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Comparative studies on wood structure and microtensile properties between compression and opposite wood fibers of Chinese fir plantation

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Abstract

The microtensile properties of mechanically isolated compression wood (CW) and opposite wood (OW) tracheids of Chinese fir (*Cunninghamia lanceolata*) were investigated and discussed with respect to their structure. Major differences in the tensile modulus and ultimate tensile stress were found between CW and OW fibers. Compared to OW, CW showed a larger cellulose microfibril angle, less cellulose content and probably more pits, resulting in lower tensile properties. These findings contribute to a further understanding of the structural–mechanical relationships of Chinese fir wood at the cell and cell wall level, and provide a scientific basis for better utilization of plantation softwood.

Keywords: Chinese fir, Tracheids, Mechanical properties, Compression wood, Opposite wood, Cellulose content, Microfibril angle

Introduction

Chinese fir (*Cunninghamia lanceolata*) is one of the main commercial plantation conifer tree species in China. Conifers consist mainly of axial tracheids, serving for support and for transport of water and dissolved minerals. Their anatomical structure, chemical composition and the spatial arrangement of polymers in the wood cell wall determines their mechanical properties. Thus, information about the micromechanics of single tracheids can be helpful to deepen our understanding of the structural–mechanical relationships of wood at the cell and cell wall level.

Considerable differences in the micro- and ultrastructure of wood tracheids have been found between compression wood (CW), opposite wood (OW), normal wood or juvenile wood [1–4]. Compared to normal

wood, CW shows the largest differences: an absence of S_3 layer of the shorter tracheids, with helical cavities and a large cellulose microfibril angle (MFA) in an S_2 layer of the cell wall. In terms of the chemical composition, the lignin content is higher and the cellulose content is lower with a lower degree of crystallinity [1, 2, 5–8]. It is well known that the mechanical properties of plant fibers depend on the anatomical structure [9], the chemical composition [10] and the MFA [11]. Plant fibers with a higher MFA have a smaller modulus of elasticity, higher flexibility but also a reduced ultimate tensile stress compared to fibers with lower MFAs [11–14]. The MFA is probably the most important structural parameter on the cell wall scale influencing the mechanical performance of plant fibers in their axial direction. At the level of the cell, pits of various forms and structural defects effect the tensile properties [15, 16] as well as cell wall thicknesses and cell geometries [9, 11]. Observed differences in the ultimate tensile stress between earlywood, transition wood and latewood with very similar MFAs could be related to

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tension buckling which primarily occurs in thin-walled earlywood fibers [13].

Tensile tests on adult single spruce fibers combined with Raman microscopy have revealed wavenumber shifts of peaks assigned to cellulose; while no wavenumber shifts for lignin-assigned peaks were detected upon mechanical loading [17]. In other words, the cellulose molecules take up the main load in these fibers with presumably small MFA and are responsible for the mechanical strength and elasticity under static stretching [18, 19]. In a recent study both normal wood (small MFA) and compression wood slices (large MFA) were subjected to static and dynamic loading conditions while the molecular deformations were studied by polarized Fourier transform infrared spectroscopy. Static loading showed molecular deformation of cellulose only, while lignin remained without any molecular deformation [19]. However, in dynamic experiments changes in lignin were observed as well as differences in lignin of compression and normal wood. Furthermore, the authors found that molecular deformations of cellulose were larger in normal wood due to the smaller MFA [19]. In general, it is difficult to study the role of lignin during tensile loading, because changes in lignin content typically occur together with changes in MFA. Recently, Özparpucu et al. [20] found on genetically modified poplar slices with similar MFAs but different lignin contents, that lignin contributed to the axial stiffness by increasing the shear stiffness of the cell wall matrix; and the influence of lignin content on axial stiffness may gradually increase as a function of the MFA. In addition to these studies, effects of chemical treatments on plant fiber mechanics were studied experimentally [10, 21]. Back to the single-fiber level, Zhang et al. [10] investigated the effects of chemical components on the tensile strength and modulus of single fibers from Chinese fir. They found that chemical treatments reduced tensile strength and modulus, except for the tensile strength of completely delignified wood fibers; the removal of hemicellulose caused more effects on the mechanical properties of cell walls than the lignin removal. Similar results exist for single bamboo fibers [21].

