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Evaluation of elastic modulus and hardness of crop stalks cell walls by nano-indentation

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ABSTRACT

Agricultural biomaterials such as crop stalks are natural sources of cellulosic fiber and have great potential as reinforced materials in bio-composites. In order to evaluate their potential as materials for reinforcement, the nano-mechanical properties of crop-stalk cell walls, i.e. those of cotton (Gossypium herbaceu) stalk, soybean (Glycine max) stalk, cassava (Manihot esculent) stalk, rice (Oryza sativa L.) straw, and wheat (Triticum aestivum L.) straw, were investigated by means of nano-indentation and atomic force microscopy (AFM). The elastic modulus of wheat straw was found to be 20.8 GPa, which was higher than that of the other four crops. The highest hardness was observed in cotton stalk at 0.85 GPa. The elastic moduli of the crop stalks were lower than those of most of the hardwood species, but higher than that of some softwoods and of lyocell fiber. The mean value of the hardness of the five crop stalks' cell walls was higher than those of wood or lyocell fiber.

1. Introduction

Agriculturally derived (agro-based) bio-fiber resources have been used by people for centuries. The use of straw for the production of paper and as a reinforcement material for mud huts was well established 2000 or more years ago (Rowell et al., 1997; Sun and Cheng, 2002). The use of agro-based fibers in paper and composite products has increased dramatically with advances in research in the fields of chemical and materials science. As a result, a considerable amount of literature has been published. Many countries are currently facing increasing pressure to find alternate sources for renewable bio-products; however, additional harvesting of forests for wood is no longer a viable alternative in many locations. Agro-based bio-fibers from agricultural residues represent an abundant, inexpensive, widely distributed, recyclable, versatile, biodegradable, and readily available source of renewable lignocellulosic biomass (Liu et al.. 2005; McKendry, 2002; Rowell et al.. 1997). Additionally, there are prospective commercial applications for the use of agricultural residues in bio-composites (Eichhorn et al.. 2001). As both world population and grain production have grown, a considerable amount of agricultural residue is now generated annually. It is thus becoming evident that these abundant agricultural bio-fiber resources can be turned to good effect in the manufacture of composites. In addition, agricultural residues have also other applications, such as bio-fuel.

Wood particles and wood fibers have been used for years as reinforcement materials in polymer composites (Ichazo et al., 2001; Mahlberg et al., 2001; Yin et al., 2007). The efficient utilization of agricultural residual fiber resource in manufacturing bio-composites is of great interest today due to the decrease in the supply of conventional raw materials from forests. Some studies (Albano et al., 2001; Eichhorn et al., 2001; Helbert et al., 1996) on polymer composites and nano-composites reinforced with crop stalk fibers have also shown their advantageous properties compared with those of wood plastic composites. In recent years, strawboard. which uses MDI as an adhesive, has been newly developed and is commercially available in several countries (Mo et al., 2005). In some respects, strawboard has better properties than wood particleboard. For example, strawboard is lighter in weight, emits almost no formaldehyde, and is more water resistant than particleboard in addition, it can be used in a growing range of applications, including automotive, packaging, and building materials (Mo et al., 2005).

To utilize agro-based fibers efficiently in composite products, it is necessary to better understand the strength properties of the individual fibers at the microscopic level. The better the understanding of the characteristics of the raw material, the more extensive the use of this kind of bio-resource is likely to be. Kersavage (1973) and Mott et al. (1995) developed methods based on use of

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the environmental scanning electron microscope (ESEM) and video image analysis (VIA) to measure the tensile modulus of a single wood pulp fiber. But natural wood cell walls properties make the ESEM–VIA method difficult to use both because it is time-consuming and because the mechanical or chemical methods to separate fibers typically result in more or less damage to the fibers (Xing et al., 2008).

The nano-indentation technique is a method to determine the mechanical properties of wood cell walls at the nano scale without requiring chemical or mechanical modifications to isolate individual wood fibers as required in the ESEM–VIA method. Further, the technique has been used to determine the longitudinal elastic modulus and hardness of wood cell walls (Gindl et al., 2004; Tze et al., 2007; Wang et al., 2006). refined loblolly pine fiber (Xing et al., 2008). and regenerated cellulose fibers (Gindl et al., 2006; Lee et al., 2007). However, there are no reported data on using this technique to evaluate the mechanical properties of the cell walls of crop stalks or straw.

