction between fibers reduces fiber cutoffs and improves the fibrillation (Gao *et al.* 2012). Theoretically, fibrillation is caused by the breakdown of fiber walls into separate lamellas and the creation and/or exposure of fibrils on fiber surfaces. As the surface area is enlarged, the adsorption capacity of the fiber surface increases and chemical components are more easily absorbed. This fiber charge affects the fiber swelling and paper/ULD_PFC properties (Bhardwaj *et al.* 2004; Molin and Daniel 2004; Marais and Wågberg 2012). Therefore, the goal of this study was to improve the mechanical properties of ULD_PFC by the refiner mechanical treatment, which will improve the bonding and absorption capacity of fibers. To clarify these issues, the microstructure of the composites was studied by scanning electron microscopy (SEM) and Fourier transform infrared (FTIR) spectroscopy. Additionally, the morphology of fibers and the mechanical properties of the paper and ULD_PFC prepared by refined pulp were tested.

EXPERIMENTAL

Materials and Pretreatment

Paperboard made of *Pinus massoniana* pulp (purchased from Qingzhou Papermaking Plant, Sanming, China) was pretreated in water (15.0 wt.%) for 120 min at ambient temperature. The board was dissociated using a beating machine (ZQS2, Northwest Institute of Light Industry Machinery Factory, Xianyang, China). After the pretreatment, the pulps and water were separated, and the pulps with 8 °SR were used for the mechanical pulping.

Aluminum sulfate and sodium silicate, as inorganic fillers, were purchased from Tianjin Fuchen Chemical Reagents Factory (Tianjin, China). Sodium dodecylbenzene sulfonate, as a foaming agent, was purchased from Jiangsu Qingting Washing Products Co., Ltd. (Yancheng, China). Chlorinated paraffins were purchased from the Changzhou Fengshuo Chemical Company, LTD (Changzhou, China).

Mechanical Pulping and Preparation of Ultra-Low Density Plant Fiber Composites

Pulps were refined in a disc refiner (ZDP-32, Jilin Paper Machinery Manufacturing Factory, Jilin, China). The preparation of the refined pulping is described in Fig. 1. Beating was processed at different beating gaps, while the beating pulp consistency (8%) and beating frequency (3 times) were constant.

Ultra-low density plant fiber composites (200 mm × 200 mm × 50 mm) were made of 55 g of dry refined *Pinus massoniana* cellulose fiber with different beating degree, with a target bulk density of 50-90 kg·m⁻³. They were manufactured separately using various parameters in a demonstration line as described in Xie *et al.* (2011) and Chen *et al.* (2015c).

Composite preparation process is also described in Fig. 1. The 500 mL Si-Al compound solution, prepared as in Chen *et al.* (2015a), was mixed with the refined fibers. The additives polyacrylamide resin, alkyl ketene dimer (AKD, water repellent), chlorinated paraffin (fire retardant), and sodium dodecylbenzene sulfonate surfactant (10% of concentration, foaming agent) were added during different manufacturing stages in all specimens, at 20 mL, 50 mL, 46 g, and 80 mL, respectively.

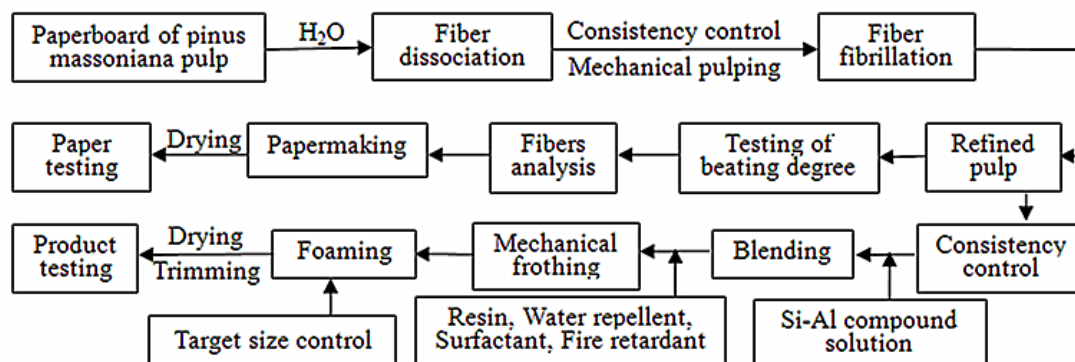


Fig. 1. The preparation of refined pulps, papers, and ultra-low density plant fiber composites

