

Moisture Transfer and Drying Stress of *Eucalyptus* Wood during Supercritical CO₂ (ScCO₂) Dewatering and ScCO₂ Combined Oven Drying

Honghai Liu,^{a,b,*} Jinge Xie,^a and Jingwen Zhang^a

Supercritical CO₂ (ScCO₂) dewatering is a lumen water expulsion process, in which CO₂ is cycled between atmospheric pressure and supercritical phase and results in a fast expulsion of free water. In this study, specimens of 100 mm length *Eucalyptus exserta* F.V. Muell. wood were dewatered using ScCO₂, then the dewatered and un-dewatered wood were dried in oven at 100 °C to investigate moisture transfer and drying stress. The ScCO₂ dewatering was very fast. When the moisture content (MC) was over 40%, the MC distribution and gradient after dewatering and oven drying were different. The oven drying MC showed a ring contour distribution, but the ScCO₂ showed a higher MC in one side and a lower MC in the opposite; the oven drying MC gradient was great in the middle and small in the end part of wood, but the ScCO₂ MC gradient was small and roughly consistent. The oven drying moisture migration rates along the fiber and transverse directions were similar; however, the ScCO₂ migration rate was faster in the transverse directions. The MC distributions of the dewatered and un-dewatered wood after oven drying were similar at 30% MC. The ScCO₂ dewatering reduced wood residual stress, and it affected the stress development in the subsequent oven drying.

DOI: 10.15376/biores.17.3.5116-5128

Keywords: *Eucalyptus*; Supercritical CO₂ dewatering; Oven drying; Moisture distribution; Moisture transfer

Contact information: a: College of Furnishings and Industrial Design, Nanjing Forestry University, Nanjing 210037 China; b: Jiangsu Co-Innovation Center of Efficient Processing and Utilization of Forest Resources, Nanjing Forestry University, Nanjing 210037, China;

* Corresponding author: liuhonghai2020@njfu.edu.cn

INTRODUCTION

Eucalyptus plantations are widely planted in the southern regions of China due to their strong environmental adaptability and fast growth rate. They are the important industrial raw materials for the paper, wood-based panels, and fiber industries (Yang *et al.* 2022; Morales *et al.* 2017; Carignato *et al.* 2018; Zhen *et al.* 2021). There are some plantation eucalyptus species that have higher mechanical properties and beautiful grains; these have potential for use in high value-added solid wood products, such as furniture, flooring, doors, and windows (Teixeira *et al.* 2009; Chen and Zhu 2019; Zhao *et al.* 2021; Zhang *et al.* 2021; Yang and Jin 2021; Ruben *et al.* 2021). However, most plantation eucalyptus woods are prone to collapse and deformation during conventional kiln drying (CKD), and special drying conditions or processing is necessary for their further application (Wingate-Hill and Cunningham 1986; Franich *et al.* 2020; Huang *et al.* 2020).

Supercritical CO₂ (ScCO₂) dewatering has great potential in the difficult-to-dry wood due to its good solubility, strong transfer capacity, and special dewatering mechanism (Todd and Baroutian 2017; Ferrentino *et al.* 2018). However, eucalyptus experiences collapse and deformation during CKD because of its poor wood permeability. The capillary tension caused by free water migration in the CKD process applies pressure to the cell wall of wood, and cells collapse when the capillary tension is greater than their compressive limit (Tiemann 1941). To reduce the collapse in CKD, microwave (Zhou *et al.* 2009), compression (Hayashi and Terazawa 1975), and pre-freezing (Zhang *et al.* 2011) treatments are used before drying (Zuo *et al.* 2016), but the collapse cannot be solved completely. There are several dewatering methods that eliminate liquid capillary tension and solve the collapse and deformation of wood cells radically (Franich *et al.* 2014; Dawson *et al.* 2020).

