Variability in the Mechanical Properties of Cell Walls of *Bambusa arundinacea* (Retz.) Willd. Based on Nanoindentation Method

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Bambusa arundinacea (Retz.) Willd. is a large and thorny bamboo species used for construction and scaffolding purposes. Bamboo fibers, which are an important part of bamboo, give bamboo its excellent mechanical properties. The goal of this work was to get more information about fiber in order to learn more about bamboo material. It was tested for different ages, heights, and radial positions using the nanoindentation method. The results showed that the mean values of indentation modulus of elasticity and hardness of this species were 20.79 GPa and 497.01 MPa, respectively, and that the mechanical properties of the fiber cell walls were less affected by the three factors and were generally consistent. In regards to the age factor, it had little effect on the indentation modulus of elasticity, while the hardness increased slightly with age. The indentation modulus of elasticity and hardness tended to increase with increasing height. The fiber strength was relatively stable in different parts of the radial direction.

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Keywords: Bambusa arundinacea (Retz.) Willd; Nanoindentation; Fiber; Indentation modulus of elasticity; Hardness

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INTRODUCTION

Bamboo is an extremely fast-growing and ecologically valuable material, and importantly, its mechanical properties can reach a stable state in a short period of time (Zhang *et al.* 2020). It is widely used as a green building material, with direct bamboo culm utilization for bamboo buildings, bamboo scaffolding, *etc.*, and is also processed into bamboo flooring and bamboo furniture (Chung and Yu 2002; Lee *et al.* 2012; Hong *et al.* 2019; Zheng and Zhu 2021). Bamboo, as a typical anisotropic material, has a tissue structure and other factors that make its mechanical properties exhibit significant differences both radially and longitudinally (Wang and Shao 2020; Liu *et al.* 2022). In order to further grasp the complex mechanical behavior of bamboo materials and to optimize the design of bamboo fiber-based composites, more information on fiber mechanics is needed (Yu *et al.* 2011).

Fibers as a reinforcing phase are distributed in a gradient structure in the basic tissue, resulting in the distinctive macroscopic properties of bamboo. Bamboo fibers are also widely used in the textile and paper industries as well as in advanced engineering materials (Wang *et al.* 2010; Guan *et al.* 2019; Lin *et al.* 2022). The excellent mechanical properties of bamboo originate from bamboo fibers. Through gaining a deeper understanding of fiber, a more thorough grasp of the properties of bamboo can be obtained (Hao *et al.* 2018). Fibers have thick cell walls. Characterization of the mechanical properties of small-sized cell walls is difficult to perform using conventional mechanical testing instruments. The birth of the nanoindentation technique overcame this difficulty and pioneered the study of the mechanical properties of cell walls, adding to the study of the properties of wood (Wimmer *et al.* 1997). Since then, the technique has become increasingly sophisticated and has been applied to the study of the fibers within the cell walls of bamboo (Dixon *et al.* 2015). The nanoindentation technique involves pressing a very fine diamond indenter needle into the surface of the object to be measured, continuously collecting the load during loading and unloading as well as the depth of indentation, and finally obtaining the hardness and indentation modulus of the sample to be measured through a specific theoretical model. Since the radius of curvature of the indenter is generally less than 100 nm, much smaller than the thickness of the bamboo fiber cell wall, it is well suited for the study of cell wall properties. *Bambusa arundinacea* (Retz.) Willd. is a large and spiny bamboo with poles 10 to 17 m high, 6 to 9 cm in diameter, 10 to 23 cm long between nodes, and 1 to 1.5 cm thick. It has a straight, thick, and solid culm that can be used for construction and scaffolding. This species is native to India and is now cultivated in Guangzhou and Hong Kong, China. Previous studies on the root distribution, phytohormones, and medicinal value of *Bambusa arundinacea* (Retz.) Willd. have been published (Joshi and Nadgauda 1997; Divakara *et al.* 2001; Kumar and Divakara 2001; Venkatachalam *et al.* 2015; Patil *et al.* 2023). As a construction material, there is little research on its mechanical properties. Bamboo is a biomass material with natural functional gradient (Habibi *et al*. 2015), which is anisotropic and heterogeneity in nature, resulting in macroscopic mechanical properties that exhibit variability and anisotropy (Ahmad and Kamke 2005; Hu *et al*. 2018). This work addresses whether there is variability in the fibers of different parts. Therefore, this study investigated the mechanical properties of individual fibers within the bamboo cell wall in terms of age, radial, and axial direction.