Testing of Beating Degree and Properties of *Pinus massoniana* Cellulose Fibers

The beating degree of pulps was determined by the Schopper Riegler method using a beating degree tester (Z-DZY-100, Sichuan Changjiang Papermaking Equipment Co., LTD, Yibin, China). Pulp samples of 2 g were diluted in 1000 mL of distilled water. The beating degree value was calculated by Eq. 1 according to GB/T 3332 (2004),

$$\text{Beating degree } (^{\circ}\text{SR}) = (1000 - V_{\text{water}}^*) / 10 \quad (1)$$

where V_{water} is the volume of transudatory water.

The properties of *Pinus massoniana* cellulose fibers including fiber weight-average length, fiber width, coarseness, curl index, degree of fibrillation, and fiber fines were characterized by a fiber morphology analyzer (Morfi Compact, Techpap Co., LTD, Saint Martin d'Hères, France).

Scanning Electron Microscopy (SEM)

The micromorphology and elemental distribution on the surface of specimens were characterized by scanning electron microscopy (SEM, Phenom ProX, Eindhoven, Netherlands, using an acceleration voltage of 15 kV) with an energy-dispersive spectroscopy (EDS, INCA Energy EDS for X-ray analysis, Phenom ProX, Netherlands). The surfaces of the specimens were coated with gold. The mapping was performed on an area displaying the additive and the matrix using an acceleration voltage of 15 kV.

Papermaking and Testing of their Properties

The refined pulps were dispersed in water with a standard pulp disintegrator (5 to 10 wt.%). Papers were prepared on a basis weight of $160 \text{ g} \cdot \text{m}^{-2}$. When papers were prepared, they were placed at constant temperature and humidity according to standard TAPPI T222 om-11 (2011) for 24 h. The physical properties of papers including tensile index, tear index, and burst index were tested in accordance with GB/T 12914 (2008), GB/T 455 (2002), and GB/T 454 (2002), respectively. The results reported are the average of 10 replicates.

Fourier Transform Infrared (FTIR) Spectroscopy Analysis

The functional groups of ULD_PFCs were characterized by FTIR analysis on a Nicolet 380 FTIR spectrometer (Thermo Electron Instruments, Kansas, USA) employing the KBr pellet method and taking 32 scans for each sample with a resolution of 4 cm^{-1} , ranging from 4000 to 400 cm^{-1} .

Testing of Mechanical Properties

The internal bond strength (IB) of each ULD_PFC was tested in accordance with GB/T 17657 (1999). The size of the specimens for testing IB was $50\text{ mm} \times 50\text{ mm} \times 40\text{ mm}$ ($L \times W \times H$). The results reported are the average of five replicates.

RESULTS AND DISCUSSION

Effect of Beating Gap on Beating Degree and Physical Properties of Fibers

The properties of *Pinus massoniana* cellulose fibers with various beating gaps are presented in Table 1. Fiber weight-average length was measured according to fiber weight used because it is more accurate than fiber number-average length, which is easily affected by fiber fines to reflect the fiber length (Chen *et al.* 2012). The beating degrees of pulp were increased with the decrease in beating gap, whereas the fiber weight-average length and width decreased. The fibers are just passed through the disc refiner with a big beating gap, without sufficient shear force and friction force, which limits the improvement of fiber quality (Chu and Ou 2001). As the beating gap decreased, the breaking process makes fibers swollen and softer. They were over cut and fibrillated by the growing function of shear force and friction force (Han *et al.* 2008). Therefore, the weight-average length and width of fibers decreased with increased beating gap, whereas the degree of fibrillation and fiber fines increased. Additionally, the kink index and curl index of refined pulp decreased with the decreasing of beating gap. The fiber kink index refers to the fiber cell wall damage by abrupt torsion. Fiber bending in the horizontal direction is used to express the curl index of fibers. When the bending of the swollen fibers is improved, the ratio of fiber fines is increased, whereas the deformation of fibers is reduced (Lai *et al.* 2003; Liu *et al.* 2009).