While datasets about native normal wood and severe compression wood fibers exist, to the best of our knowledge nothing is known about the tensile properties of CW and OW fibers despite their frequent occurrence in stems. The objective of this present study was to investigate the mechanical properties of single fibers in CW and OW from Chinese fir stem wood in microtensile tests at laboratory conditions (25 °C and 50% relative humidity). Microstructure, MFA as well as chemical composition were determined and the mechanical properties were graphically displayed with existing literature data. A deep

understanding of tracheid properties is necessary for better scientific cultivation and utilization of fast-growing softwoods.

Materials and methods

Fibers preparation

Wood blocks with similar sizes were cut from the outer parts of CW and OW of fast-grown plantation Chinese fir (*Cunninghamia lanceolata*) stems at a height of 1.3 m. All wood blocks were soaked in distilled water. Thin longitudinal-tangential tissue slices (150 µm) were cut from the transition wood between earlywood and latewood with a rotary microtome (RM2255; LEICA, Bensheim, Germany). In order to retain the single fibers (tracheids) in their natural state, the mechanical isolation procedure was performed using very fine tweezers under a light microscope [22]. After isolation the wet fibers were dried under glass sheets to avoid twisting [23].

X-ray diffraction measured and chemical analysis

A Philips X-ray diffraction system (X'pert Pro, PANalytical B.V., Almelo, NL) was used to evaluate the MFA of the different slices from CW and OW (11th–13th growth ring). The samples were attached to the sample holder by a double-sided adhesive tape. Six samples were obtained for each wood type. The calculation of the MFAs was performed according to the procedure described by Li et al. [24]. The samples of the X-ray analysis were used for chemical analysis afterwards. The content of cellulose and lignin was analyzed in accordance with the GB/T 744-2004 [25] and GB/T 747-2003 [26] standards, respectively.

Microtensile tests

The mechanical properties of the single fibers were tested in tension by using a microtensile testing device equipped with a 500 mN maximum capacity load cell, as described in detail in [27]. The air-dried single tracheids were glued onto 200-µm-thick polyester frames under a light microscope by using cyanoacrylate glue (Loctite 454). Prior to the tensile test and after glue-hardening, the test spans of the fibers were measured. The foliar frames carrying the individual cells were fixed by a pinhole assembly and were strained with a test speed of 0.5 µm/s. All the tensile testing was carried out at 25 °C and 50% relative humidity.

To calculate the ultimate stress and tensile stiffness of the fibers, both the cell and cell wall cross-sectional areas were determined according to the method described previously [28]. Typically, one part of the broken fibers were dipped into polyethylene glycol 2000 followed by sectioning with razor blades under a stereo-microscope and rinsing with warm water. The sectioned surfaces were

Table 1 The microfibril angle (MFA) and relative content of cellulose and lignin for compression wood (CW) and opposite wood (OW). Values in parenthesis are the coefficients of variation (%)

Samples	MFA (°)	Cellulose (%)	Lignin (%)
CW	25.1 (7.79)	42.64 (1.18)	36.43 (0.83)
OW	11.5 (2.43)	46.89 (0.43)	32.81 (0.75)

then observed using an environmental scanning electron microscope (ESEM; FEI Quanta 600) under low vacuum conditions to obtain images for area calculation using the software ImageJ. In total, ten successful measurements were performed for both CW and OW fibers.

Results and discussion

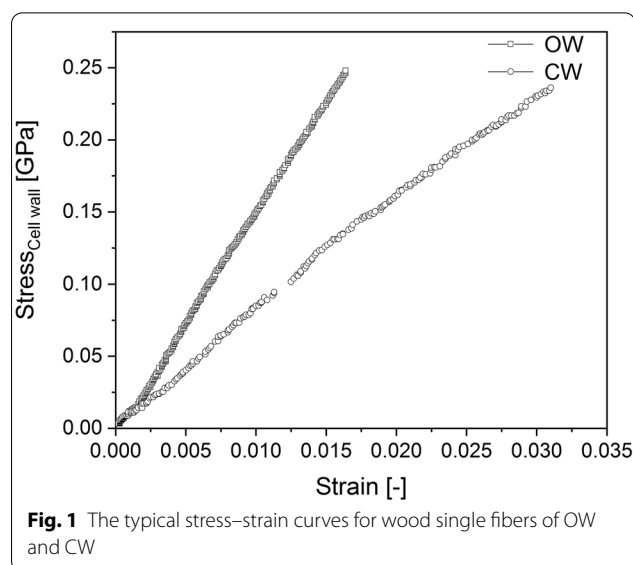
Chemical composition and structural properties

