

Modification of Ultra-Low Density Fiberboards by an Inorganic Film Formed by Si-Al Deposition and their Mechanical Properties

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To improve mechanical properties of ultra-low density fiberboards (ULDFs), Si-Al compounds were mixed together with fibers during preparation of ULDFs, forming a thin film on the surface of the fibers via hydrogen bonding. This work mainly optimized two proposed methods in which the inorganic thin film was assembled on the surface of fibers, in terms of its effect on the mechanical properties of fibers. Microstructural characterization (such as micromorphology and elemental distribution, chemical bonding, and crystalline phase) of Si-Al compounds and ULDFs was done to evaluate the effects. The results revealed that an inorganic thin film (probably $\text{Al}_2\text{O}_3\text{-SiO}_2$) covered the surface of the fibers. Compared with the control specimen, the modulus of elasticity, modulus of rupture, and internal bond strength of the specimen treated by the sol-gel process increased from 3.87 MPa to 13.19 MPa, 0.05 MPa to 0.16 MPa, and 0.010 MPa to 0.025 MPa, respectively. Based on its higher mechanical properties, a combined sol-gel method was judged to be better for enhancement of fibers than a separate deposition method.

Keywords: Ultra-low density fiberboards; Inorganic thin film; Mechanical properties; Microstructural characterization

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INTRODUCTION

The lack of resources and the problems of pollution have led researchers to focus on environmentally compatible materials derived from renewable sources that can serve in some applications as substitutes for petroleum-based polymers (Pimentel 2003; Trevors 2010; Li *et al.* 2011). Plant fiber is a satisfactory raw material that is renewable, biodegradable, and has low density and high stiffness and strength. It can be applied as a reinforcement ingredient to improve the mechanical properties of some composites (Majid *et al.* 2008). It can be also used as raw material to manufacture the ultra-low density fiberboards (ULDFs), building insulation material, and packaging buffer material (Xie *et al.* 2004, 2008a,b; Niu *et al.* 2014). This ensures that ULDF can be a competitive material with respect to environmental protection.

Ultra-low density fiberboard is manufactured by a liquid frothing principle. Water serves as the liquid medium role during the chemical reaction and in the foaming system. The fibers themselves, as the frame material, adhere through a charged adhesive and build a network structure with many air voids through the use of a surfactant. This process leads to a porous product that has good physical properties. No heating and pressing or common adhesives such as phenol-formaldehyde resin (PF) or urea-formaldehyde resin (UF) are introduced in the preparation process (Xie *et al.* 2011; Niu *et al.* 2014). Ultimately, this is good for both the cost and human health.

Plant fibers (max 85%wt in ULDF) can endow ULDF with ultra-low densities of

10 to 90 kg/m³, low thermal conductivity (e.g. 0.035), and high coefficient of sound absorption (e.g. 0.67) (Niu *et al.* 2014). However, ultra-low density corresponds with low mechanical properties, which limits the application of ULDF as a candidate for building materials and packaging materials. Therefore, it becomes important to improve the mechanical properties of the ULDF. When inorganic compounds such as SiO₂ (Saka and Ueno 1997; Fu *et al.* 2011), TiO₂ (Mahr *et al.* 2012), and Si-Al compounds (Xie and Liu 2012; Lin *et al.* 2013; Niu *et al.* 2014) were used as fillers and mixed with fibers, the mechanical properties of fibers and fiber-based composites were significantly improved. A sol-gel with semi-solid properties is formed when Si and Al compounds react with water. The inorganic material interacts with plant fibers after mechanical stirring and generates a thin inorganic film on the surface of the fibers. The inorganic film has high barrier properties on a wide range of substrates, including plant fibers. Some researchers have concluded that a film deposited on the matrix effectively enhances both the fire resistance and the decay resistance of plant fibers (Hofman-Züter *et al.* 1997; Jin *et al.* 2011; Liu *et al.* 2011; Niu *et al.* 2014).