Table 1. Properties of Refined Pulp Obtained under Different Beating Gap

| Beating Gap (μm) | Beating Degree ($^{\circ}\text{SR}$) | Weight-average Length (μm) | Width (μm) | Kink Index (%) | Curl Index (%) | Degree of Fibrillation (%) | Fiber Fines (%) |
|-------------------------------|--|---|-------------------------|----------------|----------------|----------------------------|-----------------|
| 10 | 80 | 1671 | 29.3 | 32.5 | 10.2 | 1.191 | 7.58 |
| 20 | 77 | 1712 | 28.3 | 31.8 | 9.8 | 1.080 | 5.30 |
| 25 | 58 | 1878 | 30.6 | 35.2 | 10.2 | 0.901 | 5.48 |
| 30 | 35 | 2061 | 29.9 | 34.5 | 10.2 | 0.742 | 4.02 |
| 40 | 18 | 2070 | 30.7 | 40.1 | 10.6 | 0.520 | 3.58 |
| 350 | 13 | 2131 | 31.2 | 51.9 | 13.9 | 0.362 | 3.69 |

Interestingly, the beating degree of pulp with $30\text{ }\mu\text{m}$ beating gap was 94.4% larger than pulp with $40\text{ }\mu\text{m}$ beating gap, whereas the fiber weight-average length and width were not obviously changed. Under this condition, the refining process is mainly dependent on the function of friction among fibers, fiber cut off, and length (Clark 1985; Liu *et al.* 2008; Gao *et al.* 2012). Additionally, the average width of *Pinus massoniana* fiber is approximately $31.1\text{ }\mu\text{m}$. When the beating gap is less than the fiber width, the probability for fibers to suffer from shear force and friction force gets increased. Consequently, when the beating gap was less than $30\text{ }\mu\text{m}$, changes were developed in the fibers during beating.