The values of MFA and relative content of cellulose and lignin for CW and OW from Chinese fir wood are presented in Table 1. The mean MFA values in the S_2 layer were $25.1 \pm 2.0^\circ$ and $11.5 \pm 0.3^\circ$ for CW and OW, respectively. The low MFAs of OW are in a similar range compared to the normal heartwood of six Chinese fir clones which ranged from 10.47° to 13.18° [29]. CW showed a higher MFA than OW, which is associated with environmental stimuli such as slope, winds, or other external forces [30, 31]. As shown in Table 1, the relative content of cellulose and lignin in CW was 42.64% and 36.43%, respectively; and those in OW was 46.89% and 32.81%, respectively. Clearly, compared to OW, CW had a higher lignin content and a lower cellulose content. The observed differences in lignin content between OW and CW are similar to those reported by Zhang et al. [32] for Chinese fir and Shirai et al. [33] for Ginkgo (38–39% lignin content of CW and 34–37% for OW). Based on the values of MFAs and the chemical composition analysis, the region chosen featured the mild type of CW from Chinese fir wood.

Tensile properties of single wood fibers

Exemplary stress–strain curves of CW and OW fibers from Chinese fir are shown in Fig. 1. For the individual fibers, the shape of the stress–strain curve appeared to be linear during the initial phase of the test, which has been repeatedly observed in some previous studies [10, 11, 22]. The failure strain of CW tracheids was slightly larger than that of OW tracheids, an effect that can be assigned to the larger MFA [11, 12].

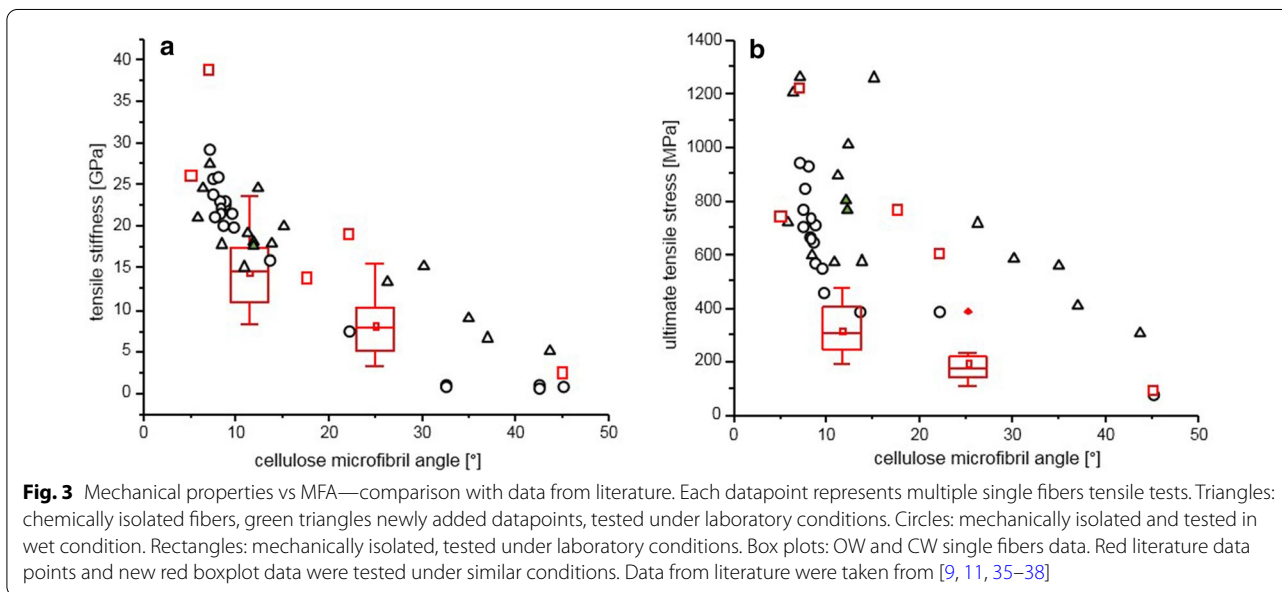
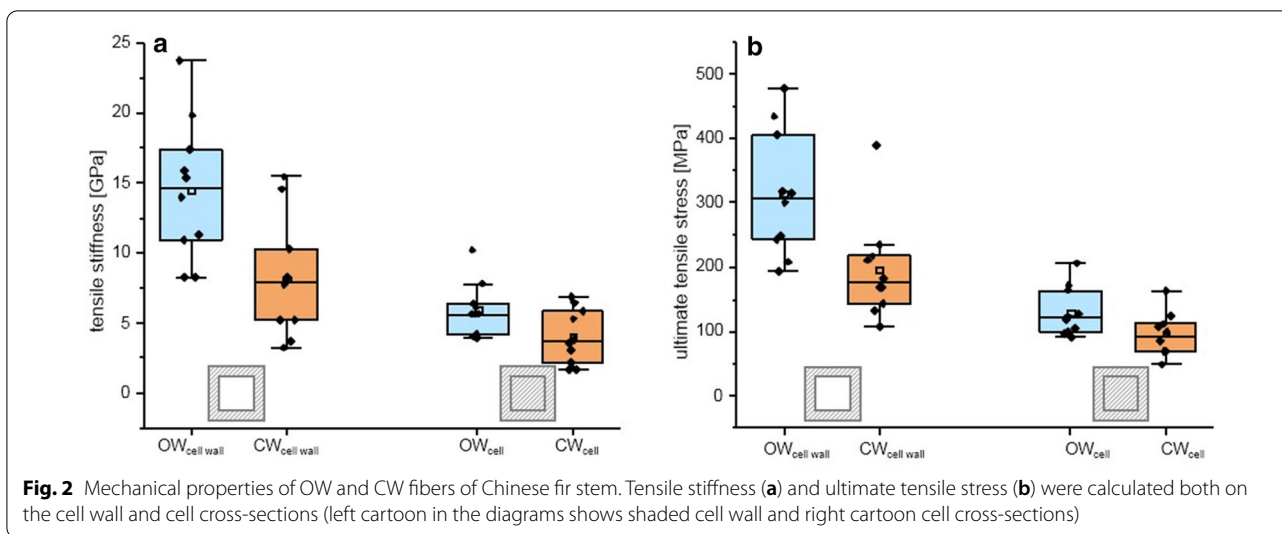
During the tensile tests, the average maximum load was 61 mN for CW fibers and 107 mN for OW fibers. Since the differences in cross-sectional geometries can be quite large between single wood fibers, stresses were calculated on the basis of cell and cell wall cross-sections