In order to get information on the mechanical properties of agricultural stocks, this research aimed to investigate the nano-mechanical properties of the cell walls of crop stalks, i.e. cotton (*Gossypium herbaceu*) stalk, soybean (*Glycine max*) stalk, cassava (*Manihot esculent*) stalk, rice (*Oryza sativa* L.) straw, and wheat (*Triticum aestivum* L.) straw, by the use of nano-indentation and atomic force microscopy (AFM) techniques.

2. Methods

2.1. Sample preparation

Five different crop stalks were collected from China. Air-dried cotton stalk, soybean stalk, cassava stalk, rice straw, and wheat straw were used. The rice straw and wheat straw segments were selected between nodes; the cotton, soybean, and cassava stalks were cut into small blocks of 2 × 2 × 5 mm in the tangential, radial, and longitudinal directions, respectively, using sharpened blades, and the rice and wheat straws were cut into 2 × 5 mm (*W* by *L*) with the natural thickness of the straw. The small specimens were then embedded in epoxy resin, which was formulated of cycloaliphatic epoxide resin (ERL-4221) (2.5 parts), polycoldieposide (DER-736) (1.5 parts), nonenyl succinic anhydride (NSA) (6.5 parts), and dimethylaminoethanol (DMAE) (0.1 parts) (Spurr, 1969). The embedded specimens were placed in a desiccator under vacuum for 12 h and then were cured in an oven at 70 °C for 8 h. The cured specimens were cut into small blocks and were glued to acrylic blocks for microtoming. The specimens were then mounted onto an ultramicrotome and cross-sectioned with a glass knife. Finally, the specimen surfaces were cut using a diamond knife to obtain a smooth surface. The smoothed specimens were conditioned at 21 °C and 60% relative humidity in the nano-indentation test room for at least 24 h before testing

2.2. Nano-indentation procedure

All nano-indentation experiments were performed by a Nano Indenter II (MTS System Corp. Oak Ridge, TN. USA) that was equipped with a three-sided pyramid diamond indenter tip (Berkovich type) at the Oak Ridge National Laboratory. The indentation experiment utilized four segments. The first segment was the approach segment with an indenter surface approach rate of 10 nm/s. Once the tip contacted the sample surface, a constant displacement rate of 5 nm/s was applied until a designed indentation depth of 200 nm was reached. At this depth, the maximum loading force was held for 30 s prior to the ultimate unloading. The holding segment was used for the thermal drift corrections. In the unloading segment, a constant displacement rate of 10 nm/s was applied until 90% of the maximum loading force was removed. At the end of the experiment, the specimen was examined by the Olympus optical microscope of the Nano Indenter II to evaluate the position and quality of the indentations. For each specimen, 40 indentations were made on the cross-section of 5–9 fiber cell walls that existed around vascular tracheids.

The hardness (H) and the elastic modulus (E) were calculated from the load–displacement data. As the indenter was allowed to penetrate into the specimen, both elastic and plastic deformation occurred and only the elastic portion of the displacement was recovered during unloading. Nano-indentation hardness is defined as follows:

$$H = \frac{P_{max}}{A} = \frac{P_{max}}{24.5h_c^2}$$

where P_{max} is the load measured at a maximum depth of penetration (h) in an indentation cycle; A is the projected contact area; and h_c is the contact depth of the indentation, which is given by

$$h_c = h - 0.75\frac{P_{max}}{S} \quad (2)$$

where S is the slope $(\frac{dP}{dh})$ of the initial portion of the unloading curve at $h = h_{max}$, and 0.75 is a constant that depends on the indenter geometry.

As the indenter was allowed to penetrate into the sample, both elastic and plastic deformation occurred, and only the elastic portion of the displacement was recovered during unloading.

The elastic modulus of the specimen was inferred from the initial unloading contact stiffness (S). The relationship among contact stiffness, contact area, and elastic modulus was derived as follows:

$$S = 2\beta E_r\sqrt{\frac{A}{\pi}} \quad (3)$$

where β is a constant that depends on the geometry of the indenter (β = 1.034 for a Berkovich indenter) and E, is the reduced elastic modulus, taking into account the elastic deformation of both the tested specimen and the indenter. For evaluating E,. the contact stiffness (S). and the contact area A could be determined accurately from load against displacement graph measured during the indentation process.