The dewatering method of ScCO₂ is proposed for difficult-to-dry wood to shorten the drying time and reduce the deformation. During the dewatering process, the CO₂ is cycled in the equipment between supercritical and gas phase through changing the pressure. The ScCO₂ has excellent heat and mass transfer properties, and it can penetrate easily into wood. The ScCO₂ turns into CO₂ gas phase by reducing the pressure, which expels the free water out of wood (Dawson *et al.* 2015; Gabitov *et al.* 2017; Dawson *et al.* 2017). Studies have shown that the water in wood is removed at concentrations much higher than its solubility limit in SuCO₂, which implies that ScCO₂ mechanically displaces some of the water from the wood structure (Aggarwal *et al.* 2019; Banerjee *et al.* 2022). This dewatering can result in very small negative capillary tension and prevent wood collapse (Zhang *et al.* 2019; Yang 2021).

The ScCO₂ dewatering method has a higher dewatering rate and less deformation, but it can only dewater wood to near the fiber saturation point (FSP) (Dawson *et al.* 2015, 2020). Behr *et al.* (2014) and Meder *et al.* (2015) have studied the water distribution of wood after ScCO₂ dewatering, but they did not analyze the characteristics of water distribution and transfer in wood fiber direction. Meanwhile, there were some discussions on the practical application of ScCO₂ dewatering. Aggarwal *et al.* (2019) pointed out that ScCO₂ dewatering for pine flakes is potentially much more cost-effective than thermal drying from both capital and operational perspectives. The dewatering test for commercial potential with green radiata pine wood specimens (100×50×300 mm) using large-diameter pressure vessels and ancillary machinery has successfully performed (Dawson *et al.* 2020). The test showed the same results as those obtained from using the larger laboratory specimens. However, a significant benefit for wood product manufacture may be the ability to sequentially dewater green wood and then undertake wood modification (Franich *et al.* 2020).

In the current study, *Eucalyptus exserta* F.V. Muell. samples were first dewatered to 40% moisture content (MC) using ScCO₂, and the dewatered and un-dewatered samples were dried to 30% MC in an oven at 100 °C. The dewatering rate, MC distribution in the middle and the end of the wood, the characteristics of MC transfer, and the drying stress of wood in the ScCO₂ dewatering, oven drying, and ScCO₂ dewatering combined with oven drying were compared.

EXPERIMENTAL

Materials

Eucalyptus exserta F.V. Muell. trees were collected from Guangxi, China. Their diameter at the collection height was approximately 200 mm. The trees were prepared into logs with 1000 mm length, and subsequently, they were prepared into lumbers with dimensions of 30(R) × 30(T) × 1000(L) mm, sealed using plastic films, and stored at 4 °C. Two lumber pieces were sawed into end-matched samples (8 pieces) with dimensions of 30(R) × 30(T) × 100(L) mm, as illustrated in Fig. 1. The average initial MC of all samples was 58%.

