Influence of the Mechanical Properties of Tobacco Stalk Fiber Cell Wall on Particleboard Panels

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***Abstract*-Natural fibers are used to improve mechanical properties of composites materials; research into the influence of the mechanical properties of various fibers' cell walls on the mechanical properties of composites is very important for producing high-quality composites. The aim of this research is to study the influence of the mechanical properties of tobacco stalk fiber cell wall on the mechanical properties of particle-based panels, comparing the properties of particleboards made with tobacco stalks from different parts of the stem (bottom, midpoint, and top) and treated tobacco stalks from the midpoint of the stem with 1% NaOH solution at 100℃ for 0, 30, 60, and 90 mins, respectively. The results show that the mechanical properties of tobacco stalk fiber cell walls from different parts of the stalk differ and are reduced after the tobacco stalk is treated with 1% NaOH solution. The mechanical properties of the tobacco stalk-based particleboard panels were negatively correlated with the mechanical properties of the tobacco stalk fiber cell wall. After the 1% NaOH treatments of 0-60 min, the middle lamellae were broken, and the mechanical properties of the particleboard were improved; after the 1% NaOH treatment of 90 min, the fiber cell walls were broken and the MOR and MOE of the panels were reduced. Thus, it was concluded that the mechanical properties of fiber cell wall can be reduced to improve the mechanical properties of particleboard panels. The improvement of the mechanical properties of particleboard with no-added- Formaldehyde resin by reducing the mechanical properties of fiber cell wall will be studied in future.**

Keywords- Tobacco Stalk; Mechanical Properties of Fiber Cell Wall; NaOH Solution Treatment; Particle Board Panels; Nano-indentation

I. INTRODUCTION

Natural fibers are widely used in a variety of industries [1-3], especially the preparation of composite materials. Research on the relationship between the mechanical properties of natural fiber cell wall and the properties of composites is very important in order to optimally use natural fibers and to manufacture composites with high mechanical properties. However, such research has rarely been reported.

Tobacco (Nicotiana tabacum) is planted worldwide to produce tobacco leaves for cigarettes. Tobacco leaves are used to make cigarettes and other tobacco products, and the stalks are normally burned as trash. Tobacco stalk is an excellent woody material that can be used to prepare paper, pulp, and wood-based panels. Because wood costs are generally rising as the demand surpasses the supply, many kinds of agricultural residues, such as wheat straw, rice straw, sorghum stalks, kiwi prunings, castor stalks, and industrial hemp, are currently being used or are being introduced to prepare pulp, paper, and composites in the wood industry [4-8]. To satisfy the needs of the wood industry, research on the properties of tobacco stalk-based particleboard, among other natural fibers, is needed.

Nanoindentation, a technology that emerged in 1987, is a method for studying objects at super-small scales; it is particularly used to study metal surfaces and extremely thin (film) surfaces [9]. In 1997, it was first used to study the mechanical properties of cell walls in wood materials [10]. Nanoindentation is now being widely used in wood science for such researches as studying the mechanical properties of adhesion layers in wood-based panels [11], the influence of modifications such as heat and chemicals on the mechanical properties of wood cell walls [12-13] and the cell walls of agricultural crop stalks [14], hardwoods [15], and softwoods [10]. Recent research has shown that there exists a significant difference in mechanical properties of cell walls along the length of silvergrass stalks [16]. However, at present few studies have involved investigation on mechanic characteristic of tobacco stalks. In this study, nanoindentation is used to test the mechanical properties of tobacco stalk fiber cell walls from different parts of the stem.

Sodium hydroxide (NaOH) is a chemical reagent commonly used to treat wooden and non-wooden fibers to improve the mechanical properties of composites. The mechanical properties of the composites using hemp fiber treated by NaOH solution show improvements [1], and the mechanical properties of the composites made with wooden fiber or powder treated by NaOH are likewise superior to the composites manufactured from untreated fiber [17]. For this reason, treatment of paper pulp with NaOH has been used to improve the properties of paper [18]. However, there is little information about the influence of NaOH treatment on the mechanical properties of the cells walls of tobacco stalk fibers and the particleboard panels made with them.

The object of this study is to investigate by nanoindentation the mechanical properties of tobacco stalk fiber cell walls from different parts of tobacco stalk along the stem and the mechanical properties of cell walls of tobacco stalk fiber treated by 1% NaOH solution, and to gather information about the relationship between the mechanical properties of fiber cell wall and the mechanical properties of the particleboard panels made from those fibers.