The specimen's elastic modulus (E_s) was then calculated as follows (Oliver and Pharr, 1992):

$$E_s = (1 - v_s^2)\left(\frac{1}{E_r} - \frac{1 - v_i^2}{E_i}\right)^{-1} \quad (4)$$

where v_s and v_i (0.07) are the Poisson's ratios of the specimen and indenter, respectively, whereas E, is the modulus of the diamond indenter (1141 GPa). In all calculations, v_s was assumed to be 0.25 for all crops.

2.3. Atomic force microscopy (AFM) scanning

After indentation, the epoxy resin blocks were glued to steel discs and mounted on the magnetic sample holder of the atomic force microscope (XE-100, PSIA Corp., Korea). The contact mode was used to scan the surface after nano-indentation. The operation parameters were set at a scanning rate of 0.5 Hz and a setpoint of 24.73 nN under ambient atmosphere. The topographies of the specimen surface with the indents were then obtained by AFM scanning. The surface of the specimens had a vertical range of 300–350 nm with root mean square surface roughness amplitude of 30–35 and an average roughness of 20–30nm. The average thickness of the cell walls of each crop stalk was estimated based on 20 cells by means of the XEI software associated with the AFM.

3. Results and discussion

3.1. Elastic modulus

The elastic modulus of cell walls in five crop stalks is shown in Fig. 1. Wheat straw has the highest elastic modulus of 20.8 GPa among the five crops. The rice straw and cassava stalk present values of 19.4 and 19.0 GPa. respectively. The soybean stalk and cotton stalk both exhibit the lowest values of 16.3 GPa among the five crop stalks. The mean value of five crop stalks was 18.4 GPa. The cell wall of the wheat straw was thicker (2.9 μm) than that of other crop stalks (1.1–2.6 μm), but thinner than that of most wood species. When the results of wheat straw were compared with soybean or cotton stalk, the percentage of difference was 21.6%. The different cell wall components (Rowell et al., 1997). structure, and microfibril angle could be important contributors to the difference. The holocellulose content in cotton stalk fiber was up to 75.1%, whereas that in wheat straw, rice straw and soybean stalk was 71.3%, 64.0%, and 51.5% (Yu, 1999). respectively. The contents of cellulose and lignin in wheat straw were 1.1% and 5.5% higher than those in rice straw (Liu and Yu, 2002). respectively. The microfibril angles were 19.4° for rice straw (Reddy and Yang, 2006). 17° for wheat straw (Reddy and Yang, 2007). 15° for cotton stalk (Reddy and Yang, 2009a) and 12° for soybean stalk (Reddy and Yang, 2009b), respectively. Those published data showed significant structural and chemical differences among the tested crops.

Table 1 lists the results of previous research on the nano-mechanical properties of 10 hardwoods (Wu et al., 2009). two softwoods (Gindl and Schöberl, 2004; Tze et al., 2007). and lyocell fibers (Lee et al., 2007). Compared with the reference data, the values of the elastic moduli of cell walls of crop stalks (16.3–20.8 GPa) were comparable to that of most of hardwood species (16.9–24.6 GPa) and were higher than those of some softwoods (15.0–18.0 GPa) and of lyocell fiber (11.5–13.2 GPa). The mean values of the elastic moduli in crop, hardwood, softwood, and lyocell fibers are shown in Fig. 2. The highest mean value of 20.2 GPa was observed in the case of hardwoods, followed by 18.4 GPa in crops, 16.2 GPa in softwoods, and 12.4 GPa in lyocell fiber. In general, crops contain the same basic chemical components as wood, but they vary in composition. Crops usually have lower cellulose content and higher hemicellulose and lignin content than wood. The ash content of crops is much higher than that of wood particles (Rowell et al., 1997). The content of ash in crop stalks can be up to 5% (Reddy and Yang. 2006, 2007, 2009a,b). Higher ash content results in higher stiffness and lower flexibility. All these may be major reasons for the differences in the elastic moduli of cell walls among crops, wood, and lyocell fibers.