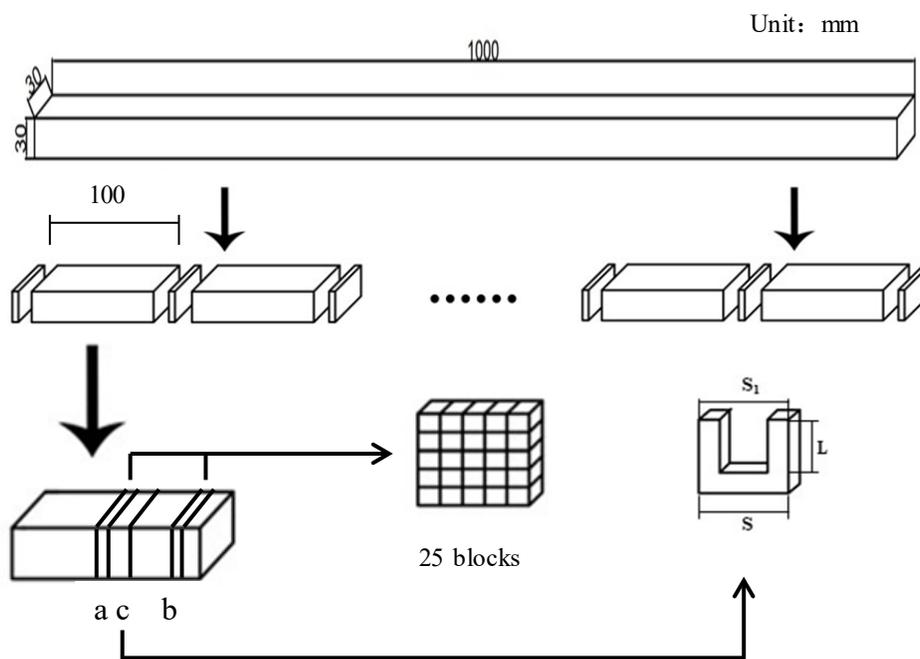


Fig. 1. Diagram of sample preparation

Equipment

The drying devices include a ScCO₂ dewatering equipment (DY221-50-06; Huanan Supercritical Fluid Extraction Co., Ltd, Nantong, Jiangsu, China), an electricity heating oven (DHG-905386-III, Shanghai Cimo Medical Instrument Co., Ltd, Shanghai, China), an electronic balance, HC2004 (0.001g, Sincere Dedication of Science and Technology Innovation Company, Shanghai, China), and an electronic vernier caliper (CD-20CPX (0.01mm, Mitutoyo Co., Ltd., Tokyo, Japan).

Wood ScCO₂ Dewatering

Wood ScCO₂ dewatering tests were conducted according to the parameters in Table 1, which was obtained based on the trial test. The drying procedures are illustrated in Fig. 2). The process included the following: 1) opening the sealing valve of the drying vessels, inserting samples into the vessels and then closing the sealing valve; 2) setting the drying temperature at 45 °C and starting the heating device; 3) opening the valve of CO₂ bottle,

transferring CO₂ to drying vessels (Fig. 2-3,4), then increasing the temperature and pressure of CO₂ to supercritical stage (45 °C/25 MPa); 4) ScCO₂ penetrating into wood and dissolving water in the drying vessels, and then delivering water to adsorption vessels (Fig. 2-5,6) by lowering the pressure of ScCO₂; 5) 15 min later, decreasing temperature and pressure of ScCO₂, taking out samples as pressure reaching to atmosphere pressure; 6) samples were kept in room temperature for 30 min to release CO₂ in wood, then were measured on a balance. This completed one full drying cycle. Several drying cycles were performed to decrease the MC of wood to 40%, and then the mass of samples was measured again. A total of 8 samples were dewatered, 4 of them were sawed and prepared for determination of MC distribution and stress, and the remaining 4 samples were oven-dried.

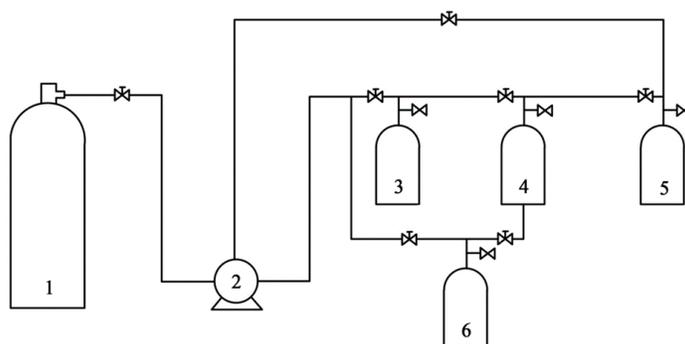


Fig. 2. Schematic diagram of the equipment and ScCO₂ dewatering process. (1) CO₂ bottle; (2) pump; (3, 4) drying vessel; (5, 6) drying adsorption vessel

Table 1. Parameters of Supercritical CO₂ Dewatering

Parameters	Values
Maximum pressure (MPa)	25
Minimum pressure (MPa)	0.1(atmospheric pressure)
Pressure rise time (min)	30
Decompression time (min)	10
Drying temperature (°C)	45
Hold time (min)	15

ScCO₂ Dewatering Combined with Oven Drying and Oven Drying

After the dewatering tests, 4 dewatered samples together with the 8 un-dewatered samples were oven-dried (OD) at 100 °C. The combined drying is termed as ScCO₂-Oven. During oven drying, all samples were tanked out in 10-min intervals and cooled in sealed bags, then were measured for mass using an electronic balance and dimensions with an electronic vernier caliper. They were oven dried until their MCs decreased to 30%.

