Effects of Ultrasound Pretreatment on Microstructure and Drying Characteristics of *Eucalyptus urophylla* × *E. grandis*

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Wood collapse-shrinkage is a severe defect that occurs in Eucalyptus timber during drying. To solve this problem and provide the technical support for the Eucalyptus application as high value-added wood products, *Eucalyptus urophylla* × *E. grandis* samples were pre-treated by ultrasound at 400 W power for 1, 3, or 6 h and then dried in the kiln. The effects of ultrasound pre-treatment on drying rates, residual stress, and total shrinkage after kiln drying were investigated. The results indicated that ultrasound pre-treatment increased drying rates, which was increased by 5%, 13%, and 11% when moisture content (MC) was above 24%MC according to the treating duration, and below 24%MC, which were increased by 25%, 28%, and 23%. Drying rate increased below 24% MC. The residual stress decreased during the later drying stage, especially for the condition of 3 h of ultrasound pre-treatment compared with the untreated samples, while it increased in the early stage of the drying process. The total shrinkage decreased after ultrasound pre-treatment, and it decreased 14.9% after 3 h pre-treatment. Ultrasound pre-treatment broke the pit membranes and modified the microstructures of the wood. The microstructure changes not only affect the drying rate, but also affect the developing of stress and strain, collapse, and its recovery.

Keywords: Eucalyptus urophylla × E. grandis; Ultrasonic pretreatment; Drying rate; Drying stress; Collapse

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INTRODUCTION

Eucalyptus is a fast-growing species widely planted in most parts of the southern hemisphere and China (Stanturf *et al.* 2013). Presently, most of the Eucalyptus trees are used for pulp production (Oudia *et al.* 2009). However, many eucalyptus trees have great potential in higher value solid wood products such as furniture, doors, and wood floorings due to the wood's nice characteristics of high density, mechanical strength, and beautiful grain. However, some species of eucalyptus are hard to dry without deformation because of its special shrinkage properties (Chafe 1985; McKenzie *et al.* 2003a).

Wood collapse is the most severe drying defect with serious deformation or great cracks inside the wood. Timbers with serious collapse normally cannot be used as solid wood product materials.

To prevent or reduce collapse, pre-treatments such as pre-steaming (Chafe 1990), pre-boiling (Chafe 1993), pre-heating (Mugabi *et al*. 2011), press or stretch (Terazawa and Hayashi 1975; Ruben *et al*. 2014), pre-freezing (Zhang *et al*. 2011), and continuous and intermittent drying (Chafe 1995) have been used. Pre-treatments have some influence on decreasing wood collapse.

Ultrasound is an efficient alternative to improve drying rate without heating up the material (Cohen and Yang 1995). Ultrasound waves cause a rapid series of alternative compressions and expansions as it is applied in liquid. Furthermore, ultrasound can produce cavitation, which contributes to the removal of moisture attached strongly to solid materials.

Microscopic channels (Wang *et al*. 2017) caused by micro-deformation of porous solid materials treated *via* ultrasonic waves contribute to the improvement of diffusion and convective mass transfer (Fuente-Blanco *et al*. 2006; Soria and Villamiel 2010; Qiu *et al.* 2016).

Recent studies have focused on the ultrasound pre-treatment of material prior to drying. Pre-treatment can reduce drying time (Mothibe *et al*. 2011), increase mass transfer rate (Cárcel *et al*. 2011), and increase the effective water diffusivity (Bantle and Eikevik 2011). However, there have been few reports so far on the effect of the drying characteristics such as drying rate, drying stress and strain, and especially the effect on the collapse property of *Eucalyptus* species of wood treated by ultrasound.

In this work, eucalyptus wood was subjected to ultrasound pre-treatment, and the effect on drying rate, drying stress and strain, and wood collapse after conventional drying were investigated. A microscopic analysis was used to investigate the wood micro-structural changes and its influence on the drying rate and collapse.

EXPERIMENTAL

Materials

Six-year-old plantation-grown eucalyptus (*E. urophylla* × *E. grandis*) was produced from Guangxi province, China. The diameter at breast height (DBH) was 26.0 cm. End matched square timbers with dimensions of 30 mm (T) × 30 mm (R) × 1000 mm (L) were processed from tree stems after felling. The initial MC was about 110%, and basic density was 0.47 g/cm^3. Finally, according to Fig. 1, 40 end-matched specimens with dimensions of 30 mm (T) × 30 mm (R) × 50 mm (L) were processed, with 30 specimens marked for ultrasound pre-treatment and 10 specimens marked for no treatment.

