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STUDY ON MIXED BIOMASS BINDERLESS COMPOSITE BASED ON SIMULATED WOOD

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ABSTRACT

This article describes techniques used to study mixed biomass fiberboards based on the simulation of wood composition, in which high-strength bamboo could serve as wood cellulose, low-density poplar could serve as wood hemicelluloses, and lignin-rich walnut shells could serve as wood lignin. The effects of different material mass ratios on board properties were discussed, and the bonding mechanisms of the mixed binderless composites were analyzed through Fourier transform infrared spectroscopy (FTIR) and environmental scanning electron microscopy (ESEM). Experimental results showed that the simulation could identify bio-fiberboard compositions with good performance.

KEYWORDS: Steam explosion technology, hybrid biomass composite, simulated wood, Fourier transform infrared spectroscopy, environmental scanning electron microscopy.

INTRODUCTION

The application of natural fibers for reinforcement in composite materials has increased due to environmental concerns, low cost, degradability, and health concerns (Ahmad et al. 2018, Russ et al. 2016). Steam explosion technology for binderless bonding mainly uses water vapor under high temperature and high pressure to treat raw fiber material, realizing the component separation and structural change of the material through instantaneous pressure release (Mosier et al. 2005, Agrupis et al. 2000). The steam explosion method could separate fiber into gossypine monomer fibers and fiber bundles, thus enhancing the plasticity of fiber, which could greatly improve the water-resistance and dimensional stability properties of fiberboard. Hemicelluloses-degraded sugar along with activated lignin during steam explosion could produce an adhesive substance to bind fiber together (Sørensen et al. 2008, Okuda et al. 2006, Laemsak and Okuma

2000).

Laemsak and Okuma (2000) adopted the steam explosion method to process oil palm leaves to manufacture binderless fiberboard. Velásquez et al. (2002) studied steam-exploded Miscanthus sinensis binderless fiberboard. Quintana et al. (2009) studied steam-exploded banana bunches. Mancera et al. (2012) incorporated treated lignin into steam-exploded binderless fiberboard made from agricultural waste. A study by Meenakshi and Krishnamoorthy (2019) indicated that the surface treatment improves the performance of natural fiber in hybrid fiber-reinforced composites.

Lu et al. (1997) studied the high-temperature, high-pressure exploding treatment of I-72 poplar to manufacture binderless particle board. They found that binderless particle board exhibited better performance under an exploding pressure of 3.0 MPa for one minute. However, the modulus of rupture (MOR) was 17.7 MPa, and the internal bonding strength (IB) was 0.183 MPa in boiling water for 2 h.

Luo et al. (2014a), Yue (2014), Luo et al. (2014b) studied steam-exploded binderless fiberboard made from bamboo. The experiment showed that the fibreboard's performance was optimal under the condition of pressure of 3.0 MPa for 180 s. Its MOR was 15.8 MPa, and its IB was 0.48 MPa. The same group also studied steam-exploded palm, revealing that the properties of binderless fiberboard made from pretreated steam-exploded palm fiber were greatly improved compared with binderless fiberboard made from untreated fiber (Hai et al. 2018). Haiyang et al. (2018) studied the energy release rates of linear vibrational welded moso bamboo joints with different combinations of the inner and outer bamboo surfaces and different moisture contents. Haitao et al. (2019) investigated the influence of length and compression directions upon the behavior of parallel bamboo strand lumber (PBSL) specimens. Yue et al. (2013) studied the feasibility of using reed as raw material of wood-based panels by steam explosion. The experiment showed that the steam explosion could improve the bond strength of UF resin.

From the above literature, the MOR of the binderless medium density fiberboard produced by a single material is no more than 18 MPa, the IB is no more than 0.5 MPa, and the 24 h water absorption thickness expansion rate (WATE) is slightly higher. Their density is high if technological compression ratio of fiberboard should be between 1.2 and 1.6. If the fiberboard was made entirely of bamboo, its density would be up to 1.14 $g \cdot cm^{-3}$.