Tshabalala and Li (2007) and Mishra and Luyt (2008) indicated that inorganic compounds not only were distributed around plant fibers, but also occupied holes constructed by the fibers, increasing the density and brittleness of the resulting material. It is known that the amount of inorganic substances in the matrix-filler interface and the interaction between the organic and inorganic substances play critical roles in ensuring improvements in the physical and mechanical properties. In addition, the formation method of the inorganic film also has an important effect on the properties of the material. So the question arises, which one is more efficient to improve mechanical properties of ULDF: separate addition or combined addition of sodium silicate and aluminum sulfate during manufacture of ULDFs?

Little literature concerning these issues has been published. Therefore, the goal of this study is to clarify these issues by measuring the microstructure of the material using scanning electron microscopy with an energy-dispersive X-ray detector (SEM-EDX), Fourier transform infrared (FTIR) spectroscopy, and X-ray diffraction (XRD) to determine the effect of the microstructure of the material on its mechanical properties. This will allow optimization of additional amounts of Si-Al compounds and explain the interaction mechanism between the plant fiber and Si-Al deposition.

EXPERIMENTAL

Materials

Kraft pulp (KP, spruce-pine-fir; Tembec Inc., Canada) with a beating degree of 34 °SR was utilized as a raw material to manufacture an ultra-low density fiberboard. Aluminum sulfate and sodium silicate, purchased from Tianjin Fuchen Chemical Reagents Factory (China) were used to generate the Si-Al depositions.

Methods

Preparation of Si-Al compound solution

The Si-Al compound solution was produced by a reaction between sodium silicate (2.5 wt%) and aluminum sulfate (3.0 wt%) in an aqueous solution at 80 °C. The aluminum sulfate solution (3.0 wt%, 100 mL) was first added to a 500-mL tri-neck round-bottom flask with a magnetic stir bar and heated at 80 °C for 10 min with vigorous stirring. The sodium silicate solution (2.5 wt%, 35 mL) was slowly added to the flask and mixed with aluminum sulfate to obtain the Si-Al compound solution. Finally, the Si-Al compound solution was cooled to room temperature with gentle stirring.

Manufacture of ultra-low density fiberboard

Ultra-low density fiberboards of 200 mm × 200 mm × 50 mm ($L \times W \times H$) were manufactured separately using various parameters in a demonstration line as described by Xie *et al.* (2011), with a target bulk density of 55 kg/m³. This demonstration line consists of a fiber dissociator, a refiner, a foaming tank with blending vanes, a foaming mould, a 20-m³ dry kiln, and a band-saw. The preparation process of the specimens is described in Fig. 1. Aluminum sulfate and sodium silicate were added during the preparation process by two methods: each separately (MT1) or combined as a sol-gel (MT2).

The additives polyacrylamide resin, alkyl ketene dimer water repellent (AKD), chlorinated paraffin fire retardant, and sodium dodecylbenzene sulfonate surfactant (foaming agent) were added during different manufacturing stages, at 23.8%, 8.4%, 9.5%, and 3.1% of dry fiber weight, respectively.