Equipment

The ultrasound treatment equipment (BILON-500, Shanghai BiLang Instrument Co., Ltd, Shanghai, China) was operated at a frequency of 20 to 25 kHz and power of 10 to 500 W. Other equipment included a constant temperature and humidity test chamber (DF-408, Nanjing FuDe Instrument Co., Ltd, Nanjing, China), electricity heat drum wind drying oven (DHG-905386-III, Shanghai Cimo Medical Instrument Co., Ltd, Shanghai, China), and scanner (CanoScan LiDe 700F, Canon, Melville, NY, USA).

Methods

Moisture content determination, ultrasound pre-treatment and conventional drying

The initial MCs of specimens were measured according to the standard GB/T 1931 (2009). As shown in Fig. 1, two 5 mm thickness wood sections were dried at 103 ± 2 °C; the average MC of these two sections were considered as the initial MC of the corresponding specimen between them. Then, 3 groups (each group contained 10 end-matched specimens) were pre-treated by ultrasound at the conditions of 400 W power and 25 kHz frequency for 1, 3, or 6 h. The treated and untreated specimens were dried at 60 °C and 66% relative humidity until all specimens reached the equilibrium moisture contents (EMC). During the drying process, specimens were removed individually from the chamber at the designated MC stage.

Shrinkage and stress measurement during drying

Once the specimen was taken out from the drying chamber, two 3 mm sections labeled: ① left side and ② right side were sawed from the middle of the specimen. Section ① was used for shrinkage measurement, and section ② was used for stress measurement.

Based on the previous research of Wu *et al*. (2007) and Yang *et al*. (2014), the shrinkage of the section area was used to evaluate the shrinkage and collapse situations during drying. The area of wood sections measured combined the technology of scanning and figure area calculation software Image J (https://imagej.nih.gov/ij/). The original area of every specimen was the area of the initial MCs sample section at the left side of the corresponding specimen which was measured after initial MCs sample sawing prior to starting the experiment. The area of specimens No. 1, No. 2, and so on, at different MCs, were measured through section ①, which corresponded to the specimen taken out from the chamber. For measurements, sections after sawing were scanned as figures into the computer, and the area of scanned figures was calculated by Image J software with a precision of 0.01 mm^2. The area shrinkage rate was calculated by Eq. 1,

$$S_i\,(\%) = (A_{orig} - A_i) / A_{orig} \times 100 \quad (1)$$

where S_i is the area shrinkage rate at MC i, (%), A_{orig} is the original area of every specimen (mm^2), and A_i is the section area of the corresponding specimen at MC i (mm^2)

As shown in Fig. 1, section ② was cut into 5 slices for stress measurement. After cutting, the initial length (L) and weight of every slice was measured immediately, and then they were stored in the room condition for MC equilibrium. After 24 h, the *f* (deflection) was measured, the residual stress index Y was calculated by Eq. 2 (GB/T6419-2012). According to the value of Y, the stress during the drying process can be observed and analyzed. In this study, the Y was the average value of surface layer in slices 1 and 5.

$$Y\,(\%) = f / L \times 100 \quad (2)$$

Micro Structure Observation

As shown in Fig. 1, material on the right side of the specimen was chosen for scanning electron microscopy (SEM; JSM-5610LV-JEOL, Tokyo, Japan). To observe the collapse of the cell and its recovery, both the cut surface and a thin slice (mirror surface) were prepared as described by Yang *et al*. (2014), and these were fixed on the holder. Finally, both samples were coated with gold and observed by SEM.