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II. MATERIALS AND METHODS

A. Materials

Tobacco stalk sections from Yunnan, China were cut from the bottom, midpoint and top parts of the stem (see Fig. 1) to 3–5 cm in length, 0.5–1.0 cm in width, and 3–12 mm in thickness using a chipping machine at the Wood-Based Panel Laboratory (College of Materials Engineering, Southwest Forestry University, Kunming, Yunnan, China). The sections were then re-chipped in the same place with a hogging machine to smaller-sized particles of 5–8 mm in length, 3–5 mm in width and 2–3 mm in thickness, and oven-dried to a moisture content (MC) of 3–5% at a constant 100±2℃ temperature.

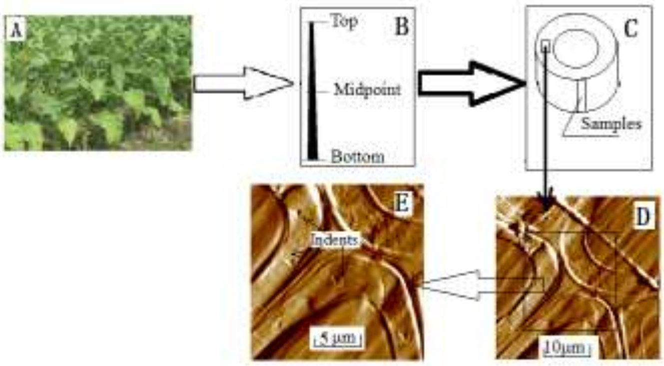

Fig. 1 Structure of tobacco stalk fiber cell wall (Note: A. Tobacco plants; B. Three groups of tobacco stalk samples in the experiment; C. The cross structure of tobacco stalk; D. The structure of tobacco stalk fiber cell wall by AFM; E. The indents on fiber cell wall.)

B. Sample Preparation

Three groups of samples from the midpoint of the stem were, after oven drying, treated with 1% NaOH solution for 0, 30, 60, and 90 mins at 100 ˚C; the liquid ratio of the 1% NaOH solution to the samples by weight was 1:100. The 1% NaOH solution was made from sodium hydroxide (A.R., bought in Kunming, Yunnan, China). The samples were then washed and neutralized with distilled water and 20% acetic acid until the pH value was about 7.0. They were then dried and prepared for nanoindentation and particleboard fabrication.

C. Nanoindentation

To investigate the mechanical properties of tobacco stalk fiber cell walls from different parts of the stem and the influences of the 1% NaOH treatments on the mechanical properties, the three groups (two samples for each group) of samples from the three locations (bottom, midpoint and top) and the three groups of treated samples from the midpoints of the stem with 1% NaOH solution for 30, 60, and 90 mins were cut into small pieces of about 2 x 6 mm in size after oven drying for nanoindentation. The individual small piece was glued on the chipboard and then put into the plastic mold. The chipboard, whose width was equal to the mold diameter, was used to keep the samples in correct direction and to make sure the axis of fibres to be parallel to the nanoindentation direction. The epoxy resin as embedding media was added into the plastic mold [19] and the molds were placed in an oven under vacuum for 30-40 mins to remove any air; they were then cured in the same oven at 70˚C for 8 -10 h. The cured samples were cut until the epoxy resin was removed and the hemp stalk surface was revealed. Then the top face was cut with a glass knife until the whole piece was cut. Finally, the top surface was smoothed with a diamond knife. The final specimens were put into a special container to protect the sample surfaces. All nanoindentation experiments were performed at the Center for Renewable Carbon using a TriboIndenter system. A Berkovich indenter, three-sided pyramid with an area-to-depth function was used for all experiments. The indentation was performed in a displacement-controlled mode and consisted of three segments. In the first segment, loading force was applied at a constant displacement rate of 5 nm/s until the designed indentation depth of 200 nm was reached; the maximum force was held at this depth for 10 s and then unloaded at a constant displacement rate of 10 nm/s until 90% of the loading force was removed. Forty to sixty indentations were made on a cross-section of six to ten cell walls for each sample (shown in Fig. 1E). At the end, the hardness (H) and the elastic modulus (E) were calculated from the load-displacement data according to the method of Oliver and Pharr [20].

D. Particleboard Manufacturing and Testing

The six groups of tobacco stalk referred to above were blended with a 12% liquid urea-formaldehyde (UF) resin (UF resin provided by Xinfeilin Wood Based Panel Co. LTD. Kunming, Yunnan, China), based on the oven-dry weight of the stalks and 1% catalyst (NH_4Cl, A. R., bought in Kunming, Yunnan, China), and the oven-dry weight of the UF resin. The resulting mat was formed manually to a size of 32 cm x 42 cm. Three panels were made for each group. The mats were pressed in a laboratory press (XLB-500X500X2, made in Qingdao, Shandong, China), at 150˚C and 3.5 MPa for three minutes, becoming particleboards with a thickness of 10 mm and the target density of 0.70 g/cm^3.