EXPERIMENTAL

Materials

The vascular bundle type of *Bambusa arundinacea* (Retz.) Willd. is mainly broken-waist type with sporadic distribution of double broken-waist type (Fig. 1). Samples were collected from the Hua’an Bamboo Garden in Zhangzhou City, Fujian Province, China, a region with a subtropical monsoon climate (Fig. 2a). Randomly selected healthy two-, three-, and four-year-old normal-growth bamboos were felled, and sections at heights of 1.5 m, 7.0 m, and 12.5 m were intercepted for the base, middle, and top specimens (Fig. 2b), respectively. The middle wall of a 1.5 m high bamboo section of two-, three-, and four-year-old bamboos was used to study age variation. Different wall layers along the radial section of the 1.5 m high bamboo segment of three-year-old bamboo were used to study radial variation. The middle walls of bamboo sections at 1.5 m, 7.0 m, and 12.5 m height of three-year-old bamboo were used to study the longitudinal variation.

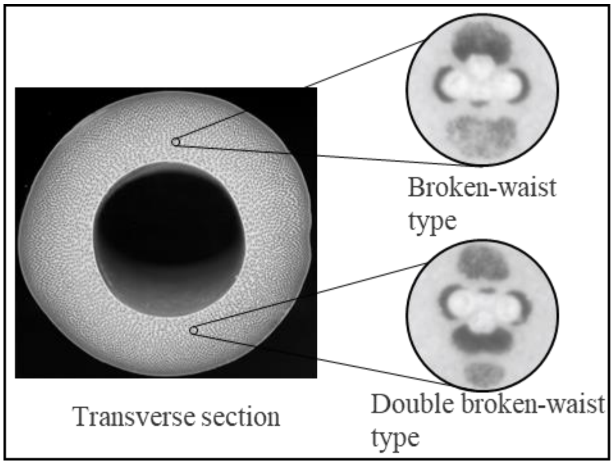

Fig. 1. Transverse section and vascular bundle types of *Bambusa arundinacea* (Retz.) Willd.

Nanoindentation Test

The bamboo section with the fibers to be tested was first prepared into a rectangle, and the fibers to be tested were located in the center of the rectangle end face (Fig. 2c). Then, the end face of the rectangle was prepared into a pyramid shape using a slicer, and the tip of the tower was the fiber to be tested (Fig. 2d). The top was polished with a diamond blade to produce a nanoindentation test sample.

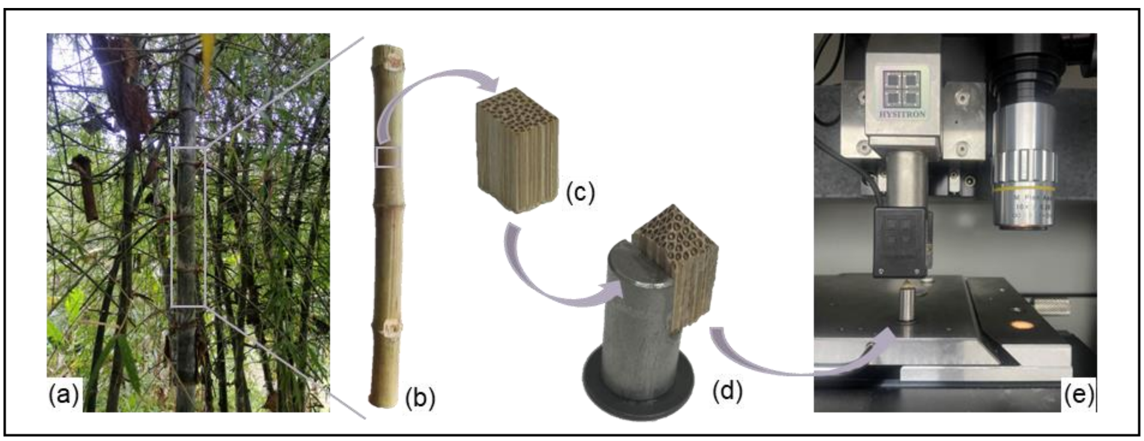

Fig. 2. a: Sample collection, preparation, and testing *Bambusa arundinacea* (Retz.) Willd. in living condition; b: Cut off bamboo segments; c: Rectangular specimens containing fibers of interest; d: Pyramid-shaped specimens on an iron stand; e: Testing using nanoindentation equipment