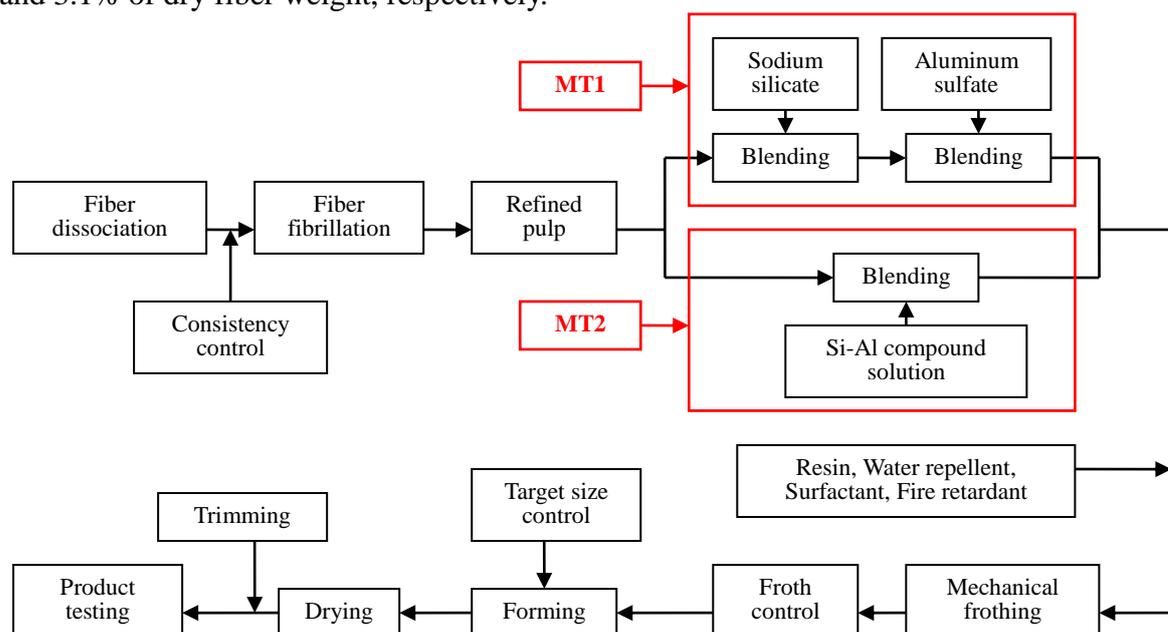


Fig. 1. The preparation process of ultra-low density fiberboard

Microstructural characterization

The micromorphology and elemental distribution on the surface of fiber specimens were characterized by SEM-EDX (Philips XL-30 TMP, The Netherlands) at an acceleration voltage of 5 kV or 20 kV. The surfaces of the specimens were coated with gold by an electro-deposition method to confer electrical conduction and reduce specimen charging before recording the SEM micrographs. EDX profiles were generated by scanning a subarea of the SEM images to quantify the chemical elements present within the subarea.

To discuss the reaction between Si-Al compounds and ULDFs, the FTIR spectrum of each specimen was tested. The FTIR analysis was performed by means of a Nicolet 380 FTIR spectrometer (Thermo Electron Instruments, USA), employing the KBr pellet method, taking 32 scans for each sample with a resolution of 4 cm⁻¹, ranging from 400 cm⁻¹ to 4000 cm⁻¹.

To evaluate the crystalline phase of ULDFs, the crystallization of each ULDF surface was determined by X-ray diffraction (XRD; X'Pert PRO MPD, Philips-FEI, Netherlands), and its patterns were recorded using Co K α radiation ($\lambda = 1.78901$ nm) with a 5-mm variable divergence slit between 4.9 ° and 65.0 ° (2θ), at a step size of 0.0129 ° and a speed of 0.133 °/s.

Testing of mechanical properties

The modulus of elasticity (MOE), modulus of rupture (MOR), and internal bond strength of each ULDF were tested in accordance with GB/T 17657 (1999). The size of the specimens for the testing of MOE and MOR was 120 mm × 50 mm × 10 mm ($L \times W \times H$). The size of the specimens for testing internal bond strength was 50 mm × 50 mm × 40 mm ($L \times W \times H$). The results were the average of five replicates.