Therefore, hybrid binderless fiberboard technologies are chosen based on the principle of composite materials. Bamboo, with its relatively high strength, can be used to bear the external load, thus simulating wood cellulose. Low-density poplar can reduce the compression ratio, fulfilling the role of wood hemicelluloses. Finally, walnut shells, which contain a large amount of lignin, can serve as wood lignin. These selections are ideal for our research since all three materials are relatively abundant in China (Zhang 2017, WU and Guo 2017, Jiang et al. 2010 and Qin 2012). China's bamboo yield, walnut cultivation area and poplar afforestation area are the first in the world. Complementation among the materials and the performance of the board will be discussed.

MATERIAL AND METHODS

Materials

Poplar (*Populus* × *euramericana* 'San Martino 'I-72) veneer, moso bamboo (*Phyllostachys edulis*) branches, and Walnut shells were, respectively, obtained from Huai'an City of Jiangsu Province, forest area of Nanjing Forestry University, and Huanglong County of Shanxi Province in China.

Materials preparing and Board production

Steam ejection carried out by a QBS-80 is capable of the sudden release of high-density energy within 0.0875 s (Yu et al. 2012). First, poplar veneer was cut into wood chips of 4 - 6 cm in length and 1.5 - 2.5 cm in width, and bamboo branches were cut into bamboo chips of 2.5 - 5.5 cm in length and 1 - 2 cm in width. Next, the poplar and bamboo chips were soaked in water for one hour at 25°C. The wood and bamboo chips were then exploded with a pressure of 3.0 MPa and time of 180 s (Luo et al. 2014a). Subsequently, they were ground into fiber with 17% above 28 meshes, 20% between 28 and 48 meshes, 28% between 48 and 100 meshes, and 35% below 100 meshes in accordance to TAPPI T233 with composition. Finally, the fiber was dried to 10% moisture content. Walnut shells were ground into 100-200 mesh flour.

The below fixed parameters were adopted for the fiberboard hot-pressing process in the experiment: board preset density 0.85 $g \cdot cm^{-3}$, board surface width 250 × 250 mm, board thickness 6 mm, board blank moisture content 11%, pressing temperature 200°C, time 70 $s \cdot mm^{-1}$, and pressure 4.5 MPa. The hot-pressing curve was divided into five stages: the pressure linearly increased from 0 to 4.5 MPa in 10 s, stayed constant for 210 s, reduced to 3.0 MPa from 4.5 MPa in 20 s, stayed constant for 150 s, and dropped to 0 MPa from 3.0 MPa in 30 s.

Morphology, chemical composition, and evaluation criteria

Based on a previous study (Luo et al. 2014a), the bamboo fibers before and after several treatment conditions were air-dried and coated with gold powder in a sputter coater and observed with a Quanta 200 scanning electron microscope operated at 15 kV. Fourier transform infrared spectroscopy (FTIR) analysis was conducted to observe the functional groups in fibers before and after steam explosion using a Nicolet iS10 FTIR Spectrometer. All spectra were collected in the wavenumber range of 4000 - 650 cm^{-1} with 4 cm^{-1} resolution and 32 scans of each sample.

Phenyl alcohol extract in the chemical composition analysis was conducted in accordance with the provision GB10741-1989; the NREL in the provision GB10741-1989 method was adopted for cellulose, hemicelluloses, and lignin content tests.

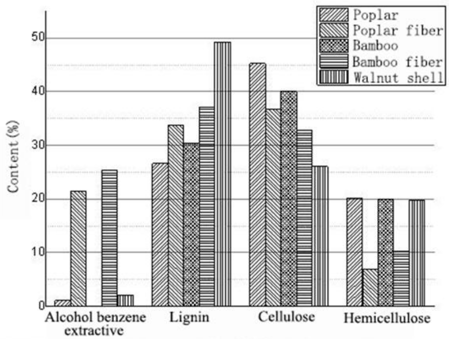
The modulus of elasticity (MOE), IB, MOR, and 24 h WATE of the binderless boards were evaluated in accordance with the Japanese Industrial Standard A 5905-2003.

RESULTS AND DISCUSSION

Feasibility of mixed biomass binderless composite

The chemical compositions of poplar, bamboo, and walnut shell were similar, as shown in Fig. 1. It was indicated that there was good compatibility among the three materials, which demonstrated the feasibility of using these materials to create mixed binderless simulated wood.

However, their chemical contents were somewhat different. Although the chemical composition of bamboo was the same as poplar, bamboo's cellulose and hemicelluloses contents were slightly lower, and its lignin content was slightly higher, which was a key bond factor and reflected the IB values reported in previous studies.