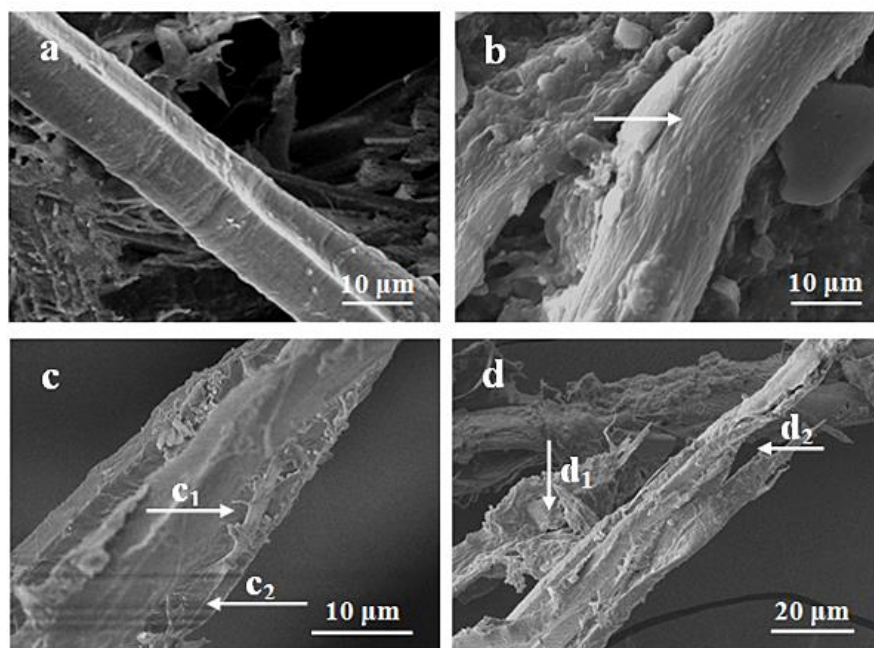


Fig. 2. SEM micrographs showing the morphology of *Pinus massoniana* cellulose fiber (a) without beating, (b) and (c) after beating with a 40 µm beating gap, (d) and after beating with a 20 µm beating gap

Figure 2 shows the *Pinus massoniana* fiber surface morphology at different beating gaps. As shown in Fig. 2a, the fiber without beating was intact, and its surface was smooth. When the beating gap was 40 µm, there was fibrillation on the fiber surface, and the fiber longitudinal direction produced splitting (Fig. 2b). The surface fibrillation made fibers loose, and the S1 layer was peeled away from the fiber wall (Fig. 2c, arrow c_1). There was slight damage to the S2 layer (Fig. 2c, arrow c_2). As the decreasing of beating gap, the shear force and friction force removed part of the outermost cell wall, leading to severe damage in the S2 layer (Fig. 2d, arrow d_1). The fibrillar structure at the end of the fiber and a fracture pattern caused by the function of shear force and friction force was apparent (Fig. 2d, arrow d_2) (Molin and Daniel 2004; Chen *et al.* 2012). Consequently, the small beating gap caused fibers to become over cut. This not only decreased the properties of fibers, but it also consumed more energy and increased the paper-making cost. To obtain good quality *Pinus massoniana* cellulose fibers and control the cost of beating, a suitable beating gap should be considered.

Effect of Beating Degree on Properties of Papers from *Pinus massoniana* Cellulose Fibers

The tensile index, tear index, and burst index of papers are affected by fiber properties. Tensile index refers to the maximum load of fracture in the vertical direction. The burst index means that the papers that can stand for the maximum stress will evenly increase on a unit area. They are mainly affected by the combination among fibers and their average length (Bhardwaj *et al.* 2004).

The energy for tearing the paper is used to express the tear index of fibers, which is mainly affected by the average length and strength of the fibers and their combination (Yuan 2004; Gao *et al.* 2012). The properties of paper made from *Pinus massoniana* cellulose fiber with different beating degree are presented in Fig. 3.

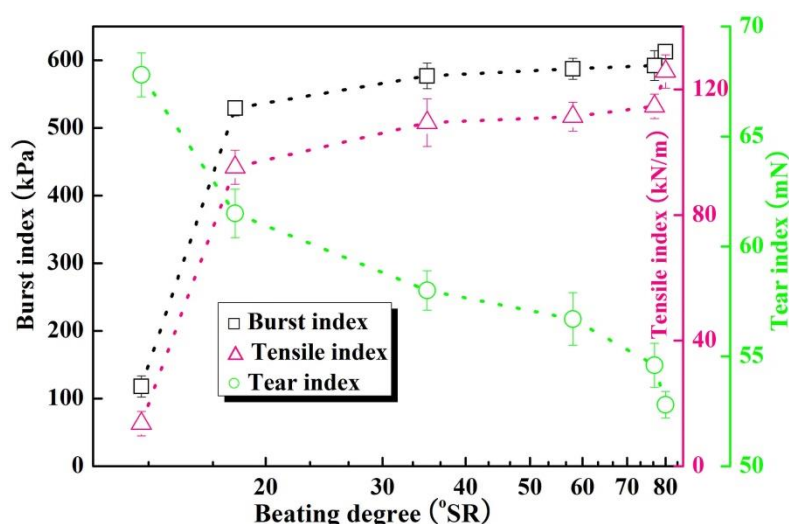


Fig. 3. Effect of various beating degree on the properties of paper from *Pinus massoniana* cellulose fiber