RESULTS AND DISCUSSION

Micromorphology and Elemental Distribution of ULDFs

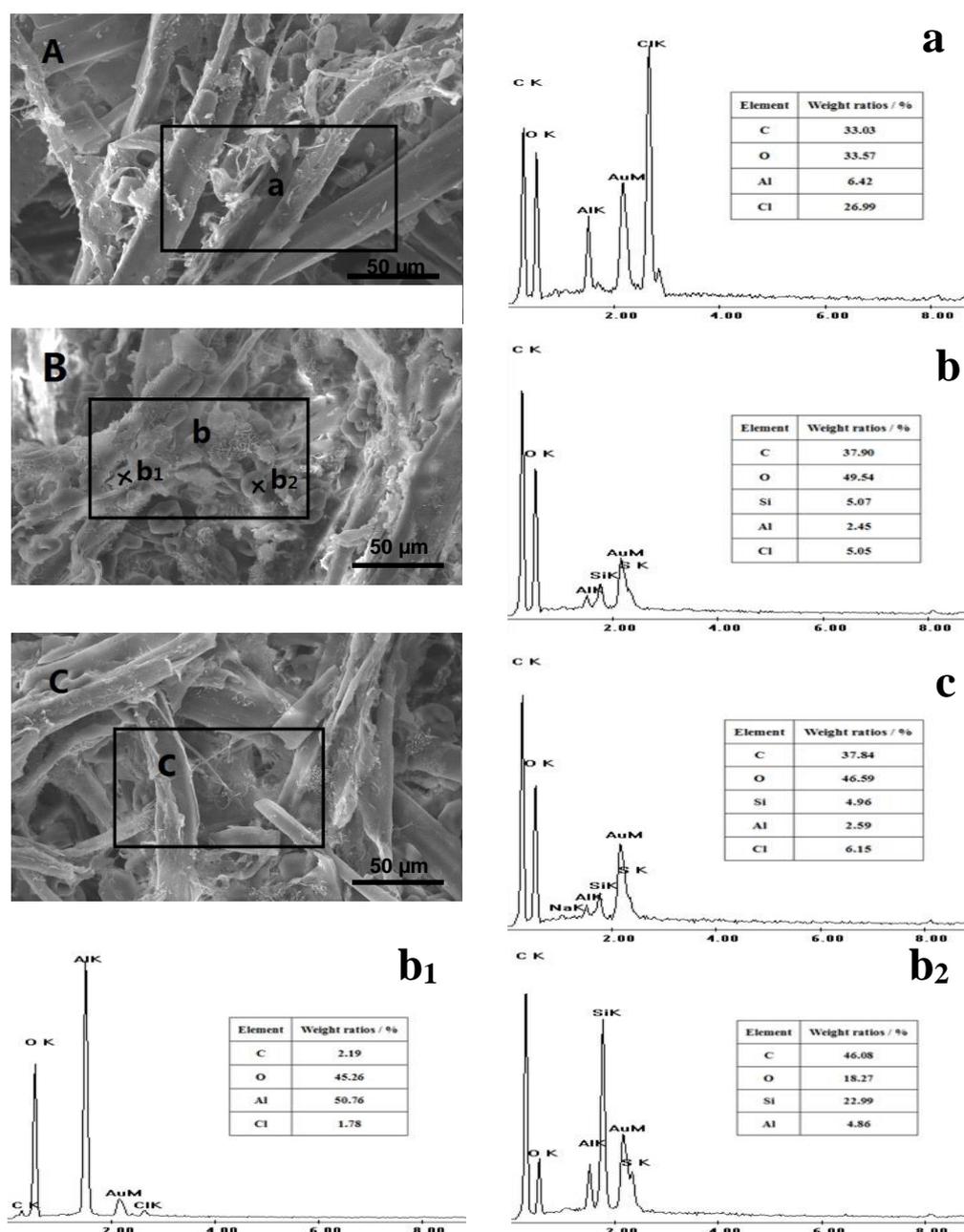


Fig. 2. SEM (left) and EDX profiles (right) of the specimens. (A) Control specimen without Si-Al deposition; profile (a), the result of EDX scan area a, shows the distribution and weight ratios of the elements; (B) specimen with MT1; profiles (b), (b₁), and (b₂) are results of EDX spot scans; and (C) specimen with MT2; profile (c) is the result of EDX scan area c.