Note: Lignin content is the sum of acid-soluble lignin and acid-insoluble lignin.

Fig. 1: Chemical compositions of the materials.

After steam explosion treatment, more hemicelluloses degraded in poplar than in bamboo, and the lignin content of bamboo was higher than poplar. At a given density, single-material poplar fiberboard had a higher MOR but lower IB than single-material bamboo fiberboard. Thus, the MOR and IB of the fiberboard made from mixed poplar and bamboo were improved.

After steam explosion treatment, phenyl alcohol extract increased significantly. Under the condition of high temperature and high pressure during steam explosion, cell walls were separated from the intercellular layers, and hemicelluloses in the cell walls were degraded into monosaccharides (Shevchenko et al. 2000), including dextrose, xylose, glucomannan, and arabinose. These monosaccharides along with monosaccharides from the material were dehydrated and generated furfural.

The lignin content of walnut shells was up to 49.23%, far more than that of wood and bamboo. In the hot-pressing process, lignin could condense with furfural and combine with hemicelluloses, generating lignin-carbohydrate complex (LCC) (Jin 2002, Santiago-Medina et al. 2017). The IB value of the board was improved by the appropriate addition of walnut shell. Benzyl ester bond linkages, benzyl ether linkages, phenolic glycosidic linkages, etc., were created between lignin and carbohydrates.

Material ratio research

Effects on fiberboard properties by the ratio between poplar and bamboo

Different ratios among the materials were a key factor in the properties of the fiberboard. Tab. 1 shows the properties of the binderless fiberboard with changing ratios of poplar to bamboo raw materials. When using a single raw material, the IB value of the poplar binderless fiberboard was 0.54 MPa, and the IB value of the bamboo fiberboard was 24% higher at 0.67 MPa.

When using mixed materials, the walnut shell flour content was tentatively set at 10%, the total content of poplar and bamboo was 90%, and the mass ratio of poplar to bamboo was adjusted. With the decrease of poplar fiber and the increase of bamboo fiber, the IB of the board first went up and then down. When the ratio was 6:4, the IB reached a maximum value of 1.07 MPa, greater than that of any other fiberboard with different poplar to bamboo ratios. Due to the high strength and great aspect ratio of bamboo fiber, its interwoven degree among fibers was better than that of poplar fiber, while the density of poplar fiber solved the high compression ratio problem. Therefore, fiberboard made from mixed poplar and bamboo fiber as well as walnut shell flour could reflect their respective advantages.

Tab. 1: Effects on properties of steam-exploded binderless fiberboard by the ratio between poplar wood and bamboo.

No	**Poplar: bamboo: walnut shell ratio (%)**	**Density ($g·cm^{-3}$)**	**IB (MPa)**	**MOR (MPa)**	**MOE (MPa)**	**24 h WATE (%)**
1	100:0:0	0.87(0.01)	0.54(0.02)	20.9(1.37)	2389(95.87)	6.7(0.39)
2	0:100:0	0.88(0.01)	0.67(0.02)	18.8(1.01)	2248(89.57)	7.7(0.22)
3	90:0:10	0.88(0.02)	0.68(0.06)	21.4(1.01)	2758(145.24)	7.7(0.63)
4	72: 18:10	0.87(0.02)	0.81(0.06)	23.1(1.12)	3170(132.76)	8.9(0.51)
5	**54: 36:10**	**0.88(0.01)**	**1.07(0.03)**	**24.6(1.10)**	**3303(93.16)**	**7.6(0.21)**
6	36: 54:10	0.88(0.02)	0.65(0.07)	20.6(1.31)	2539(96.73)	9.1(0.24)
7	18: 72:10	0.88(0.04)	0.58(0.03)	19.6(1.97)	2478(125.16)	7.7(0.54)
8	0: 90:10	0.88(0.02)	0.78(0.05)	20.1(1.09)	2637(121.13)	7.8(0.24)

Note: The value in parentheses is standard deviation.

The MOR and MOE of the board increased first as the ratio decreased. When the ratio was 6:4, the MOR reached a maximum of 24.6 MPa, and the MOE reached a maximum value of 3303 MPa, which were greater than those of fiberboard solely made from bamboo or poplar. After that, the MOR and MOE of the board decreased slightly as the ratio further decreased.