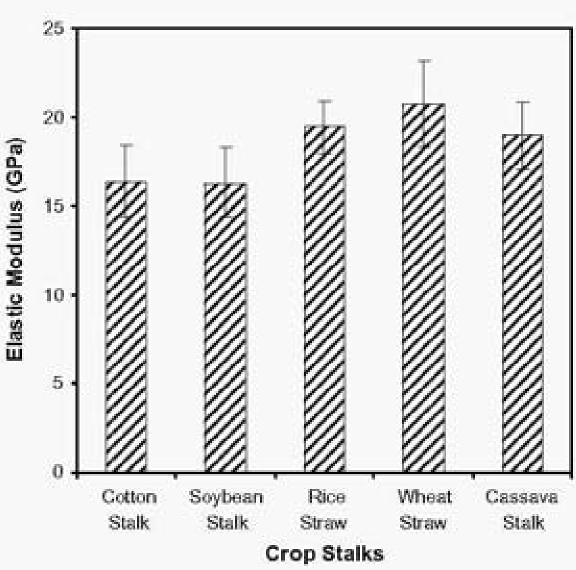

Fig. 1. Elastic modulus of cell wall in five crop stalks.

Table 1
Comparison of nano-mechanical properties of hardwoods, softwoods and lyocell fibers measured by nano-indentation.

Species		Elastic modulus (MPa)	Hardness (MPa)
Hardwood	Poplar	16.9 (11.1)	0.49 (9.54)
	Iroko	22.9 (10.8)	0.51 (7.67)
	Alder birch	18.5 (10.1)	0.49 (6.21)
	Manchurian ash	17.5 (11.8)	0.48 (10.1)
	Asian white birch	19.7 (5.69)	0.45 (7.31)
	Red oak	22.6 (6.39)	0.55 (6.83)
	White oak	19.5 (8.03)	0.49 (6.02)
	Mongolian oak	18.4 (10.7)	0.44 (10.6)
	Kwila	21.2 (6.92)	0.54 (4.03)
	Keranji	24.6 (8.14)	0.56 (5.62)
Softwood	Loblolly pine	14.2–17.6 (1–7)	0.43–0.53 (2–7)
	Spruce	15.0–18.0 (10–15)	0.34 (7)
Fiber	Lyocell	11.5–13.2 (7.0)	0.33–0.44 (4.5)

Note: The coefficient Ofvariation (CV, in bracket; units in percentage) represents the variability of the elastic modulus and hardness. Data for hardwood from Wu et al. (2009), data for Loblolly Pine from Tze et al. (2007), data for Spruce from Gindl et al. (2004), data for Lyocell fiber from Lee et al. (2007).

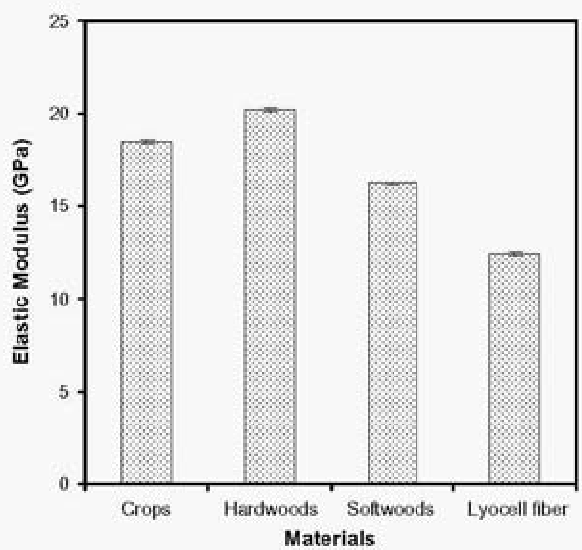

Fig. 2. Comparison of mean value of elastic modulus in different materials (Data for hardwood from Wu et al. (2009), data for softwood from Tze et al. (2007) and Gindl et al. (2004), data for Lyocell fiber from Lee et al. (2007)).