The control specimen shown in Fig. 2A exhibited a typically smooth surface. As shown in the EDX spectrum of the control specimen (Fig. 2a), the weight ratios of C, O, Cl, and Al were 33.03%, 33.57%, 26.99%, and 6.42%, respectively. The content of Al element was presented in the EDX spectrum, but Si element was not. This was because chlorinated paraffin containing aluminum was added in the preparation process to improve the fire resistance of ULDF. Compared to the control specimen, the SEM and EDX for the specimens MT1 (Fig. 2b) and MT2 (Fig. 2c) presented different results. There were differences almost equivalent to the weight ratio of C, O, Si, Al, and Cl when comparing MT1 and MT2. Especially, the content of aluminum in Fig. 2 (a)-EDX was higher than those of specimens shown in Fig. 2 (b) and (c). This was because the specimens with MT1 and MT2 were covered by a thin inorganic film consisting of Si and Al. The result was consistent with the content of chlorine in Fig. 2 (b) and (c)-EDX, which was also lower than that of specimen shown in Fig. 2 (a). On the other hand, the weight ratios of C and Cl elements were only 2.19% and 1.78%, respectively, but the weight ratios of O and Al elements reached up to 45.26% and 50.76% (Fig. 2b₁). This indicates the presence of Al₂O₃. Compared to Fig. 2b₁, Fig. 2b₂ presents a different result: the weight ratios of Si (22.99%) elements were more than Al (4.86%). That means that one of the constituents of agglomeration (b₂) is SiO₂. We can thus deduce that the thin inorganic film that covered the fiber surface may consist of aluminum-silicon oxides (Al₂O₃-SiO₂) or their hydroxide. The weight ratio of Au was present in each EDX profile because samples were coated with gold when tested by SEM-EDX.

The SEM image of the specimen in which the ULDF was coated with inorganic materials separately (Fig. 2B) shows that there was much agglomeration of the particles with orbicular or nubby objects, which indicated that the dispersion was not completely even. In Fig. 2C, there was almost no agglomeration on the fibers' surface. This suggested that the addition method affects the formation of the inorganic film on fiber surface. For example, there was a uniform distribution on the surface with MT2, which might affect the mechanical properties of ULDF. From above, it can be seen that the combined sol-gel-treated specimen had a better homogeneous dispersion of the inorganic filler on the surface.

Chemical Bonding of Si-Al Compounds and ULDFs

In order to discuss the reaction between Si-Al compounds and ULDFs, the FTIR spectra of Si-Al compounds, the control fiberboard, MT1-coated fiberboard, and MT2-coated fiberboard were carried out, and results are shown in Fig. 3.

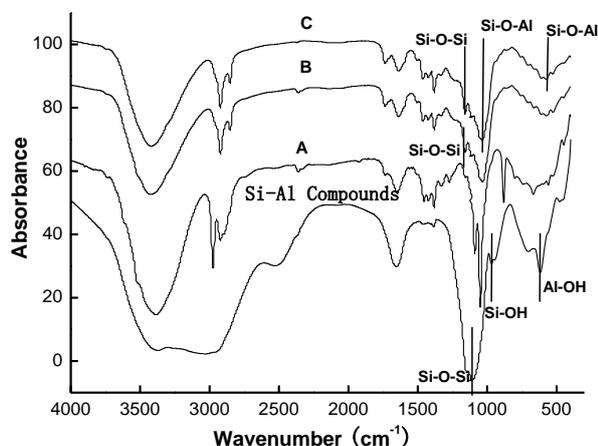


Fig. 3. FTIR profiles of the Si-Al compounds, (A) control fiberboard, (B) MT1-coated fiberboard, and (C) MT2-coated fiberboard