The prepared samples were equilibrated in a sealed chamber for more than 24 h at a temperature of 23 ± 5 °C and a relative humidity of 40 ± 0.5% (Li *et al.* 2020). The test apparatus was a Triboindenter (Hysitron, Minneapolis, MN, USA) with a Berkovich diamond tip (diameter of the tip ≤ 100 nm) (Fig. 2e). A three-stage segmental application of load to the fiber of interest in force control mode was selected. The set nanoindentation test procedure is shown in Fig. 3, first loading at 50 $\mu N \cdot s^{-1}$, loading for 5 s to reach a maximum load of 250 μN, and the maximum load was held for 6 s. The holding load was for viscoelastic materials to avoid experimental errors due to creep; finally, after a 3 s unloading segment, and collecting data from the unloading segment for computational analysis. The indentation modulus of elasticity and hardness of the samples were calculated using the equations of Oliver and Pharr (1992), based on specific data and procedures described by Eder *et al.* (2013) and Yan *et al.* (2011),

$$1 / E_r = (1 - v^2) / E + (1 - v_i^2) / E_i \quad (1)$$

$$H = P / A_c \quad (2)$$

where H is hardness; P is peak load; A_c is the projected area at peak load; E_r is the composite response modulus, which can be calculated based on the load-displacement curve and elastic contact theory; E_i and v_i are the elastic modulus and Poisson ratio of the tips, respectively. For diamond tips, E_i is 1141 GPa, and v_i is 0.07. E and v are, respectively, the indentation modulus of elasticity and Poisson ratio of samples, where the Poisson ratio of samples is 0.22 (Yu *et al.* 2011; Yuan *et al.* 2021).

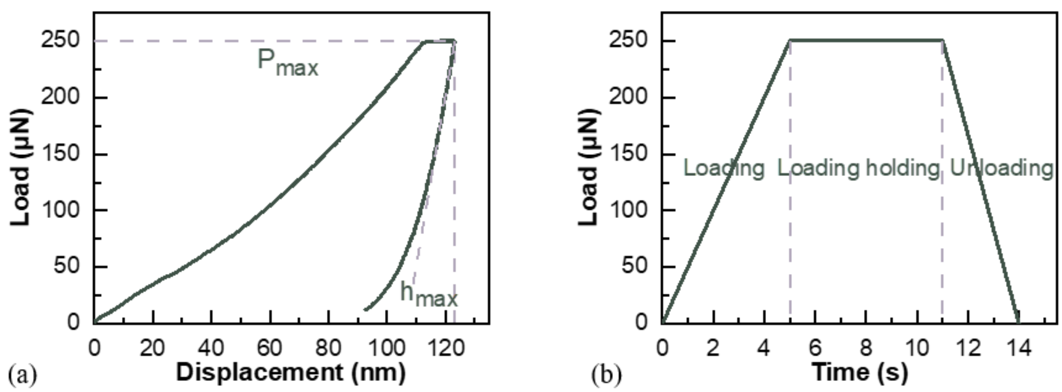

Fig. 3. a: Load-Time control schematic (loading section, loading holding section, and unloading section in that order); b: Typical force-displacement curves (corresponding in turn to the three loading processes in Fig. 3.a)