Determination of Moisture Content and Moisture Distribution

The MC of the samples was determined in accordance with GB/T 1931 (2009). When the MC of wood decreased to 40 and 30% during dewatering or oven drying, thin slices were sawn in the middle (Fig. 1a) and the right end (Fig. 1b) of three samples. A thin slice was immediately split into 25 small blocks (Zhou *et al.* 2009; Gabitov *et al.* 2017), which were dried in an oven at 103 ± 2 °C to obtain their MCs (Meder *et al.* 2015). The

MC distribution charts were plotted using the data of three samples and Origin 2021 software (OriginLab Corporation, Northampton, USA).

Residual Stress Determination

The residual stress was determined according to GB/T 6491 (2012). After the oven drying, a 10-mm-thick sample (Fig. 1c) was sawn from a sample and then dried in an oven at 103 ± 2 °C for 2 h, which produced a stress sample with a concave shape as shown in Fig. 1. The initial dimensions of S and L were measured using an electronic digital caliper, and then it was placed in a ventilated place at room temperature for 24 h. The final dimension of S_1 was measured using an electronic digital caliper. The residual stress index Y was calculated using Eq. 1, which was the average values of four stress samples,

$$Y = (S - S_1) / 2L \quad (1)$$

where Y is the residual stress index (%), S is the initial width of the stress sample (mm), S_1 is the final width of the stress sample (mm), and L is the length of the stress sample (mm).

RESULTS AND DISCUSSION

Drying Curves of ScCO₂-Oven and OD

Figure 3 shows the curves of combined process of ScCO₂-Oven and OD alone. The MC decreased from 58% to 39% in 1 h in OD process, while, it took 1.5 h until the MC decreased to 36% in ScCO₂ dewatering process. The drying rate of OD was faster because the water in wood was converted into steam at 100 °C. Compared with the curves of ScCO₂ dewatering to OD in Fig. 3, the ScCO₂ dewatering rate was 73% of OD, indicating a fast dewatering.