The Si-Al compounds showed a band at 3432 cm^{-1} , which was attributed to the -OH stretching line. The peaks at 1108 , 967 , and 617 cm^{-1} were attributed to the Si-O-Si, Si-OH, and Al-OH vibrations, respectively. But the peaks of Si-OH and Al-OH were disappearing while the Si-Al compound was added in fiberboard, because the reaction was occurring between the Si-Al compounds and fibers. The FTIR spectrum of the control specimens (Figs. 3A) showed characteristic peaks at 1454 cm^{-1} (C-H bending of CH_2 groups), 1381 cm^{-1} (C-H bending of CH_3 groups), and 2921 cm^{-1} (CH_2 stretch). The peaks at 1650 and 1272 cm^{-1} were the characteristic peaks of lignin, and the peaks at 1088 , 881 , and 667 cm^{-1} were the characteristic peaks of cellulose. The FTIR spectrum of Figs. 3B and 3C show most of the characteristic peaks of the control specimens (Fig. 3A). The additional broad peaks that were not found in the spectrum of the control specimens at 1158 , 1034 , and 575 cm^{-1} may be attributed to Si-O-Si, Si-O-C, and Si-O-Al bonds, respectively, which can explain why covalent bonds were formed. Among these covalent bonds, Si-O-C may be formed from the reaction between Si-Al compounds and fibers or the organic fillers. When fibers react with Si-Al compounds, the mechanical properties of ULDF enhance the interconnection between the fibers. Just as above, the mechanical properties were also enhanced by the inorganic films that were formed between the Si-Al compounds and organic fillers with Si-O-C. The covalent bonds contribute to the mechanical properties of materials, which indicated that Si-O-C might play an important role in the mechanical properties of ULDF. On the other hand, the FTIR spectrum of Fig. 3B was similar to Fig. 3C, which meant that the different additive methods did not influence the reaction between inorganic fillers and fibers.

The results of EDX and infrared spectra of specimens MT1 and MT2 indicate that the fiber surfaces were covered with a thin inorganic film whose chemical composition may consist of aluminum-silicon oxides ($\text{Al}_2\text{O}_3\text{-SiO}_2$), which appears to be consistent with the surface characteristics analysis attached to hydroxide groups. And the result indicated that the fibers were partly covalently bound to Si-Al compounds.

Crystalline Phase of ULDFs with and without Si-Al Depositions

As presented in Fig. 4A, the diffraction peak at about $2\theta = 18^\circ$ corresponds to the (101) plane of the fiber crystalline structure in the control specimens. In addition, the diffraction peaks located at $2\theta = 26.46^\circ$ and 40.64° can also be perfectly indexed to the (002) and (040) crystal faces of the fiber, respectively. The results showed that the fiber, which contains a large number of polar groups (the hydroxyl groups in its side chain), is easy to crystallize.

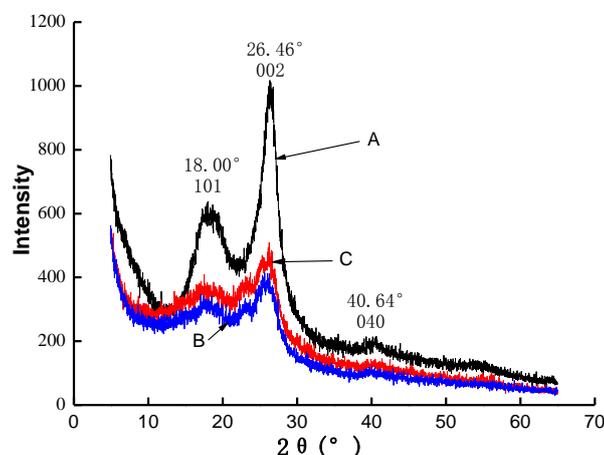


Fig. 4. XRD patterns of the (A) control fiberboard, (B) MT1-coated fiberboard, and (C) MT2-coated fiberboard

However, the intensity of the peaks shifted and decreased for specimens MT1 and MT2. The curves in Figs. 4B and 4C revealed that the crystallinity of the fiber was largely influenced by the inorganic film; the extent of crystallinity was limited, due to a substantial proportion of amorphous SiO₂ networks (Shao *et al.* 2003). The amount of hydroxyl groups were decreased when the sodium silicate and aluminum sulfate which could link with fibers by Si-O-C and Al-O-C bonds were added. Apparently, a thin inorganic film (Al₂O₃-SiO₂) was deposited on the fiber surface of specimens MT1 and MT2, consistent with the SEM-EDX and FTIR analysis.