RESULTS AND DISCUSSION

Strength of Fibers of Different Ages

Figure 4 illustrates the strength of the fibers in the middle of the bamboo wall of 1.5-m-high sections of two-, three-, and four-year-old bamboos. Overall, the indentation modulus of elasticity of the fibers of the different ages of the bamboos did not differ greatly in value at around 20.5 GPa. The indentation modulus of elasticity of the four-year-old fibers was the highest, with a mean value of 21.4 GPa. The stiffness of the fiber cell wall showed a small increase with age. The hardness of two-year-old bamboo fibers was the smallest at 441 MPa, while the hardness of four-year-old bamboo fibers increased to 536 MPa. It has been shown that for wood science, microfibril angle, cellulose rate, and density are considered to be the main determinants of mechanical properties (Evans and Elic 2001). The cell wall density and cellulose proportional content of mature wood fibers are almost the same, so the microfibril angle is the most important factor affecting the properties for mature wood fibers (Evans and Elic 2001). There is a negative correlation between the microfibril angle and two mechanical properties of the cell wall, which are the indentation modulus of elasticity and hardness. That is, the indentation modulus of elasticity and hardness decreases with increasing microfibril angle (Tian *et al.* 2010). The difference in microfibril angle between bamboo fibers of different ages was not significant (Liu *et al.* 2014). Therefore, the age factor had little effect on fiber cell wall strength.

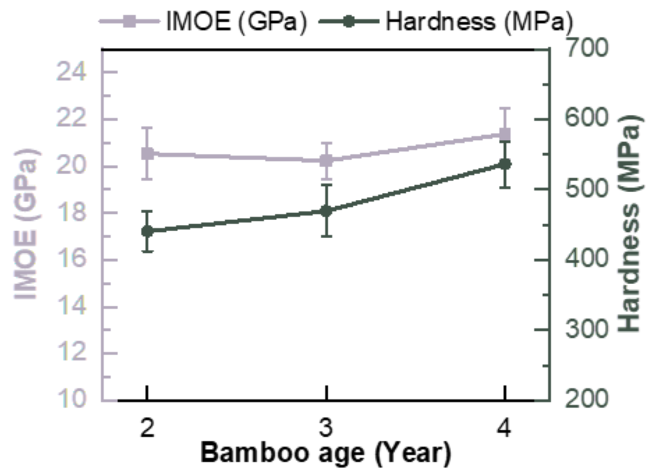

Fig. 4. Indentation modulus of elasticity (IMOE) and hardness of cell walls of fibers of different ages

Variability of the Strength of the Fibers in the Height Direction

The nanoindentation data were analyzed for the middle part of the three-year-old bamboo wall at different heights (Fig. 5). The indentation modulus of elasticity and hardness increased with height in a relatively smooth trend. The indentation modulus of elasticity and hardness of the top fiber cell wall were 21.9 GPa and 550 MPa, respectively, which were slightly higher than the strength of the other two parts. The age factor had little effect on the microfibril angle, but in the height direction, the average microfibril angle decreased with increasing height of bamboo culms, which may make the cell wall strength increase with increasing height (Zhang *et al.* 2020).

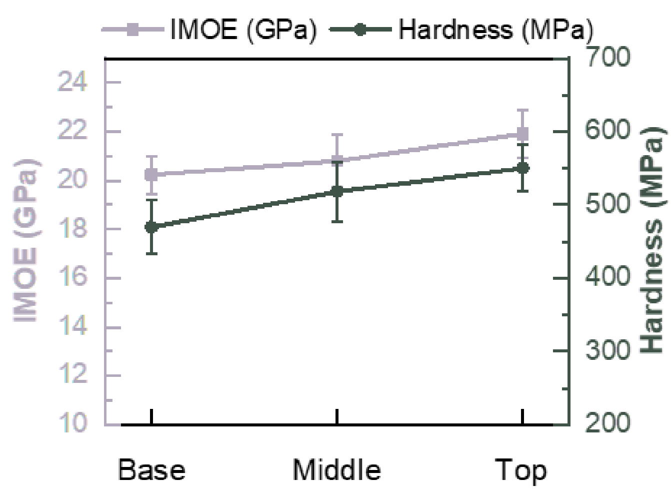

Fig. 5. Variability of indentation modulus of elasticity and hardness of cell walls of fibers in the height direction

Variability of the Strength of Fibers in the Radial Direction of the Bamboo Wall

Figure 6 clearly shows the trend of the fiber cell wall strength from the outer skin to the inner skin. Both the indentation modulus of elasticity and the hardness were almost unchanged, with only small fluctuations that did not produce significant differences. In general, the cell wall strength was stable from the outside to the inside of the bamboo wall, with 95% confidence intervals for the mean values of the indentation modulus of elasticity ranging from 20.2 to 20.5 GPa. Similarly, 95% confidence intervals for the mean values of the hardness ranged from 464.7 to 477.5 MPa. The strength of the fiber cell wall remained almost constant along the radial direction of the bamboo wall, probably because the microfibril angle along the radial direction was almost constant (Yu *et al*. 2007).