As shown in Fig. 3, the tensile index and burst index of paper increased with an increase in beating degree, while the tear index decreased. As the fiber surface is fibrillated during beating, more hydroxyl groups are fractionated, resulting in increased bonding ability of the fiber. Beating also produces many tiny fibers in the pulp, which fill in the space gaps among the long fibers, thus increasing the number of hydrogen bonds and improving fiber combination. The tiny fibers play a bridging role in the paper and contribute to its network structure (Zhang *et al.* 2011; Chen *et al.* 2012). However, the tear index is mainly attributed to the strength and coarseness of the fibers. When the beating degrees of the pulp continue to increase, the ratio of tiny fibers and small components was sharply increased, and the fiber weight-average length was reduced. The decreasing of single-fiber strength leads to decreasing tear index (Wei *et al.* 2010). Consequently, to obtain good quality papers, a suitable beating degree of pulp should be considered.

Chemical Bonding of Ultra-Low Density *Pinus massoniana* Cellulose Fiber Composite

The FTIR profiles of ultra-low density *Pinus massoniana* cellulose fiber composite with different beating degrees are presented in Fig. 4. The control specimen was the composite made of cellulose fiber at 18°SR beating degree without Si-Al compounds. The infrared spectra of composites exhibited a strong -OH peak at around 3450 cm^{-1} and 2920 cm^{-1} , which reflected the presence of intramolecular and intermolecular hydroxyls groups, respectively (Alemdar and Sain 2008). Compared to the control specimen, two peaks become weaker. Those parts of hydroxyl are consumed by the hydroxyls of Si-Al compound (Chen *et al.* 2015c). Due to the breaking of intramolecular and intermolecular hydrogen bonds, the two peaks weakened gradually with increased beating degree, as shown in Fig. 5 (Lu *et al.* 2005). The absorption band at 1630 cm^{-1} corresponds to the -OH bending vibration from absorbed water. This peak strengthened with increased beating degree, which is caused by the addition of aluminum sulfate attaching to crystal water and the hydrophilic sodium silicate. The peaks at 1088, 1050, 881, and 667 cm^{-1} were the characteristic peaks of cellulose. Among these peaks, the peak at 881 cm^{-1} , attributed to the β -1,4-glycosidic linkage vibrations, was obviously weakened when the Si-Al compounds were added in composites, as the Si-Al compound solution is a kind of acid with the pH value of 3.8. When they are added in the preparation process of ULD_PFC, the β -1,4-Glycosidic linkage of cellulose is easily broken during drying in the oven.

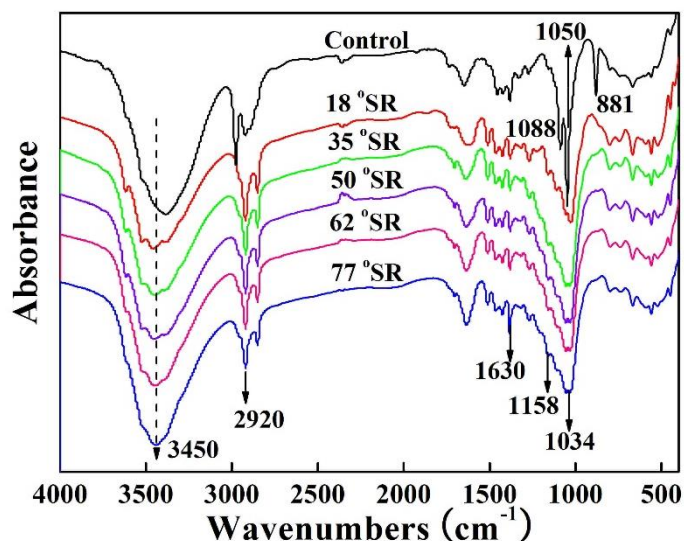


Fig. 4. FTIR profiles of ultra-low density *Pinus massoniana* cellulose fiber composite with different beating degrees