Hemicellulose is the most water-absorbent substance in wood. A large amount of hemicelluloses degraded after steam exploding, which improved the water resistance of the board. It can be seen in Tab. 1 that the 24 h WATE of the fiberboard ranged from 6.7% to 9.1%, which was far lower than the 17% requirement of the Japan JIS A 5905-2003 standard. Therefore, steam-exploded mixed binderless fiberboard had a water resistance property. It was obvious that properties of the board were much improved at the 6:4 ratio with 10% walnut shell flour.

Effect on fiberboard properties by the ratio of walnut shell

Further, the amount of walnut shell flour in the board was evaluated at the poplar to bamboo mass ratio of 6:4, as shown in Tab. 2.

Tab. 2: Effect on properties of steam-exploded binderless fiberboard (with poplar to bamboo mass ratio of 6:4) by the ratio of walnut shell.

No.	Walnut shell ratio (%)	Density ($g·cm^{-3}$)	IB (MPa)	MOR (MPa)	MOE (MPa)	24 h WATE (%)
9	0	0.87(0.02)	0.73(0.02)	19.3(1.02)	2696(85.59)	7.5(0.21)
10	5	0.89(0.01)	0.86(0.02)	20.6(1.02)	2834(51.97)	8.3(0.20)
11	10	0.88(0.01)	1.07(0.03)	24.6(1.10)	3303(93.16)	7.6(0.21)
12	15	0.88(0.01)	0.94(0.03)	18.9(1.13)	2563(85.22)	8.3(0.18)
13	20	0.87(0.01)	0.66(0.02)	16.1(0.94)	2347(49.14)	8.6(0.23)

Note: The value in parentheses is standard deviation.

It can be seen in Tab. 2 that the IB of the board increased first and then decreased with the growing proportion of walnut shell flour at the constant 6:4 ratio of poplar to bamboo. The IB of the board was maximized at 1.07 MPa when the content of walnut shell flour was 10%. The main reason is that walnut shell flour contains a lot of lignin, which could further exert a condensation effect at the glass transition temperature, and the bonding force between fibers could be increased.

Furthermore, the addition of walnut shell flour filled the gaps between bamboo fiber and wood fiber and provided stress transferring. The MOR and MOE of the board were maximized at the same ratio as the IB. Actually, when there was too much flour and insufficient fiber, the flour's stress transferring action was changed into a bearing stress transferring action, the interwoven degree between materials decreased, and the performance of the board was reduced.

As adding walnut shell flour into mixed fiberboard could increase water resistance performance, the 24 h WATE of the board was maintained between 7.5% and 8.6% for all flour content ratios, which was lower than the 17% requirement of the Japan JIS A 5905-2003 standard. This indicated that the change of walnut shell flour ratio had no evident impact on the WATE of the board, and the fiberboard with walnut shell flour still exhibited water resistance property.

Through comprehensive analysis, it can be concluded that the properties of the board were optimal in the experiment when the material mass ratio was set as 54% poplar fiber, 36% bamboo fiber, and 10% walnut shell flour. Its mechanics' properties were higher and the 24 h WATE was much lower than the requirements of the Japan JIS A 5905-2003 standard.

Environmental scanning electron microscopy

Surface morphological features of poplar and bamboo after steam explosion treatment are displayed in Figs. 2 and 3, resp.

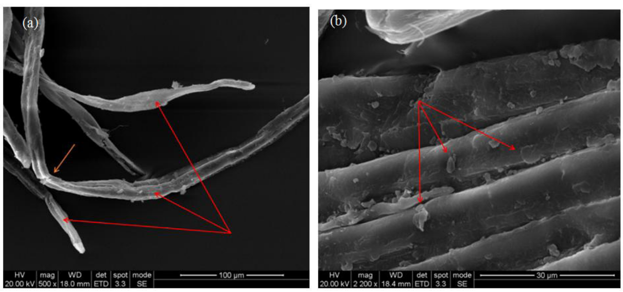


Fig. 2: The surface morphology of poplar after steam explosion. (a) 500X. (b) 2200X.