3.2. Hardness

Fig. 3 presents cell-wall hardness in five crop stalks. The cotton stalk cell walls showed the highest hardness, at 0.85 GPa. The lowest hardness, 0.48 GPa. was observed in soybean stalks. The five crops differed in hardness by up to 43.5%. Cotton stalk fiber had a higher degree of polymerization (DP) of 14,000 and a crystallization content up to 90% compared with the other crop fibers (Wang and Quan, 1996). The holocellulose in cotton stalk fiber was up to 75.1%. whereas that in wheat straw, rice straw and soybean stalk was 71.3%. 64.0%. and 51.5% (Yu. 1999). respectively. Higher DP content of crystallization. and holocellulose in cotton stalk all contributed to the higher hardness. The elastic modulus and hardness

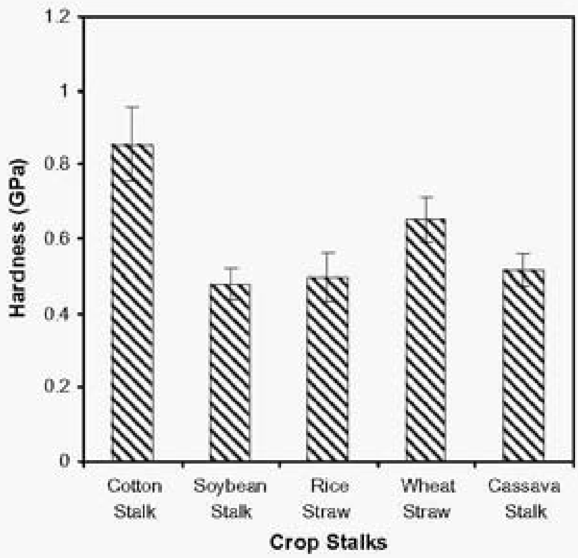

Fig. 3. Hardness of cell wall in five crop stalks.

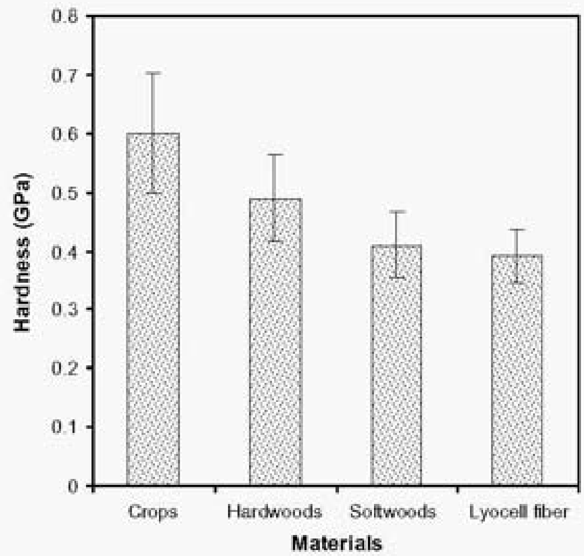

Fig. 4. Comparison of mean value of hardness in different materials (Data for hardwood from Wu et al. (2009), data for softwood from Tze et al. (2007) and Gindl et al. (2004), data for Lyocell fiber from Lee et al. (2007)).

Table 2
Nano-mechanical properties of crops stalk cell walls by nano-indentation.

Species		Elastic modulus (GPa)[b]	Hardness (GPa)[b]
Crops	Cotton stalk	16.3 (12.4)	0.85 (11.7)
	Cassava stalk	19.0 (10.0)	0.52 (8.26)
	Soybean stalk	16.3 (12.2)	0.48 (8.59)
	Rice straw	19.4 (7.69)	0.50 (13.2)
	Wheat straw	20.8 (11.6)	0.65 (9.19)
Mean		18.4 (10.8)	0.60 (10.2)
Percentage (%)[a]		21.6	43.5

[a] The percentage war the difference between maximum and minimum.
[b] The coefficient of variation (CV, in bracket; units in percentage) represents the variability of the elastic modulus and hardness.

of cell walls of wheat straw were higher than those of rice straw. The contents of cellulose and lignin in wheat straw were 1.1% and 5.5% higher, respectively, than those in rice straw (Liu and Yu, 2002). This agreed with the results of Gindl et al. (2002) that increasing lignin content might contribute to the higher measured hardness.