It is also important to note that the intensity of the peaks in Fig. 4C was stronger than those in Fig. 4B. Comparing Fig. 4C to Fig. 4B, it is clear that specimen MT2 had a thinner inorganic film than MT1. The sodium silicate and aluminum sulfate can be added uniformly on the fiber surface through the combined method. The results were in agreement with the SEM analysis.

Mechanical Properties of ULDFs

The mechanical properties of the ULDF specimens are shown in Fig. 5. The bulk densities of the specimens corresponding to A, B, and C were 47.1, 50.3, and 55.1 kg/m³, respectively.

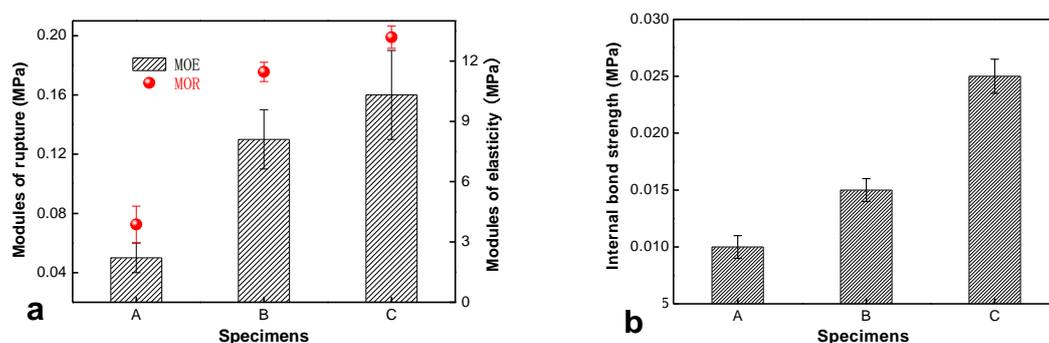


Fig. 5. (a) MOE and MOR and (b) internal bond strength of (A) control fiberboard, (B) MT1-coated fiberboard, and (C) MT2-coated fiberboard

The MOE (11.46 MPa) and MOR (0.13 MPa) of specimen MT1 were higher than the control specimens, whose MOE and MOR were 3.87 and 0.05 MPa, respectively. At the same time, the internal bond strength also improved, from 0.010 to 0.015 MPa. It can be concluded that the inorganic filler plays a critical role in improving the mechanical properties of the material. The reason for this enhancement is the effect of the Si-Al compounds. The hydroxyl ion and aluminum sulfate can form a typical polyol complex, which has remarkable absorption capacity for the fibers with hydrogen bonds. The mechanical properties of the ULDF can be enhanced by improving the interconnection between the fibers when they react with the Si-Al compounds and form the covalent bonds of Si-O-C between the Si-Al compounds and organic fillers. These effects were also evident in the FTIR and XRD analysis.

The MOE (13.19 MPa) and MOR (0.16 MPa) of specimen MT2 were higher than those of specimen MT1. They only increased by 10.2% and 23.1% respectively, so their variation was small. On the contrary, the internal bond strength of specimen MT2 (0.025 MPa) was 0.01 MPa more than the specimen MT1. In combination with the SEM and XRD analysis, this result can be attributed to the fact that there was better homogeneous dispersion of the inorganic filler on the combined method-treated specimen surface. The results confirmed that the mechanical properties of ULDF were enhanced with sodium silicate and aluminum sulfate, especially by the combined method as a sol-gel process.

Micromorphology of Inorganic Film Covering the Surface of Single Fiber

According to Fig. 6a, the cross-sectional surface of the fibers was not found to be rough. Although it was covered by a thin resin film, the grain of wood still could be seen. It was indicated that there was not an inorganic thin film on the surface of the control specimen. In contrast, the specimen MT2 appears to have a thin inorganic film on the surface (Fig. 6b). It is also important to note that the cross-sections of these specimens reveal some particles with platelike shapes (Fig. 6b). Comparing Figs. 6a to 6b, it can be deduced that these platelike shapes may be formed by the Si-Al compound solution. The inorganic films were separated from the fibers when they were cut off (Fig. 6b). The result indicated that the interface between the fibers and Si-Al compounds was not perfect.