The mechanical properties of the cell walls of the fibres of *Bambusa arundinacea* (Retz.) Willd. were found to be somewhat lower than those of the more widely used fibres of moso bamboo (21.69 GPa and 0.61 GPa in compressive modulus of elasticity and hardness, respectively) (Huang and Fei 2017). However, *Dendrocalamus farinosus*, which is also a clumping bamboo, has an indentation modulus and hardness of only 18.56 GPa and 410.72 MPa respectively, which are lower than those of *Bambusa arundinacea* (Retz.) Willd. (Yang *et al*. 2014). This shows that from the point of view of the mechanical properties of the cell wall, the fibres of *Bambusa arundinacea* (Retz.) Willd. have great potential for industrial use.

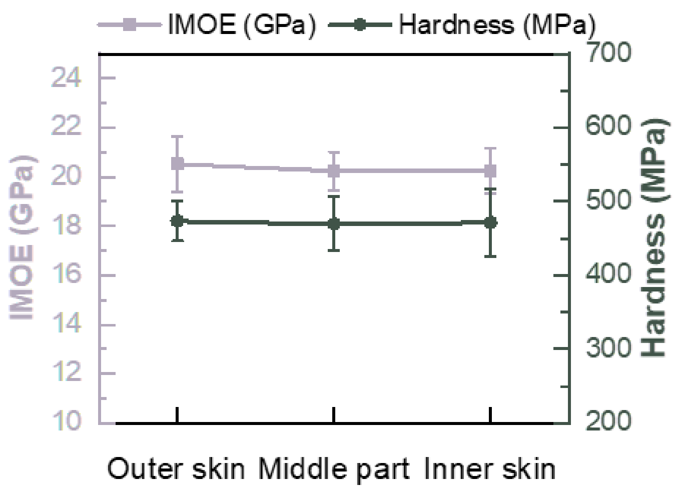

Fig. 6. Variability of indentation modulus of elasticity and hardness of cell walls of fibers in the radial direction of the bamboo wall

CONCLUSIONS

1. The mechanical properties of fibrous cell walls of Indian cambium were tested by nanoindentation method at different ages, heights, and radially superior sites. The mean values of indentation modulus and hardness for all specimens of *Bambusa arundinacea* (Retz.) Willd. were 20.8 GPa and 497 MPa, respectively, and the results indicated that the three factors studied had little effect on the mechanical properties of the fiber cell walls.

2. The age factor had little effect on the indentation modulus of elasticity, while the hardness increased slightly with age. The indentation modulus and hardness tended to increase with increasing height. The strength of the fibers was relatively stable in different parts of the radial direction.

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Author Contributions Statement

F. D. wrote the main manuscript, Z. W. was responsible for data curation, L. Y. performed formal analysis, T. Z. supplied the resources, H. W. came up with the methodology, G. T. was responsible for funding acquisition, methodology, and project administration. All authors reviewed the manuscript.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data Availability Statements

The datasets generated during and/or analysed during the current study are available from the corresponding author on reasonable request.

REFERENCES CITED

Ahmad, M., and Kamke, F. A. (2005). "Analysis of Calcutta bamboo for structural composite materials: Physical and mechanical properties," *Wood Sci. Technol*. 39, 448-459. DOI: 10.1007/s00226-005-0016-y

Chung, K. F., and Yu, W. K. (2002). "Mechanical properties of structural bamboo for bamboo scaffoldings," *Eng. Struct.* 28(2), 429-442. DOI: 10.1016/S0141-0296(01)00110-9

Divakara, B. N., Kumar, B. M., Balachandran, P. V., and Kamalam, N. V. (2001). "Bamboo hedgerow systems in Kerala, India: Root distribution and competition with trees for phosphorus," *Agroforest. Syst.* 51, 189-200. DOI: 10.1023/A:1010730314507

Dixon, P. G., Ahvenainen, P., Aijazi, A. N., Chen, S. H., Lin, S., Augusciak, P. K., Borrega, M., Svedström, K., and Gibson, L. J. (2015). "Comparison of the structure and flexural properties of Moso, Guadua and Tre Gai bamboo," *Constr. Build. Mater*. 90, 11-17. DOI: 10.1016/j.conbuildmat.2015.04.042

Eder, M., Arnould, O., Dunlop, J. W. C., Hornatowska, J., and Salmén, L. (2013). "Experimental micromechanical characterisation of wood cell walls," *Wood Sci. Technol.* 47, 163-182. DOI: 10.1007/s00226-012-0515-6

Evans, R., and Elic, J. (2001). "Rapid prediction of wood stiffness from microfibril angle and density," *Forest Prod. J.* 51(3), 53-57.