instead of analyzing forces. Furthermore, this allows a thorough comparison with literature data. Based on the stress strain diagrams tensile stiffness and ultimate tensile stress were determined (Fig. 2a, b), we found significant differences (Mann–Whitney test, statistically significant at the 0.05 level, Origin Pro 2020) between the tensile properties of OW and CW, except for the tensile stiffness when calculated on the basis of the cell cross-sections. Naturally, the tensile properties calculated on the cell cross-sections (box plots in the right corners of Fig. 2a, b) were lower than those calculated on cell wall cross-sections since the empty lumen does not contribute to tensile stiffness and strength. This is also a reason why the differences between the two-fiber groups appear more pronounced for the cell wall data.

The OW tracheids calculated on the cell wall cross-sections had the highest tensile modulus and ultimate tensile stress of 14.5 GPa and 314 MPa, respectively, which was more than 1.5 times higher than the tensile modulus and ultimate stress of CW tracheids (8.17 GPa and 196 MPa, calculated on the cell wall cross-sections). The lower mechanical properties of CW fibers can be explained by the larger MFA (25.1° for CW and 11.5° for OW) which have been shown to decrease with increasing MFA [11–14]. Unfortunately, only few literature data exists for single-fiber properties of Chinese fir. However, Yu et al. [11] determined the average tensile modulus of 17.6 GPa and ultimate tensile stress of 770 MPa for chemically isolated Chinese fir sapwood fibers with a MFA of $\sim 12^\circ$.

To get a better idea about these differences in wood fiber properties, the present mechanical data were plotted versus the MFA together with literature data—all calculated for cell wall cross-sections (Fig. 3a, b). The



majority of the plotted data has been published in a similar diagram before [34] and was supplemented by more recent data on Masson pine and Chinese fir [11]. The datasets of Fig. 3a show a clear relationship between the tensile stiffness and MFA, both for chemically isolated fibers tested under laboratory conditions (triangles), mechanically isolated fibers tested in the wet state (circles) and mechanically isolated fibers, tested under laboratory conditions (red rectangles). Data displayed in red were created under similar test conditions as the present data (box plots). Whereas larger datasets exist for both wet tested mechanically isolated fibers and for chemically isolated fibers, data about dry-tested mechanically

isolated fibers with known MFA is scarce. However, for the series of chemically isolated fibers (triangles) the newly added data points for Chinese fir and Masson pine (green filled) are in the same range as the other triangles. This suggests that differences in tensile behavior between tree species are small—at least after chemical isolation. The two circles at ~ 33° MFA (Ginkgo and Juniperus) suggest that species-related differences could also be small for mechanically isolated fibers. However, this is just speculative.