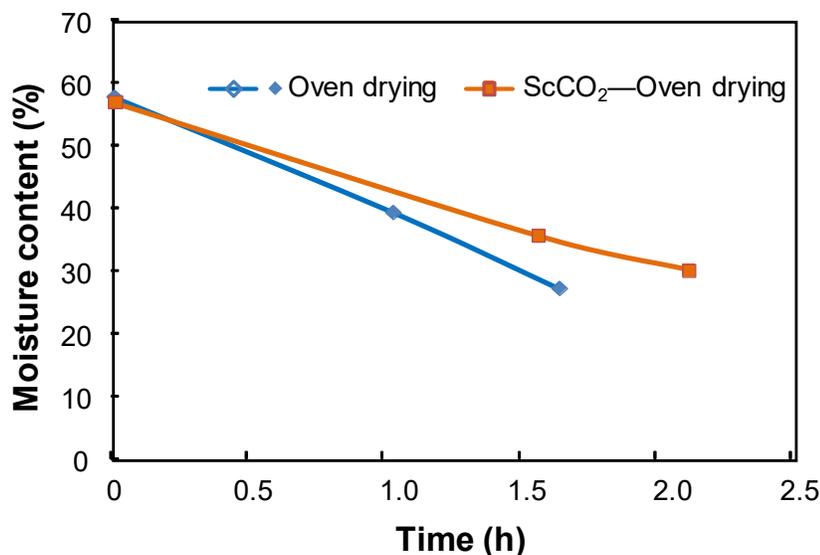


Fig. 3. Moisture change curves of oven drying and ScCO₂-oven drying

In contrast to previous conventional kiln drying with the same sample size performed at 50 °C and 84% relative humidity (Zhang *et al.* 2020), the dewatering rate of ScCO₂ was 2.5 times higher. Therefore, ScCO₂ has a very fast dewatering rate when the MC is above 40%. When the MC decreased from 40 to 30%, the drying rate of OD alone increased a little, while samples after ScCO₂ dewatering decreased in this period. The early ScCO₂ dewatering results in low initial temperature (45 °C) of wood and different MC gradients (Fig. 4), leading to a relative low drying rate in the subsequent oven drying. Furthermore, the ScCO₂ dewatering opened up the pore openings within the wood. The improved permeability of wood is beneficial for water removal and modifier penetration in the subsequent treatments, such as drying and impregnation modification (Newman *et al.* 2016; Yang and Liu 2020; Xu *et al.* 2021; Yang *et al.* 2021; Cao *et al.* 2021; Pang *et al.* 2021).

MC Distribution in Wood after Oven Drying and ScCO₂-Oven

Figure 4 shows a two-dimensional distribution MC in the middle (Fig. 4a) and in the end (Fig. 4b) of the wood, when the MCs were at initial, 40 and 30% stages for ScCO₂-Oven and oven alone. Figure 5 is the average MC of the wood blocks (Fig. 1a, b) and their standard deviations (error bars) at the corresponding MC stages.

The initial MC of samples used for two dryings tests are uniform, showing a similar average MCs and error bars (Fig. 5). When the MC decreased to 40%, the distribution of MC of wood after ScCO₂-Oven and oven alone were different. The MC of wood after oven drying presented a ring contour distribution, showing a higher MC in central and lower MC outside (Fig. 4a, b). The contour in the middle of the sample was crowded, but become thin in the end of the sample. This result indicated a greater MC gradient in the middle and a smaller gradient in the end. A similar phenomenon was found in Fig. 5a, b. The error bars of the average MC in the middle of the sample were much larger than those in the end, indicating a bigger MC gradient in the middle of the sample. However, the MC of wood after dewatering showed a higher MC distribution in one side and a lower MC in the opposite side (Fig. 4a, b). Whether in the middle or the end of the samples, the MC contour was gently transitioned from one side to the opposite side, showing a small and roughly consistent MC gradient. The error bars of the sample in the middle and in the end after dewatering were similar, indicating that the MC distribution and difference were quite small.

When the MC decreased from 40% to 30%, all samples underwent oven drying. Thus, the MC of the samples showed a similar tendency, *i.e.*, a higher MC distribution in one side and a lower MC in the opposite side. The MC distribution of oven drying alone was similar in the middle and in the end of sample, but the error bars in the middle were greater than that in the end (Fig. 5a, b.), indicating a higher MC gradient in the middle. The errors of MC in the end of samples (Fig. 5a, b.) after ScCO₂ dewatering were greater than the middle, showing a higher MC gradient in the end. This result could be attributed to the effect of ScCO₂ dewatering to wood (Franich *et al.* 2014).