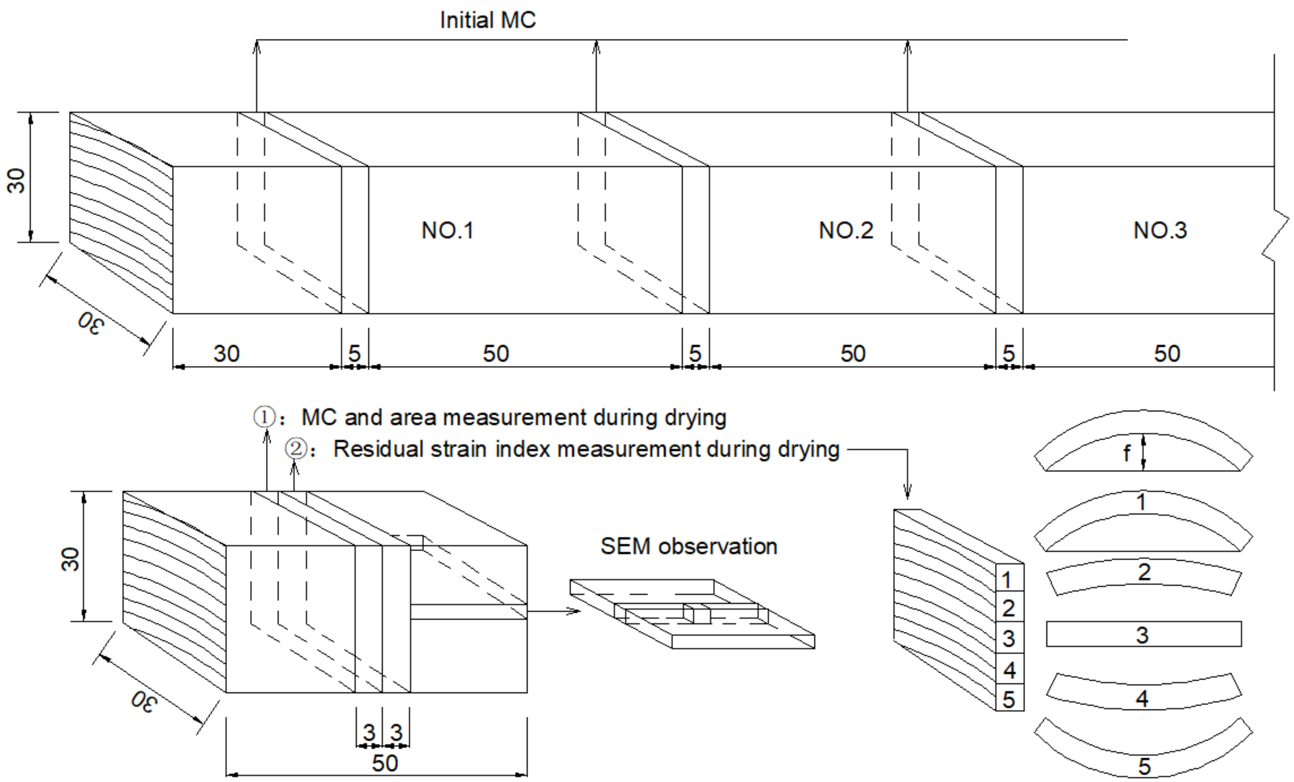

Fig. 1. Illustration for specimen, moisture content, residual strain, and samples of SEM observation

RESULTS AND DISCUSSION

Effect of Ultrasound Treatment on Wood Microstructure

The SEM micrograph of specimens treated after drying is presented in Fig. 2. Ultrasound treatment had a noticeable effect on the tissue structure of treated specimens. Some of the pit membranes broke down, and there were some micro-channels on the pits (the red circles) and similar observations are also reported in other studies (Qiu *et al.* 2016, 2017, He *et al*. 2017). Meanwhile, collapse mainly occurred in untreated wood ray cells during conventional drying from the SEM observation on the tangential cell section (Fig. 3 a, c). By the comparison of collapse extent and quantity between the collapsed cell (a-1, c-2, c-3) and the recovered cell (b-1, d-2, d-3) at mirror conditions, it was observed that collapse was slight in the specimens after ultrasound treatment. The observation may be attributable to the physical effects of acoustic waves and the micro-turbulence generated by transient cavitation bubbles in liquid (Donohoe *et al*. 2008). Finally, the permeability is likely to have improved after the ultrasound treatment. The drying rate was increased and collapse reduced compared with untreated specimens.