Figs. 2a and 3a show that the fibers were thinner and fluffy with smaller diameters, and there were more long fibers retained through the steam-exploding process of poplar and bamboo; in fact, the slender ratio was over 100. More retention of long fibers may have occurred because fibers were more easily split longitudinally and less easily cut laterally, and the intercellular layer was separated from the cell wall. The poplar fiber and bamboo fiber could deteriorate the interwoven degree between fibers and affect the MOR of board. There were many active hydroxyl groups (–OH) on the fiber surface. When cell wall was exposed, the contact area between fibers was enlarged. During the hot pressing process, the distance between fibers was shortened. When the distance was close enough ($<2.9\times10^{-10}$ -3.0×10^{-10} m), the hydrogen atom in the hydroxyl group of cellulose would bond not only with the oxygen atom by the main valence but also with the negatively charged hydrogen atom in the hydroxyl group of an adjacent cellulose molecule, forming a hydrogen bond (0H…H) by the negative valence bond. In addition to the van der Waals force between cellulose molecules, the binderless bonding effect of the board was intensified with improved IB value. After steam explosion, fiber cells were crushed and flattened. Capillaries and microcapillaries in fiber cells normally transfer and store water. When the cells were crushed, the water absorption capacity of the fiber decreased (Xu 2002), which improved the water resistance property of the board.

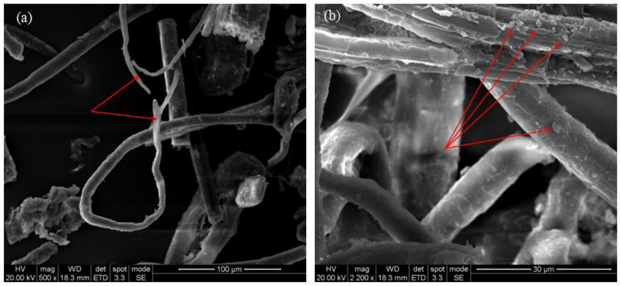


Fig. 3: The surface morphology of bamboo after steam explosion. (a) 500X. (b) 2200X.

It can be seen in Figs. 2b and 3b that after explosion, transverse cracks occurred on some single fibers and thus fiber strength decreased, adversely affecting the MOR of the board. After the steam exploding process, an amorphous scraping substance could be found on the surface of poplar fiber and bamboo fiber, which was composed of accumulated degrading hemicelluloses and lignin (Chen and Li 1999), creating conditions for the combination of lignin and carbohydrates to form new LCC.

Fourier transform infrared spectroscopy analysis

Through literature review (Yue 2014, Jin 2002), infrared spectrum characteristic peak attributions were determined as shown in Tab. 3. The FTIR results of each sample are shown in Fig. 4, in which No. 1 is poplar flour, No. 2 is poplar fiber after steam explosion, No. 3 is bamboo flour, No. 4 is bamboo fiber after steam explosion, No. 5 is walnut shell flour, No. 6 is mixed binderless fiberboard with 60% poplar fiber and 40% bamboo fiber, and No. 7 is mixed binderless fiberboard with 10% walnut shell flour, 54% poplar fiber, and 36% bamboo fiber. Test samples were all broken down by a grinder and sieved for 80 mesh flour.

Tab. 3: Infrared spectrum characteristic peak attributions.

Wave number (cm^{-1})	Definition
3415	O-H stretching vibration
2920	C-H stretching vibration
1740	C=O stretching vibration
1601	Benzene ring and conjugate alkene
1509	Benzene ring skeleton
1462	CH2 bending vibration
1328	O-H out-of-plane vibration
1254	C-O-C stretching vibration
1054	C-O stretching vibration

As shown in Fig. 4a, at the wavenumber of 3415 cm^{-1} was the hydroxyl stretching vibration absorption peaks of lignin and total cellulose. After steam explosion, the –OH stretching vibration absorption peak of poplar and bamboo was evidently enhanced, especially that of bamboo.