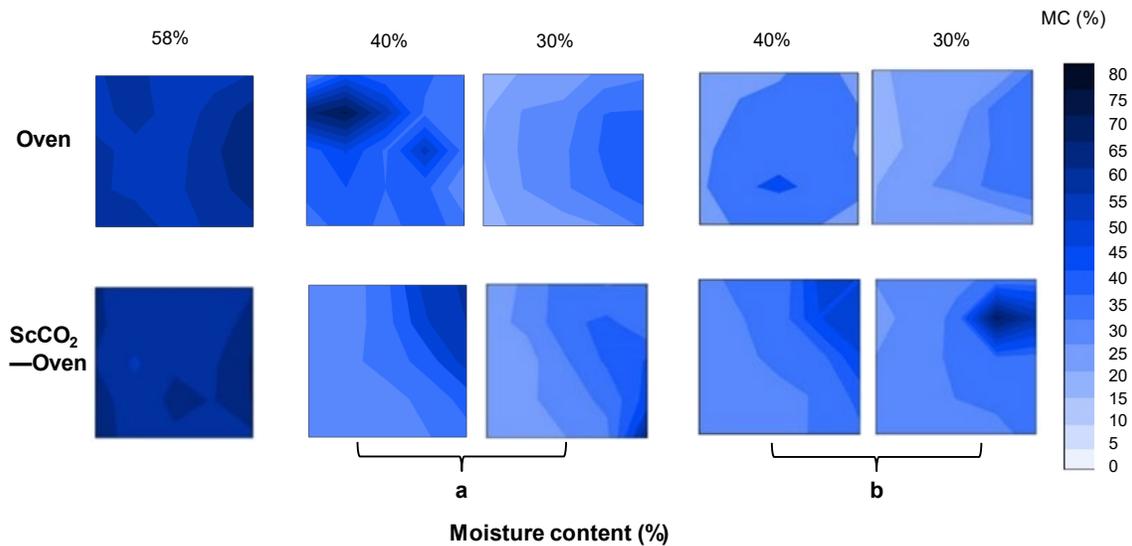


Fig. 4. Moisture distributions in wood during drying. a: middle of sample and b: end of sample

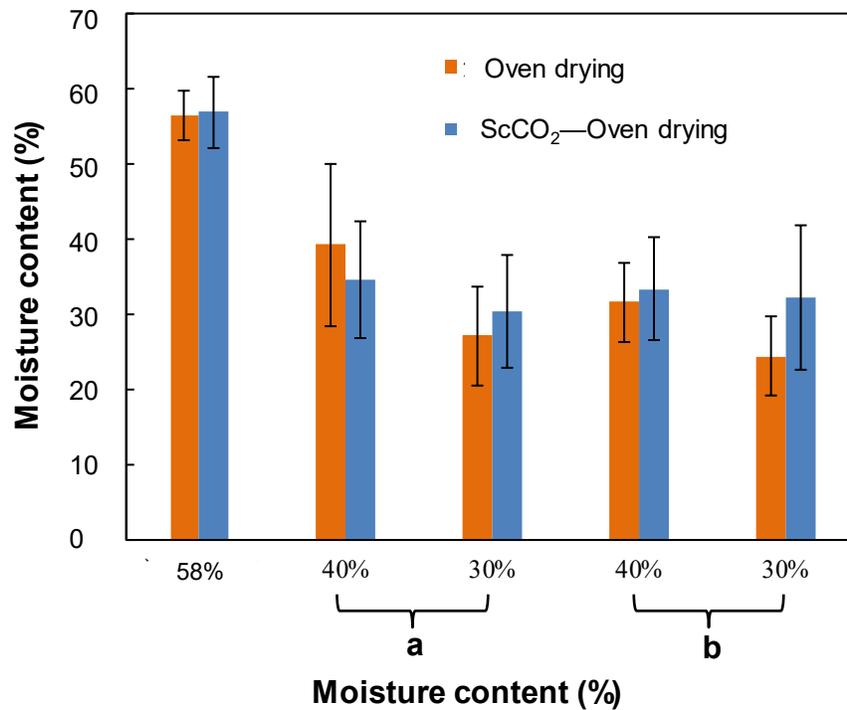


Fig. 5. Moisture content and its deviation of MC samples. a: middle of sample and b: end of sample. Bars indicate the standard deviations for the wood blocks cut from each sample.