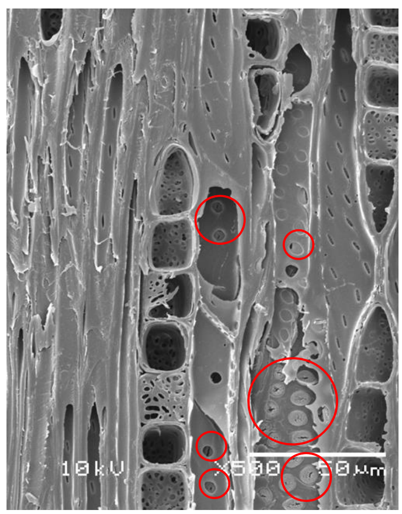

Fig. 2. The microstructure characteristics of specimens treated in 3 h (MC around 10%)

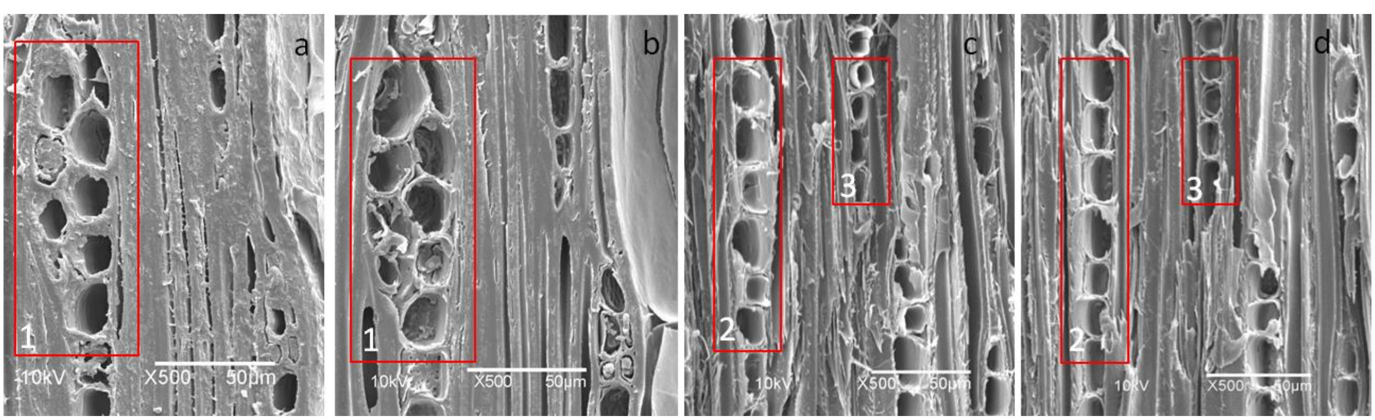

Fig. 3. Collapses and recovery of cells in specimens untreated (a, b) and treated in 3 h (c, d). (a, c) collapse; (b, d) recovery at the same location with a mirrored photo (MC around 10%)

Effect of Ultrasound Treatment on the Drying Rate

The drying curves of MCs *versus* time for treated and untreated specimens are shown in Fig. 4. MCs decreased more quickly in treated specimens than in untreated specimens, especially for the 3 h treatment. The drying rate was compared above and below 24% MC near the fiber saturated point (FSP). Drying rates of specimens were improved by 5%, 13%, and 11% above 24% MC and 25%, 28%, and 23% below 24% MC after 1, 3, and 6 h ultrasound treatments, respectively (Table 1). Drying rates were obviously improved when MC was below 24%. Drying rate showed significant difference between 3 h pre-treatment and untreated. The improvement in drying rates may be attributable to ultrasound-related cavitation or physical effects of the acoustic waves during pre-treatment, which resulted in pit membranes being broken, some collapse, and micro-channels on the pits (Fig. 2). The similar observations of broken pits were reported in related studies (Donohoe *et al*. 2008; Fernandes *et al*. 2011; Bussemaker and Zhang 2013; Ozuna *et al*. 2014). After ultrasound treatment, moisture diffusion coefficient was improved (Yao *et al.* 2016) and extracts was decreased (Xu *et al*. 2017), which are benefit for MC removal, therefore, ultrasound treatment improved wood drying rate.

In this study, drying rate was obviously improved for the 3 h pre-treatment, while it was not significant for the 6 h pre-treatment. This may be attributed to the short length of the specimen. Due to moisture removal mainly occurs in fiber direction, the 6 h ultrasound treatment did not bring obvious effect on drying rate improvements for short samples compared with that in 3 h.