Table 2 and Fig. 3 list the nano-mechanical properties of hardwoods, softwoods, lyocell fibers, and crop stalks as measured by nano-indentation. In contrast to our expectation, the mean value of hardness in crop stalks was the highest one (0.60 GPa) compared with the lower value of 0.39 GPa in lyocell fiber (Fig. 4) and 0.49 GPa in hardwoods and 0.41 GPa in softwoods. The content of ash in crop stalks was 8 times higher than that of wood species (Chen and Li, 1999). which might be the main factor that contributes to higher hardness. Approximately 60% of the ash in crop stalks was SiO_2. which obviously played an important role in conferring higher hardness. The different degrees of orientation of the cellulose chains between crops and wood species may be another factor that can explain this discrepancy. The degree of orientation of cellulose chains was 40–60% in the crop fibers and 60–80% in the wood fibers (Wu. 1991). The higher degree of crystalline orientation in wood fibers results in less lateral bonding and makes the fiber prone to lateral splitting or fibrillation (Mortimer and Peguy, 1996). According to Gindl et al. (2006). the load was applied to the sample by nano-indentation not only along the fiber direction, but to a certain extent also along the transverse direction due to the geometry of the indenter pyramid. Hence, in this study, the hardness evaluated by nano-indentation is also subject to the effect of differences in the degree of lateral bonding of the fibers. Therefore, the higher measured hardness in the crop stalk fibers indicates that there is more lateral bonding in crop stalk fibers than in wood fibers.

4. Conclusions

It is difficult to fully characterize the mechanical properties of cell walls of crop stalk using conventional measurements due to the small cell wall thickness. However, nano-indentation can be used to investigate the nano-mechanical properties of cell walls of crop stalk at nanoscale. The elastic modulus determined using nano-indentation was in the range of 16.3–20.8 GPa and the hardness was in the range of 0.48–0.85 GPa in the cell walls of the five studied crop stalks. This indicates that crop stalk fibers have relatively better nano-mechanical properties than wood or lyocell fibers.

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References

Albano, C., Ichazo, M., González, J., Delgado, M., Poleo, R., 2001. Effects of filler treatments on the mechanical and morphological behavior of PP + wood flour and PP + sisal fiber. Mater. Res. Innovat. 4, 284–293.
Chen, H., Li, Z., 1999. Research on the method of wheat straw steaming exploration. Cellulose Sci. Technol. 7 (4), 14–22.
Eichhorn, S.J., Baillie, C.A., Zaferiropoulos, N., Mwaikambo, L.Y., Ansell, M.P., Dufresne, A., Entwistle, K.M., Herrera-Franco, P.J., Ercamilla, G.C., Groom, L., Hughes, M., Hill, C., Rials, T.G., Wild, P.M., 2001. Review: current international research into cellulosic fibers and composites. J. Mater. Sci. 36, 2107–2131.

Gindl, W., Gupta, H.S., Grünwald, C., 2002. Lignification of spruce tracheid secondary cell walls related to longitudinal hardness and modulus of elasticity using nano-indentation. Can. J. Bot. 80, 1029–1033.

Gindl, W., Schöberl, T., 2004. The significance of the elastic modulus of wood cell walls obtained from nanoindentation mearurements. Compos.: Part A 35, 1345–1349.

Gindl, W., Gupta, H.S., Schöberl, T., Lichtenegger, H.C., Fratzl, P., 2004 Mechanical properties of spruce wood cell walls by nanoindentation. Appl. Phys. A 79, 2069-2073.

Gindl, W., Konnerth, J., Schöberl, T., 2006. Nanoindentation of regenerated cellulose fibres. Cellulose 13, 1–7.

Helbert, W., Cavaillé, J.Y., Dufresne, A., 1996. Thermoplastic nanocomposites filled with wheat straw cellulose whiskers. Part I: processing and mechanical behavior. Polym. Comp. 17 (4), 604–611.

Ichazo, M.N., Albano, C., González, J., Perera, R., Candal, M.V., 2001. Polypropylene/wood flour composites: treatments and properties. Comp. Struct. 54, 207–214.

Kersavage, P.C., 1973. A System for Automatically Recording the Load-Elongation characteristics of single Wood Fibers under Controlled Relative Humidity Conditions. USDA, US Government Printing Office, Washington, DC, USA. 46pp.

Lee, S.H., Wang, S., Pharr, G.M., Kant, M., Penumadu, D., 2007, Mechanical properties and creep behavior of lyocell fibers by nanoindentation and nano-tensile testing. Holzforschung 61, 254-260.

Liu, H., Yu, Z., 2002 Properties of straw fiber. J. Donghua Univer. 28 (2), 124-129.