Characteristics of Moisture Transfer in Wood during Drying

Figure 6 shows the MC gradients in wood fiber and transverse direction (perpendicular to the fiber direction) when the MCs were at 40 and 30% for ScCO₂-Oven and oven alone. The characteristics of moisture transfer during drying were studied based on the MC distributions in Fig. 4 and the MC gradients in Fig. 6. When the MC was 40%, the gradients of oven drying alone in fiber direction were approximately 8.3%, which are

largely similar to those in the middle and end. This result indicates that the moisture transfer in fiber and transverse direction were almost same, showing a very fast moisture migration. As the MC decreased to 30%, the MC gradients decreased to an average value of 3.4%, but with obvious differences. The MC gradient in the transverse direction in the middle was the greatest, followed by the end, and the lowest value was in the fiber direction. The MC gradient in the transverse direction was 1.4 times that in fiber direction, indicating that MC migration occurred mainly along the transverse direction in this stage.

The MC gradient variation of the ScCO₂-Oven drying was different from oven drying alone. When the MC was 40%, the MC gradient had a positive value, indicating that the MC in the middle was greater than the end. However, the MC gradients in the middle and end were both negative values, indicating that wood in the central MC was lower than wood surface. This result indicates that wood central moisture migrated to the end and the side surfaces of wood simultaneously, but the migration rate in transverse direction was greater than the fiber direction. As the MC decreased to 30%, the MC gradient value in fiber direction became negative, indicating the MC in the end was greater than the central part. In this stage, the MC gradient value in the middle of sample was still negative, but became much smaller than that at 40% MC. However, the MC gradient value in the end became positive, indicating the MC in the central part in the end of sample was greater than at the surfaces part. This data indicated that subsequent oven drying affected the MC distribution of wood dewatered by ScCO₂, which resulted in a fast transverse migration of water in the surface parts in the middle and end of samples.

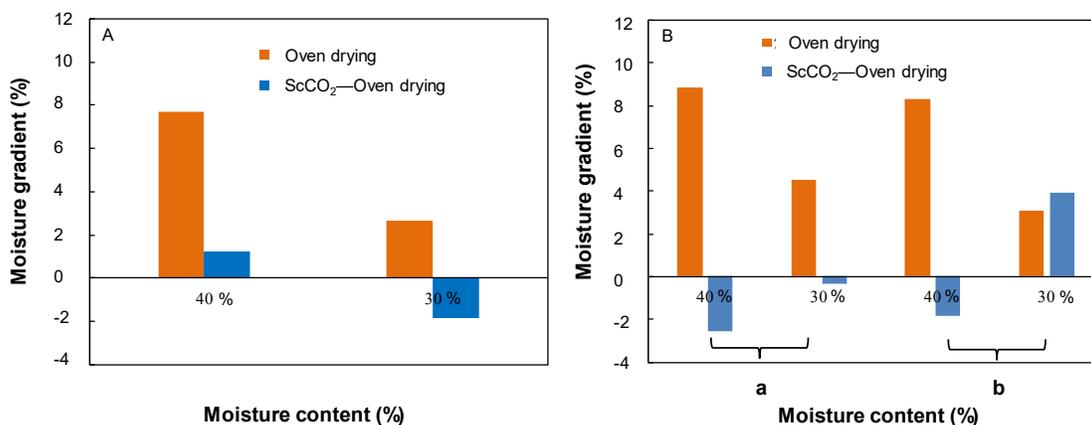


Fig. 6. Moisture content gradients in fiber direction (A) and transverse direction (B) of the samples. a: middle of sample and b: end of sample

Residual Stress of Wood after Drying

Figure 7 presents the residual stress in wood when the MCs were at 40 and 30% stages for ScCO₂-Oven and oven alone. The residual stress of oven drying alone was higher than ScCO₂ dewatered along and ScCO₂-Oven drying when the MC was close to 30%. This result indicates ScCO₂ dewatering can reduce the residual stress, but it affects the stress development in the subsequent oven drying. The reason may due to the effect of ScCO₂ dewatering on the MC distribution in wood, which influences the shrinkage and stress when the MC of wood was lower than fiber saturated point (FSP). Furthermore, the micro-

structure of wood was modified by early ScCO₂ dewatering (Newman *et al.* 2016), which affects the moisture migration, shrinkage, and stress development in the subsequent oven drying. When the MC was 40%, the residual stress was 5%, indicating ScCO₂ dewatering also can result in stress at high MC stages. It can be explained by the MC distribution in Fig. 4. In this stage, the average MC was about 40%, but the MC distribution was uneven. The MC of samples in certain locations was also lower than FSP, where wood has begun to shrink (Yang *et al.* 2020), leading to stress. The residual stress between ScCO₂-oven and oven alone was not clearly significant when the MC dropped to 20%, indicating the effect of ScCO₂ dewatering on transfer and distribution of moisture were progressively weaker, and oven drying controlled the development of the stress.