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Samples for the various tests were prepared from the experimental boards according to the PR China standard (GB/T 17657-1999) for wood-based panels. The target properties included the modulus of rupture (MOR), modulus of elasticity (MOE), and internal bond strength (IB). The MOR and MOE tests used four samples with dimensions of 250 mm x 250 mm x 10 mm, and the IB test used six samples with dimensions of 50 mm x 50 mm x 10 mm. The tests were completed using a Universal Testing Machine (AG-50KNI, Japan).

E. SEM Analysis

Scanning electron microscopy (SEM) was used to research the surface morphology and microstructure of tobacco stalk samples treated and untreated by NaOH solution. These samples were oven-dried at 70˚C for 8 -10 h after 1% NaOH solution treatments of different time or hot water treatment of 240 mins. Then these samples were cut into small pieces of about 5 mm X 5 mm for research and the samples were coated with gold. The instrument was a Quanta 200 (FEI Co., Holland) with a coating instrument of E-1010 (HITACHI Co., Japan).

III. RESULTS AND DISCUSSION

A. Mechanical Properties of Fiber Cell Walls and Panels Made from Different Parts of Tobacco Stalk

The mechanical properties of fiber cell walls of different sections of tobacco stalk compared to the mechanical properties of particleboard panels made from the different parts of tobacco stalk along the stem are shown in Table 1.

TABLE 1 THE PROPERTIES OF FIBER CELL WALL AND PARTICLEBOARD OF DIFFERENT STALK PARTS

Samples	Elastic modulus (GPa)	Hardness (GPa)	MFA (˚)	IB (MPa)	MOE (MPa)	MOR (MPa)
Bottom	11.5 (0.5)	0.45 (0.08)	20.7 (1.0)	0.59 (0.06)	2440 (25.1)	23.3 (1.1)
Midpoint	13.4 (0.7)	0.49(0.05)	18.0 (1.0)	0.50 (0.09)	2289 (24.0)	22.8 (1.1)
Top	9.48 (0.6)	0.35 (0.06)	24.9 (1.1)	0.63 (0.05)	2845 (26.7)	24.2 (1.0)

Note: The standard deviations (in bracket) represent the variation of elastic modulus, hardness, MFA, IB, MOE and MOR, respectively.

The mechanical (including elastic modulus and hardness) properties of fiber cell walls cut from the stalk midpoint were the highest; those of fiber cell walls from the top part were the lowest, and those of fiber cell walls from the bottom part were between those of the top part and the midpoint part. These results agree with previous report [19]. The mechanical properties of fiber cell walls are affected by a number of factors, including the density, the MFA, and the chemical composite content (especially the lignin content); as the MFA of the cell walls increases, the mechanical properties (especially the elastic modulus) decrease [21]. In this research, the MFA was negatively correlated with the mechanical properties of the tobacco stalk fiber cell wall, and this agrees with the research reported in [21].

In addition, the mechanical properties of the tobacco stalk-based particleboard panels were negatively correlated with the mechanical properties of the tobacco stalk fiber cell walls. The coefficient of correlation between the IB and the elastic modulus was -0.97 (P value was 0.08), and the coefficient of correlation between the IB and the hardness was -0.90 (P value was 0.09). The coefficient of correlation between the MOR and the elastic modulus was -0.99 (0.05), and the coefficient of correlation between the MOR and the hardness was -0.99 (P value was 0.05). The coefficient of correlation between the MOE and the elastic modulus was -0.99 (P value was) and the coefficient of correlation between the MOE (P value was 0.04) and the hardness was -0.99(P value was 0.01). Thus, it was demonstrated that for the purposes of making particleboard, the mechanical properties of the materials can be improved by reducing the mechanical properties of fiber cell wall.

Particleboard is made by quickly compressing the fiber cell walls under conditions of high temperature and high pressure. Consequently, if the mechanical properties of fiber cell wall are low and the fiber cell wall is easy to be compressd, the mechanical properties of the fiber itself can be retained and a good interface between the resin and fiber may be formed more easily. If the moisture content (MC) of particles is too low, it can reduce the physical and mechanical properties of particleboard while the mechanical properties of fiber cell wall are increased as the MC of fiber cell wall is reduced [22]. Otherwise, the fiber cell walls are hard to be compressd and the mechanical properties of the fiber itself [23] are not retained, reducing the mechanical properties of the resulting particleboard panels.