Guan, M., An, X. Y., and Liu, H. B. (2019). "Cellulose nanofiber (CNF) as a versatile filler for the preparation of bamboo pulp based tissue paper handsheets," *Cellulose* 26, 2613-2624. DOI: 10.1007/s10570-018-2212-6

Habibi, M., Samaei, A., and Gheshlaghi. (2015). "Asymmetric flexural behavior from bamboo's functionally graded hierarchical structure: underlying mechanisms," *Acta Biomater* 16, 178-186. DOI: 10.1016/j.actbio.2015.01.038

Hao, H. l., Tam, L.-h., Lu, Y., and Lau, D. (2018). "An atomistic study on the mechanical behavior of bamboo cell wall constituents," *Compos. Part B: Eng.* 151, 222-231. DOI: 10.1016/j.compositesb.2018.05.046

Hong, C. K., Li, H. T., Lorenzo, R., Wu, G., Corbi, I., Corbi, O., Xiong, Z. H., Yang, D., and Zhang, H. Z. (2019). "Review on connections for original bamboo structures," *J. Renew. Mater.* 7(8), 713-730. DOI: 10.32604/jrm.2019.07647

Huang, Y. H., and Fei, B. H. (2017). "Comparison of the mechanical characteristics of fibers and cell walls from moso bamboo and wood," *BioResources* 12(4), 8230-8239. DOI: 10.15376/biores.12.4.8230-8239

Hu, D., Song, B., Dang, L., and Zhang, Z. (2018). "Effect of strain rate on mechanical properties of the bamboo material under quasi-static and dynamic loading condition," *Compos. Struct.* 200, 635-646. DOI: 10.1016/j.compstruct.2018.05.107

Joshi, M. and Nadgauda, R. S. (1997). "Cytokinins and in vitro induction of flowering in bamboo: *Bambusa arundinacea* (Retz.) Willd," *Curr. Sci. India* 73(6), 523-526. https://www.jstor.org/stable/24100161

Kumar, B. M., and Divakara, B. (2001). "Proximity, clump size and root distribution pattern in bamboo: A case study of *Bambusa arundinacea* (Retz.) Willd., Poaceae, in the Ultisols of Kerala, India," *Journal of Bamboo and Rattan* 1(1), 43-58. DOI: 10.1163/156915901753313605

Lee, C. H., Chung, M. J., Lin, C. H., and Yang, T. H. (2012). "Effects of layered structure on the physical and mechanical properties of laminated moso bamboo (*Phyllostachys edulis*) flooring," *Constr. Build. Mater.* 28(1), 31-35. DOI: 10.1016/j.conbuildmat.2011.08.038

Li, J., Yu, Y., Feng, C., and Wang, H. K. (2020). "Mechanical characterization of *Pinus massoniana* cell walls infected by blue-stain fungi using in situ nanoindentation," *J. Forestry Res.* 31, 661-665. DOI: 10.1007/s11676-018-0848-6

Lin, Q. Q., Jiang, P., Ren, S. H., Liu, S. Q., Ji, Y. H., Huang, Y. X., Yu, W. J., Fontaine, G., and Bourbigot, S. (2022). "Advanced functional materials based on bamboo cellulose fibers with different crystal structures," *Manuf.* 154, article 106758. DOI: 10.1016/j.compositesa.2021.106758

Liu, P. C., Qin, Y. Y., Zhou, Q. S., Xiang, P., Zhou, X. H., and Shan, Y. (2022). "Experimental investigation on the physical and mechanical properties of *P. edulis* bamboo and their correlations," *Eur. J. Wood Wood Prod.* 80(05), 1-16. DOI: 10.1007/s00107-021-01773-4