Liu, R., Yu, H., Huang, Y., 2005. Structure and morphology of cellulose in wheat straw. Cellulose 12, 25–34.

Mahlberg, R., Paajanen, L., Nurmi, A., Kivistö, A., Koskela, K., Rowell, R.M., 2001. Effect of chemical modification of wood on the mechanical and adhesion properties of wood fiber/polypropylene fiber and polypropylene/veneer composites. Holz als Roh und Werkstoff 59,319–326.

McKendry, P., 2002. Energy production from biomass (part 1): overview of biomass. Bioresour. Technol. 83,37–46.

Mo, X., Wang, D., Sun, X., 2005. Straw-bared biomass and biocompoistes. In: Mohanty, A.K., Misra, M., Drzal. L.T. (Eds.), Natural Fibers, Biopolymers, and Biocomposites. CRC Press, Boca Raton, pp. 473–495.

Mortimer, S.A., Peguy, A.A., 1996. Methods for reducing the tendency of lyocell fibers to fibrillate. J. Appl. Polym. Sci. 60, 305–316.

Mott, L., Shaler, S., Groom, L.H., Liang, B., 1995. The tensile testing of individual wood fibers using environmental scanning electron microscopy and video image analysis. Tappi 78, 143–148.

Oliver, W.C., Pharr, G.M., 1992. An improved technique for determining hardness and elastic modulus using load and displacement sensing indentation experiments. J. Mater. Res. 7, 1564–1583.

Reddy, N., Yang, Y., 2006. Properties of high-quality long natural cellulose fibers from rice straw. J. Agric. Food Chem. 54, 8077–8081.

Reddy, N., Yang, Y., 2007. Preparation and characterization of long natural cellulose fibers from wheat straw. J. Agric. Food Chem. 55, 8570–8575.

Reddy, N., Yang, Y., 2009a. Properties and potential applications of natural cellulose fibers from the bark of cotton stalks. Bioresour. Technol. 100,3563–3569.

Reddy, N., Yang, Y., 2009b. Natural cellulose fibers from soybean straw. Bioresour. Technol. 100, 3593–3598.

Rowell, R.M.. Young. R.A., Rowell, J.K., 1997. Paper and Composites from Agro-Based Resources. CRC Lewis Publishers, Boca Raton, FL, USA p. 446.

Spurr, A.R., 1969. A low-viscosity epoxy resin embedding medium for electron microscopy. J. Ultrastruct. Res. 26, 31–43.

Sun. Y.. Cheng, JY., 2002. Hydrolysis of lignocellulosic materials for ethanol production: a review. Bioresour. Technol. 83 (1), 1–11.

Tze, W.T., Wang, S., Rial, T.G., Pharr, G.M., Kelley, S.S., 2007. Nanoindentation of wood cell wall: continuous stiffness and hardness measurement. Comp. A – Appl. Sci. Manufact. 38, 945–953.

Wang, P., Quan, J., 1996. Configuration and Structure of Plant Fibers. Nanjing Forestry University, Nanjing. pp. 5.

Wang, S., Lee, S.H., Tze, W.T.Y., Rials, T., Pharr, G.M., 2006. Nanoindentation as a tool for understanding nano-mechanical properties of cell wall and biocomposites. In: International Conference on Nanotechnology for the Forest Products Industry, Marriott Marquis, Atlanta, GA, 26–28 April, 7pp.

Wu, Y., 1991. Chemistry of Plant Fibers. Beijing light industry publishing company, Beijing, China.

Wu, Y., Wang, S., Zhou, D.. Xing, C., Zhang, Y., Pharr. G.M., 2009. The use of nanoindentation and silviscan to determine the mechanical properties of ten hardwood species. Wood Fiber Sci. 41 (1), 64–73.

Xing, C., Wang, S., George, M.P., Groom, L.H., 2008. Effect of thermo-mechanical refining pressure on the properties of wood fibers. Holzforschung 62, 230–236.

Yin, S., Wang, S., Rials, T.G., Kit, K.M., Hansen, G.M., 2007. Polypropylene composites filled with steam-exploded wood fibers from beetle-killed loblolly pine by compression-molding. Wood Fiber Sci. 39 (1), 95–108.

Yu, J., 1999. Research on the utilization of wheat straw resources. Resour. Saving Utilizat. 4, 31–35.