B. Mechanical Properties of Fiber and Panels Treated by Naoh

To further assess the influence of the mechanical properties of fiber cell wall on the mechanical properties of particleboard panels, the properties of the particleboard panels using tobacco stalk particles treated with 1% NaOH solution for varying durations were studied. The mechanical properties of the fiber cell walls and the resulting particleboards are shown in Table 2.

TABLE 2 THE PROPERTIES OF TOBACCO STALK FIBER AND PARTICLEBOARD TREATED BY NAOH SOLUTION

Treatment time (min)	Elastic modulus	Hardness (GPa)	MOE (MPa)	MOR(Mpa)	IB(Mpa)	Density（g/cm^3)

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	(GPa)					
0	13.4(0.7)	0.49 (0.05)	2289(24.0)	22.8(1.1)	0.50(0.1)	0.72(0.07)
30	12.4(1.3)	0.36(0.03)	2713(22.2)	24.3 (1.2)	0.57(0.1)	0.69(0.02)
60	11.9(1.1)	0.31(0.04)	3242(21.0)	25.8 (1.7)	0.64(0.1)	0.71(0.05)
90	11.4(1.1)	0.27(0.03)	2154(25.1)	18.3 (1.3)	0.76(0.1)	0.72 (0.04)

Note: The standard deviations (in bracket) represent the variation of elastic modulus, hardness, MOE, MOR, IB and density.

As shown in Table 2, the mechanical properties, including MOE and hardness, of the tobacco stalk fiber cell walls were reduced as the 1% NaOH solution treatment time increased from 0 min to 90 min. The exact chemical composition of the cell wall can also affect the mechanical properties of the cell walls. The fiber cell wall is a composite of cellulose, hemicelluloses and lignin arranged together closely (shown in Fig. 2A). There exists a positive relationship between the lignin content and the mechanical properties of the cell wall, with the hardness of the cell wall being particularly affected [24]. These chemical composites are distributed throughout the different cell wall layers, including the middle lamella, the primary wall (P), and the secondary walls (S1 layer, S2 layer, and S3 layer). Cellulose acts as a reinforcing material embedded in the lignin and hemicelluloses; after the samples were treated with 1% NaOH solution, considerable amounts of lignin, hemicelluloses, and non-cellulose were extracted by the solution [25] and the middle lamellae were ruptured (Shown in Fig. 2B and Fig. 2C). As the 1% NaOH solution treatment time increased, more and more lignin, hemicelluloses, and non-cellulose were removed from the fiber cell walls, and the fiber cell walls' mechanical properties reduced more (show in Fig. 2D and Table 2).

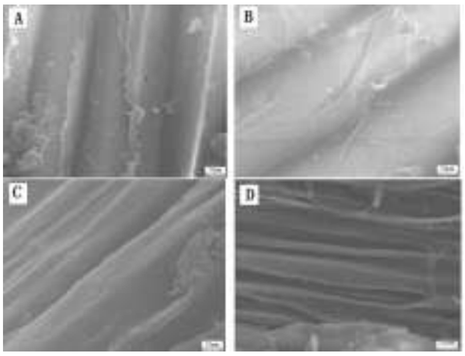

Fig. 2 The surface morphologies of tobacco stalks treated by sodium hydroxide solution for different time
Note: A. No treatment; B. Treatment time is 30 min; C, Treatment time is 60 min; D, Treatment time is 90 min.

With the reduction of the mechanical properties of tobacco stalk fiber cell wall, the fiber cell wall itself becomes easier to be compressed, permitting an improved interface between the resin and fiber. As a result, the IB of the panel improved after the particles were treated with 1% NaOH solution for zero to sixty minutes. However, the fiber cell walls were broken following ninety minutes of treatment with 1% NaOH solution, reducing both the MOR and MOE of the particleboard. Both the MOR and MOE of the particleboard improved when the particles were treated for zero to sixty minutes.

IV. CONCLUSIONS

The mechanical properties of tobacco stalk fiber cell walls from different parts of the stem were different and the mechanical properties of the tobacco stalk fiber cell walls taken from the midpoint of the stem were reduced after the treatments with 1% NaOH solution. The reduced mechanical properties of fiber cell walls could lead to increased mechanical properties of the particleboards made from those fibers. Thus, the mechanical properties of tobacco stalk-based particleboard panels can be improved by reducing the mechanical properties of the stalk fiber cell walls.

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