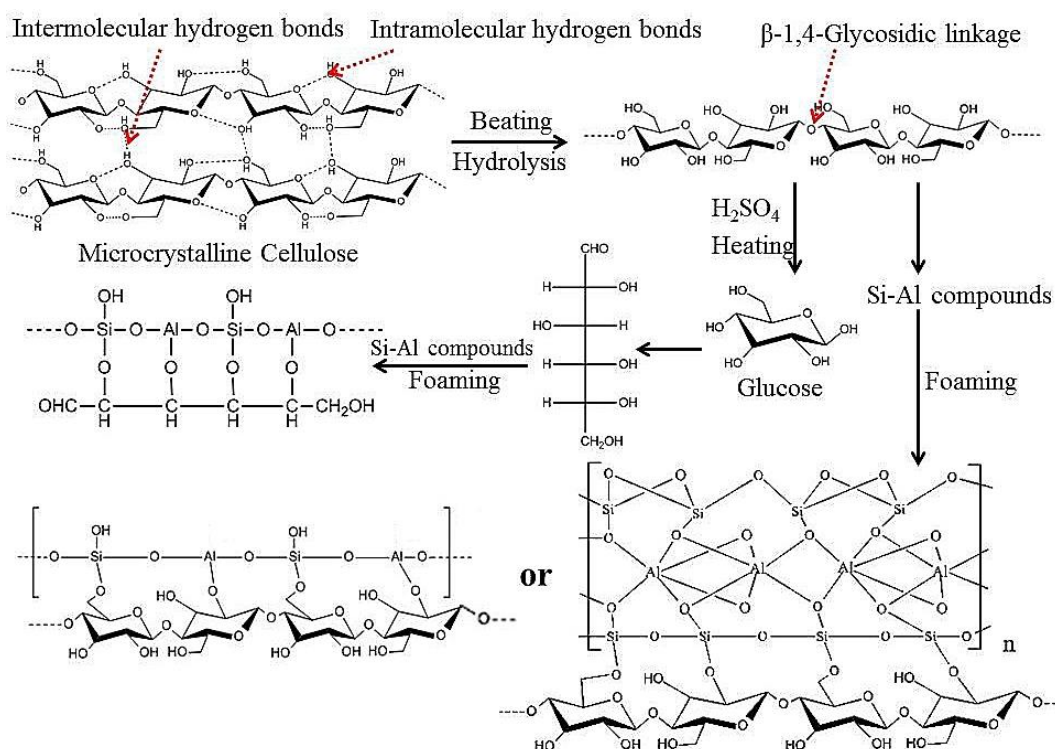


Fig. 5. The reaction between cellulose fiber and Si-Al compounds

Compared to the control, the additional broad peak at around 1158 and 1034 cm^{-1} , which may be attributed to Si-O-C and Al-O-C bonds, was present in the infrared spectra of ULD_PFCs (He *et al.* 2014; Chen *et al.* 2015c, d, 2016). The cellulose units expose a number of hydroxyl groups and bonding sites, allowing the additives with hydroxyl groups to form a large amount of hydrogen bonds. Therefore, the covalent bonds of Si-O-C are mainly formed from the reaction between Si-Al compounds and fibers with the function of dehydration condensation, which would improve the mechanical properties of ULD_PFCs by enhancing the interconnection between Si-Al compounds and fibers (Chen *et al.* 2012). The corresponding reaction is diagrammed in Fig. 5.

Effect of Beating on Internal Bond Strength of Ultra-Low Density Plant Fibers Composite

As shown in Fig. 6, the IB of ULD_PFCs increased first and then decreased with the increasing of beating degree on *Pinus massoniana* cellulose fiber. Especially, the IB of ULD_PFCs with 35 °SR beating degree (50.9 kPa) was 73.1% larger than the 13 °SR beating degree (29.4 kPa). Due to the fact that fibers in ULD_PFCs are mainly linked through hydrogen bonding, the intrinsic properties of fibers (*i.e.*, fiber length) and the strength of fiber combination are important. So, the IB of ULD_PFCs may be the same as tear index of paper, the decreasing of single-fiber strength leads to decreasing IB, when the beating degrees of the pulp continue to increase.