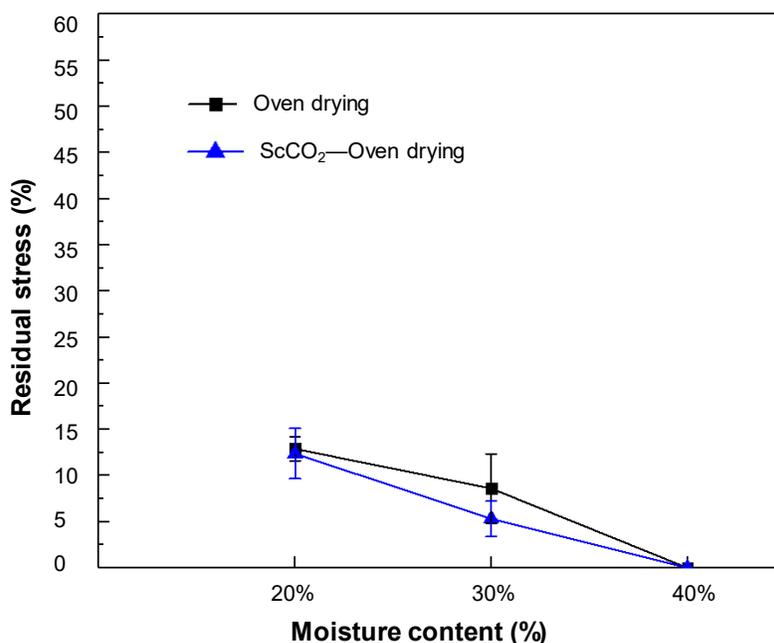


Fig. 7. Residual stress of oven drying and ScCO₂-oven at various MC stages

CONCLUSIONS

1. When the moisture content (MC) was above 40%, the drying rate with supercritical carbon dioxide (ScCO₂) was very fast, *i.e.* 73% and 2.5 times of that in oven and conventional kiln drying, respectively. The drying rate of ScCO₂-O was less than the oven drying.
2. When the MC was above 40%, the MC distributions of ScCO₂ dewatering and oven drying were different. The MC of oven drying showed a ring contour distribution with a higher MC in the center and lower MC outside. The MC gradient was great in the middle and small in the end part of wood. However, the ScCO₂ dewatering showed a higher MC distribution in one side and a lower MC in the opposite side. The MC gradient in the middle and the end of wood was small and roughly consistent. The

moisture migration rate along the fiber and transverse directions in the oven drying was roughly same; however, it was faster in the transverse directions in the ScCO₂ dewatering.

3. When the MC decreased to 30%, the MC distribution of dewatered and un-dewatered wood after oven drying was similar, showing a higher MC in one side and a lower MC in the opposite side. The moisture migration in oven drying was mainly along transverse direction.
4. At all MC stages, the residual stresses in the oven were greater than ScCO₂ dewatering and ScCO₂-Oven drying. The ScCO₂ dewatering reduced the residual stress of wood, and affected the stress development in the subsequent oven drying.

ACKNOWLEDGMENTS

The authors are grateful for the support of the National Natural Science Foundation of China (Grant Nos. 31870545 and 31570558) and Nanjing Forestry University Undergraduate Innovation Training Project (2021NFUSPITP0700).

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Article submitted: June 2, 2022; Peer review completed: June 29, 2022; Revisions accepted: July 1, 2022; Published: July 14, 2022.
DOI: 10.15376/biores.17.3.5116-5128