Despite the lack of a large literature datasets, CW fibers from Chinese fir fit well into the tensile stiffness diagram. The data for OW are slightly lower than comparable

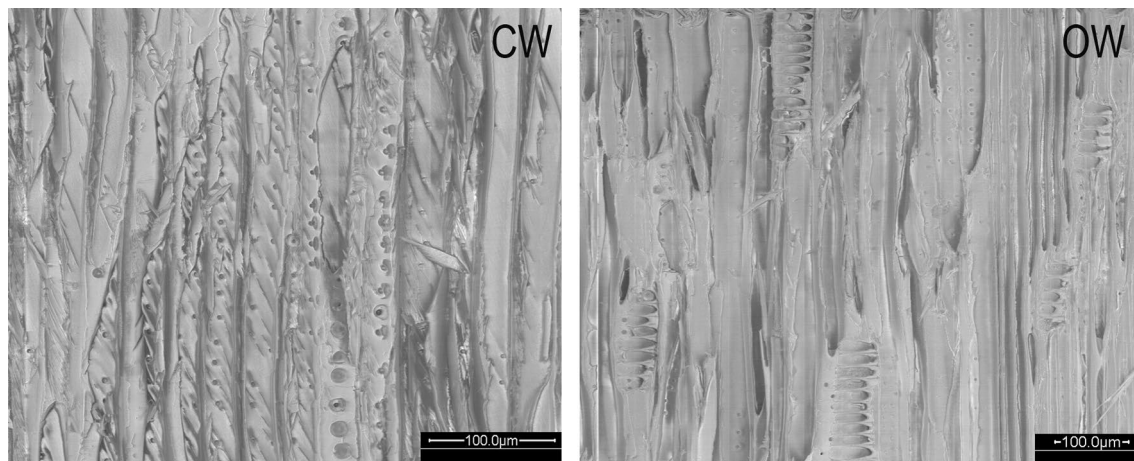


Fig. 4 Scanning electron micrographs of the distribution and size of pits on radial sections of CW and OW from Chinese fir wood

data from literature. In terms of ultimate tensile stress (Fig. 3b), literature data are broadly scattered. Reasons are that experimenters cannot pre-determine a site of fracture by tapering, etc., which means that the fibers will always break at sites with high stress concentrations close to the clamps or at areas along the fibers with high stress concentrations such as structural defects or pits. Furthermore, sample geometries such as cell wall thickness play a role concerning the fracture mode. This can be seen well in the diagram (Fig. 3b) for MFAs with a large number of datapoints (around 10°), regardless of the isolation procedures. However, ultimate tensile stresses of CW (and also of OW, but to a lesser extent) are lower than all literature data for a comparable MFA. It can only be speculated that heavily pitted CW tracheids, as shown in Fig. 4, or helical cavities promote early fracture. The reasons for the early fracture of OW tracheids remain unclear at this stage.

Another reason for the observed lower values of Chinese fir fibers could be differences in chemical composition. Based on already published data and the presented data, no conclusions can be made since numerous protocols for chemical analyses exist and the results are not directly comparable. However, our results suggest that single-fiber tests of wood of various species with differences chemical composition could contribute to answer the long-standing question of the role of wood polymers for cell wall mechanics of native wood. Each tree consists of wood with a wide range of MFAs. By comparing fibers with the same MFA and similar geometries but different chemical composition it might be possible to gain a much deeper understanding.

Conclusion

The structural characterization, chemical composition, and microtensile properties of the single tracheids between CW and OW from Chinese fir wood in fast-growth plantation were tested and compared. The tensile modulus and ultimate tensile stress of CW tracheids were lower than those of OW due to the larger MFA but the relatively lower cellulose content, more pits and the missing S3 layer might also play a role. A comparison with literature data showed slightly lower mechanical values than expected. These results can contribute to a further understanding of the structural–mechanical relationships of Chinese fir wood at the cell wall level, and provide a scientific basis for better utilization of plantation softwood.

Abbreviations

CW: Compression wood; OW: Opposite wood; MFA: Microfibril angle.

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Authors' contributions

ZL, ME, JL and JC conceived and designed the experiments; ZL and JJ wrote the manuscript; ZL and TZ performed the experiments and analyzed data. All authors read and approved the final manuscript.

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Availability of data and materials

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

Competing interests

The authors declare that they have no competing interests.

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