The microstructure of ULD_PFC is presented in Fig. 7a. When the beating degree was less than 40 °SR, the fiber surface was fibrillated, and fiber length was maintained with the increasing of beating degree. The fibrillation of cellulose fibers was further increased in a beating degree more than 40 °SR, but the fiber length was shortened because of the overcut. For example, the weight-average length of fibers with 35 °SR (2061 μm) and 80 °SR (1671 μm) beating degree were 3.4% and 27.5% smaller than the fibers with 13 °SR beating degree (2131 μm), respectively.

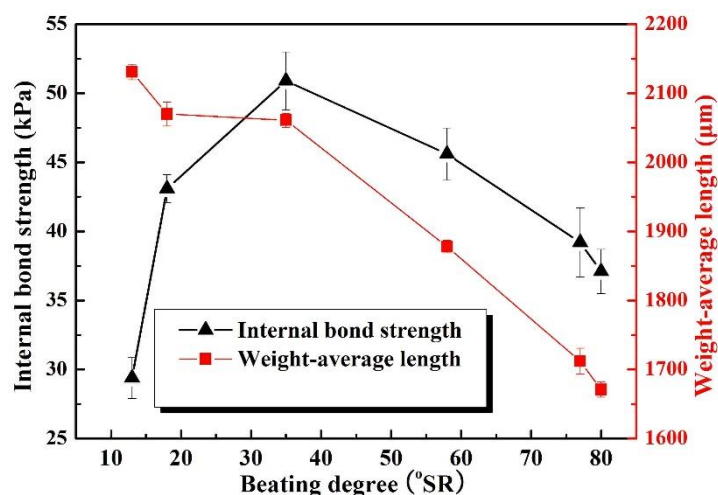


Fig. 6. Effect of various beating degree on the weight-average length of *Pinus massoniana* cellulose fiber and internal bond strength of ultra-low density *Pinus massoniana* cellulose fiber composites

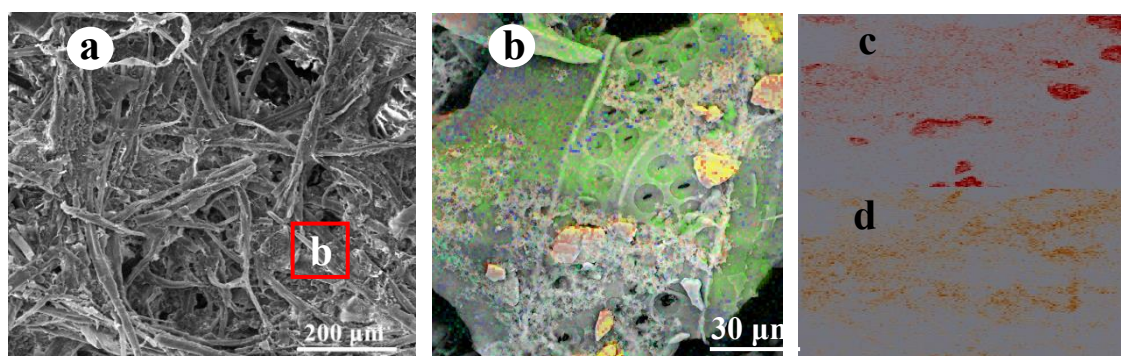


Fig. 7. (a) SEM micrographs of ULD_PFCs with the beating degree 35 °SR. (b) Element mappings of ULD_PFCs from a, (c) and (d) stands for the aluminum and silicon element mapping from b.