Table 1. Drying Rates of Treated and Untreated Samples below and above FSP

Duration (h)	Initial MC (%)	Final MC (%)	Drying Rate(% / h)		
			MC > 24%	MC < 24%	Total
0	105.8	10.0	4.06	0.52	2.19
1	106.5	9.0	4.26	0.65	2.30
3	101.5	8.5	4.60	0.67	2.33
6	103.2	9.0	4.52	0.64	2.30

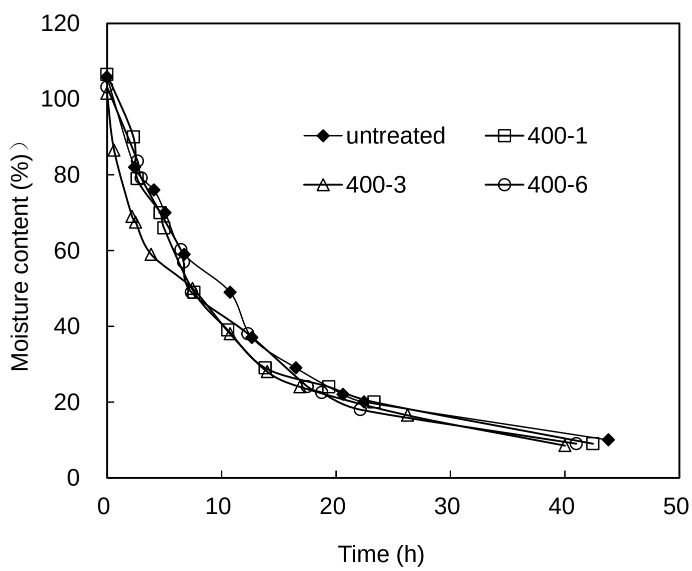

Fig. 4. Curves of moisture content change for treated and untreated samples

Effect of Ultrasound Treatment on the Stress

Figure 5 shows the residual stress curves of specimens treated and untreated during drying. Both of the residual stress variation trends are similar. During the initial period of 10 h from drying, moisture removal in the surface layer was fast and resulted in greater MC gradient in the wood cross sections. Additionally, the shrinkage was obviously affected by MC gradient. Therefore, the residual stress index Y increased quickly from zero to the maximum and then reduced gradually due to the shrinkage of the middle layers, 2 and 4 (Fig. 1), which slowed down the tensile stress in the outer layer. As drying continued, MCs in the core layer decreased to the FSP and the core layer wood began to shrink. The shrinkage in the core layer prevented and slowed down the tensile stress in the surface layer, and finally the tensile stress was fixed. As the residual strain was composed with visco-elastic creep strain and mechano-sorptive strain, the characteristics of strain change can be explained *via* the wood rheology property of visco-elastic creep strain and mechano-sorptive strain. Mechano-sorptive strain occurs at any MCs, and performs differently in surface or inner layers. During the drying process, surface layers (1 and 5) maintained tensile strain while inner layer maintained compress strain. Previous studies (Li and Gu 1999; Tu *et al*. 2004) indicated that tensile strain of visco-elastic creep strain and mechano-sorptive strain rose to the maximum during the initial period of drying because of the increased stress; it then decreased gradually as drying continued due to the direction of stress changing. However, the change of stress only affected mechano-sorptive strain partly, and the form in the initial period of drying was maintained. It can be seen that the performance of residual stress index (Y) in Figure 5 was similar to the results above. In the initial period of drying, Y increased for the increased stress and then decreased as direction of stress changing. During this period, the visco-elastic creep strains transformed into compress strains, while the mechano-sorptive strain did not change and always maintained tensile strain, therefore wood surface layer always maintained the situation of residual tensile strain.