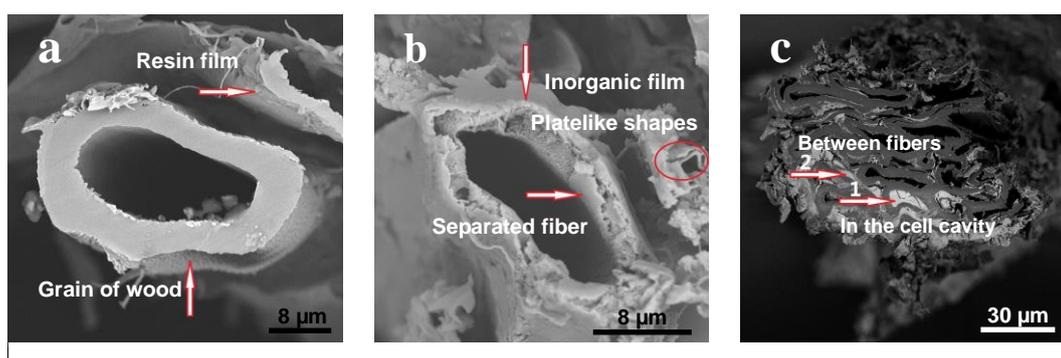


Fig. 6. SEM images of fiber cross-sections. (a) individual control fibers, (b) individual MT2-treated fibers, and (c) a bunch of MT2-treated fibers.

One of the most common causes of pore film adhesion is that the thin inorganic film was just deposited on the fibers' surface with a hydrogen bond, but did not react to the fiber with a covalent bond. The other contributing cause is that the inorganic fillers were not uniformly distributed on the fibers' surface, which caused an uneven coating when the specimens were cut off.

As seen in Fig. 6c, there was some deposition between the fibers, which could affect the mechanical properties of composites (Fig. 6c, arrow 2). At the same time, the fillers were also found in the cell cavity (see Fig. 6c, arrow 1). Because the fiber is quite porous, the particles of Si-Al compounds can enter the cell cavity through the interspaces. Therefore, the inorganic nano-fillers had two functions in enhancing the mechanical properties of ULDF. One is that the Si-Al compound solution deposits on the fiber surface when the inorganic coating is formed. As the reaction proceeds, Si-O-C covalently bonds and may form hydrogen bonds with the fiber surface -OH groups between the inorganic coating and fiber surface. The other function is that the Si-Al solution enters the cell cavity through the fiber pits (Xie *et al.* 2011) and then becomes deposited. After the reaction, the inorganic nano-filler enhances the mechanical properties of the ULDF because of its filling effect.

CONCLUSIONS

1. Deposition of inorganic films resulting in more homogeneous distributions on the surface of the fibers through a combined sol-gel process (MT2) in comparison to when they were added separately (MT1). As seen by the microstructure of ULDF, an inorganic thin film was deposited on the surface of the fiber, probably consisting of aluminum-silicon oxides ($\text{Al}_2\text{O}_3\text{-SiO}_2$).

2. After ULDFs were modified by MT1 and MT2, their mechanical properties were well improved; MT2 was better than MT1 for improving the mechanical properties of ULDF, especially for internal bond strength. Compared with the control specimen, MOE, MOR, and internal bond strength of specimen MT2 were increased by 240.8%, 220.0%, and 150.0%, respectively.

ACKNOWLEDGEMENTS

The authors are grateful for the financial support of the National Science and Technology Support Program (2008BADA9B01) and the National Natural Science Foundation of China (NSFC) (30781982).

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Article submitted: June 19, 2014; Peer review completed: October 25, 2014; Revised version received and accepted: November 9, 2014; Published: November 26, 2014.