Additionally, the Si-Al compounds with a number of hydroxyl groups were added in ULD_PFCs to improve their mechanical properties. They remained in ULD_PFCs through absorbing on the surface of fibers. Therefore, the bonding ability of fiber is also

important for improving the mechanical properties of ULD_PFCs. During beating, fiber is torn under the function of mechanical force and friction force, and the cell walls are broken, resulting in fibrillation. As the specific surface area is enlarged, the adsorption capacity of fiber increases; and chemical components become easier to absorb on fibers (Bhardwaj *et al.* 2004; Molin and Daniel 2004; Marais and Wågberg 2012). As shown in Fig. 7b, c, and d, the distributions of Si and Al elements on the surface of fibers were relatively spread out and even, but they were easy to agglomerate on the fibrillated surface of ULD_PFCs fibers (Fig. 8-arrow). So, the compactness of ULDFs' surfaces, which will be beneficial to their mechanical properties, is growing higher with the increasing of beating degree (Fig. 8), indicating that the absorption of Si-Al compounds on the fibrillated surface of fibers increased. Consequently, a suitable beating process that improves the fibrillation of cellulose fibers and maintains their length is important to obtain good mechanical properties of ULD_PFCs.

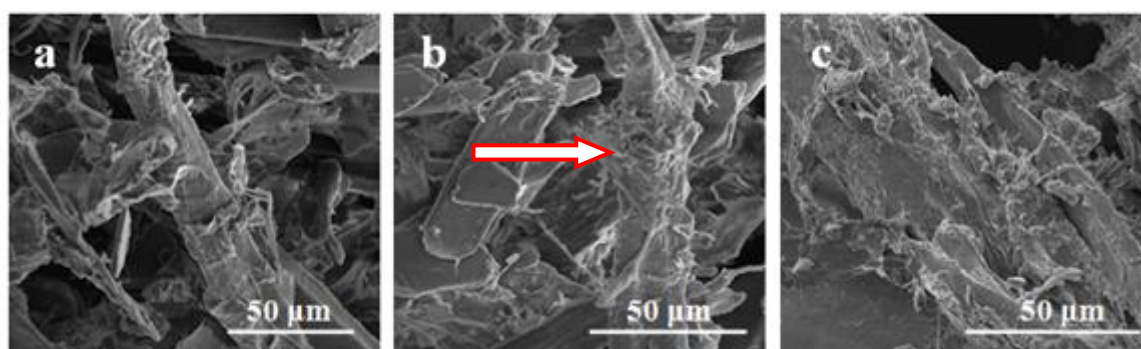


Fig. 8. SEM images of the ultra-low density *Pinus massoniana* cellulose fiber composites with different beating degree: (a) 18 °SR, (b) 35 °SR, and (c) 77 °SR

CONCLUSIONS

1. After refining, the physical properties of *Pinus massoniana* cellulose fiber are changed. When the beating gap decreases, the beating degrees, degree of fibrillation, and fiber fines increase, while the fiber weight-average length, width, kink index, and curl index decrease. When the beating gap is 30 µm, the beating degree is 169.2% larger than the beating gap of 350 µm, while its weight-average length is only decreased by 3.4%.
2. The tensile index and burst index of paper shows an increasing trend with an increase in beating degree, while the tear index decreases. At a beating gap of 30 µm, the tensile index (109.4 kN/m), burst index (577.0 kPa), and tear index (58.0 mN) recorded optimum values. The chemical bonding results show that there were no drastic changes in the functional groups of cellulose fiber during refining, except for the breaking of intermolecular hydrogen bonds. However, the intramolecular hydrogen bonds are broken, and the covalent bonds of Si-O-C and Al-O-C are formed in ULD_PFCs.
3. The internal bond strength (IB) of ULD_PFCs shows subsequent increase and decrease when the beating degree of *Pinus massoniana* cellulose fiber continuously increases. Consequently, a suitable beating gap of 30 µm with a beating degree of 35 °SR is obtained in this study. The corresponding IB is 50.9 kPa, which increased by 73.1% over the composite with a beating degree of 13 °SR.

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