Liu, X. E., Yang, X., Yang, S. M., Tian, G. L., and Shang, L. L. (2014). "Variation of microfibril angle in *Dendrocalamus farinosus* analyzed based on X-ray diffraction spectrum and its effect on tensile properties," *Spectrosc. Spect. Anal.* 34(6), 1698-1701. DOI: 10.3964/j.issn.1000-0593(2014)06-1698-04

Oliver, W. C., and 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. DOI: 10.1557/JMR.1992.1564

Patil, S., Singh, D., and Baghel, A. (2023). "Pharmacognosy, physicochemistry, phytochemistry, pharmacological and ethnomedicinal profiles of *Bambusa arundinacea* (Retz.) Willd. seeds: A scoping review," *Plant Sci. Today*. DOI: 10.14719/pst.2031

Tian, G. L., Wang, H. K., Yu, Y., and Yu, Y. S. (2010). "Study the effect of MFA to the elastic modulus and hardness of wood cell wall," *Nanosci Nanotechnol* 7(2), 63-33.

Venkatachalam, P., Kalaiarasi, K., and Sreeramanan, S. (2015). "Influence of plant growth regulators (PGRs) and various additives on in vitro plant propagation of Bambusa arundinacea (Retz.) Wild: A recalcitrant bamboo species," *Journal of Genetic Engineering and Biotechnology* 13(2), 193-200. DOI: 10.1016/j.jgeb.2015.09.006

Wang, F. L., and Shao, Z. P. (2020). "Study on the variation law of bamboo fibers' tensile properties and the organization structure on the radial direction of bamboo stem," *Ind. Crop Prod.* 152, article 112521. DOI: 10.1016/j.indcrop.2020.112521

Wang, Y. P., Wang, G., Cheng, H. T., Tian, G. L., Liu, Z., Xiao, Q. F., Zhou, X. Q., Han, X. J., and Gao, X. S. (2010). "Structures of bamboo fiber for textiles," *Text. Res. J.* 80(4), 334-343. DOI: 10.1177/0040517509337633

Wimmer, R., Lucas, B. N., Oliver, W. C., and Tsui, T. Y. (1997). "Longitudinal hardness and Young's modulus of spruce tracheid secondary walls using nanoindentation technique," *Wood Sci. Technol.* 31, 131-141. DOI: 10.1007/BF00705928

Yang, X., Tian, G. L., Shang, L. L., Lv, H. F., Yang, S. M., and Liu, X. E. (2014). "Variation in the cell wall mechanical properties of *Dendrocalamus farinosus* bamboo by nanoindentation," *BioResources* 9(2), 2289-2298. DOI: 10.15376/biores.11.1.1609-1619

Yu, Y., Fei, B. H., Zhang, B., and Yu, X. (2007). "Cell-wall mechanical properties of bamboo investigated by in-situ imaging nanoindentation," *Wood and Fiber Science: Journal of the Society of Wood Science and Technology* 39(4), 527-535. DOI: 10.1007/s11295-007-0082-4

Yu, Y., Tian, G. L., Wang, H. K., Fei, B. H., and Wang, G. (2011). "Mechanical characterization of single bamboo fibers with nanoindentation and microtensile technique," *Holzforschung* 65(1), 113-119. DOI: 10.1515/hf.2011.009

Yuan, T. C., Wang, Z. S., Han, X., Yuan, Z. R., Wang, X. Z., and Li, Y. J. (2021). "Multi-scale evaluation of the effect of saturated steam on the micromechanical properties of Moso bamboo," *Holzforschung* 75(11), 1052-1060. DOI: 10.1515/hf-2020-0266

Zhang, B., Guo, Y. H., Liu, X. E., Chen, H. Y., Yang, S. M., and Wang, Y. G. (2020). "Mechanical properties of the fiber cell wall in *Bambusa pervariabilis* bamboo and analyses of their influencing factors," *BioResources* 15(3), 5316-5327. DOI: 10.15376/biores.15.3.5316-5327

Zheng, Y. W., and Zhu, J. G. (2021). "The application of bamboo weaving in modern furniture," *BioResources* 16(3), 5024-5035. DOI: 10.15376/biores.16.3.5024-5035

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