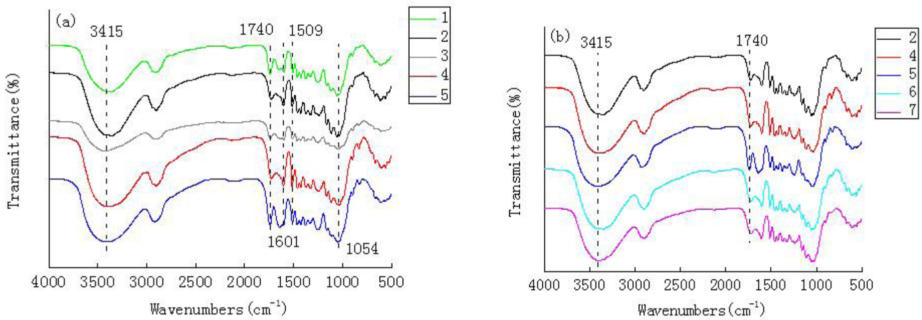


Fig. 4: FTIR spectroscopy of different samples.

At the wavenumber of 1740 cm^{-1} was the characteristic absorption peak of hemicelluloses, generally considered as the stretching vibration peak of carbonyl and carboxyl (C=O) of polyxylose. After steam explosion, the characteristic peaks of hemicelluloses were weakened in poplar and bamboo. The reason was that, after steam explosion, the hemicelluloses of the materials had undergone deacetylation, forming organic acid (Chen and Li 1999) and leaving a reduced amount of hemicelluloses. At the wavenumbers of 3415 cm^{-1} and 1740 cm^{-1}, the characteristic peaks all shifted to the right, affected by the hydrogen bond between molecules. During the steam explosion process, each component was degraded to a certain extent, among which the hemicelluloses were affected the most.

The break of the cycling bond of cellulose and hemicelluloses exposed more hydroxyl groups on the surface of the fiber, increasing the concentration of –OH, which enabled the possible formation of hydrogen bonds between molecules. The absorption peaks at the wavenumbers of 1601 cm^{-1} and 1509 cm^{-1} were characteristic peaks of lignin. After steam explosion, the lignin absorption peaks of poplar and bamboo were enhanced. After steam explosion, in the range of wavenumbers from 1000 cm^{-1} to 1600 cm^{-1}, the characteristic peaks of the CO and CH_2 groups of cellulose and lignin both increased to some degree, indicating that partially dissolved lignin and hemicelluloses had re-formed into lignin-like substances under high exploding pressure and long pressuring time (Chen and Liu 2007). Walnut shell contains a lot of lignin, far more than that of poplar and bamboo. Therefore, at the wavenumbers of 1601 cm^{-1} and 1509 cm^{-1}, the lignin characteristic peaks of No. 5 were both stronger than those of No. 1 and No. 3 with more evident enhancement extent, which is consistent with the results of chemical composition analysis.

Fig. 4b shows that, at the wavenumber of 3415 cm^{-1}, the characteristic peak of the hydroxyl group of No. 7 was weakened and had a broadened peak shape compared with that of No. 6, which was related to the formation of hydrogen bonds and the IB of the board could be improved with the addition of walnut shell flour, and at the wavenumber of 1740 cm^{-1}, there was no significant change of the hemicelluloses' characteristic peak of No. 7, and the addition of walnut shell flour had little impact on the water resistance of the board. At the wavenumber of 3415 cm^{-1}, the stretching vibration absorption peak of the hydroxyl (O-H) of No. 7 was lower than that of No. 2, No. 4, and No. 5, which were all raw material. This was related to the formation of hydrogen bonds contributing to the IB of the board. At the wavenumber of 1740 cm^{-1}, the hemicelluloses' characteristic absorption peak continued to drop compared with the raw material, indicating that the hemicelluloses continued to hydrolyze, generating furfural, decyl alcohol, organic acid, and so on. Thus, a smaller amount of hemicelluloses remained after hot-pressing.

CONCLUSIONS

It is feasible and reasonable to manufacture eco-friendly, hot-pressed mixed biomass binderless composites of high-quality using fast-growing poplar processing residue, bamboo branches, and walnut shell as raw materials. High-strength bamboo can simulate wood cellulose, low-density poplar can improve the board compression ratio, simulating wood hemicelluloses, and lignin-rich walnut shells can serve as wood lignin.

The experimental results show that when the material mass ratios are set to 10% walnut shell powder, 54% poplar fiber, and 36% bamboo fiber, the properties of the fiberboard are optimized.

Steam-explosion technology can be adopted for the preparation of poplar and bamboo fiber, separating cell walls and intercellular layers and partially degrading cellulose and hemicelluloses to generate monosaccharides and form new LCC such that there is good bonding between fibers.

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