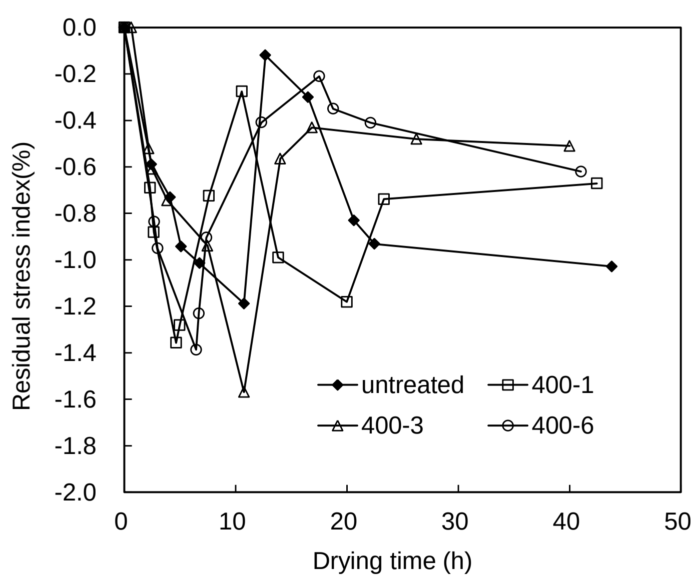

Fig. 5. Curves of residual stress for treated and untreated samples

Furthermore, the maximum residual stress index Y of pre-treated specimens during early drying was greater than that of the untreated specimens, while it became small in comparison to the untreated specimens during the later period. It can be seen in Figure 2, some pits were broken and new micro-channels were created after the ultrasound treatment. The broken pits may improve the permeability and resulted in fast drying in the surface layer and greater shrinkage during the initial period, while the small Y during the later period was caused by the harden surface due to fast moisture removal during the initial period which prevented surface deforming (Li and Gu 1999).

Effect of Ultrasound Treatment on the Shrinkage Character

Figure 6 shows that total shrinkage occurred for both the treated and untreated specimens above the FSP due to the wood cell collapse (Liu 1994a, b, Wu 2005) and presented similar trends. The total shrinkage increased to some extent gradually and then recovered slowly as drying continued. The collapse did not continue to rise immediately above the FSP, which may be explained by two reasons. First, the collapse in the surface layer did not continue as the capillary tension disappeared due to no free water being present, and secondly, the collapsed cells recovered gradually due to the viscoelasticity property of the wood. The shrinkage curves of the treated and untreated specimens presented almost parallel results below the FSP (especially in MC below 24%), which is the obvious property for shrinkage curves with collapse (Liu 1994). Wood did not collapse anymore below the FSP due to there being no possible conditions resulting in cell collapse. Therefore, total shrinkage presented almost parallel outcomes for the treated and untreated specimens.

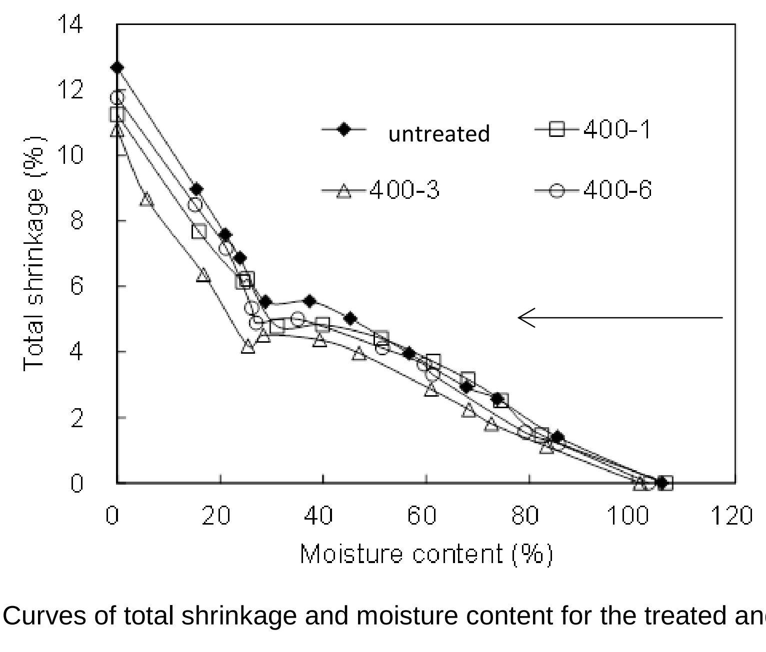

Fig. 6. Curves of total shrinkage and moisture content for the treated and untreated samples

The total shrinkage of specimens reduced by 11.3%, 14.9%, and 7.2%, respectively, after different treating durations, and the total shrinkage was smallest for the 3 h pre-treatment. Generally, wood collapse needs three conditions: the holes in the pits are small enough; the lumens are saturated, impermeable, and no bubbles are in the water; and the contact angles are small between the water and the cell wall. Some pits were broken (Fig. 2) after the ultrasound treatment, and new channels for moisture removal were created. The conditions causing wood collapse were damaged after ultrasound treatment. In addition, growths stresses may be released during the ultrasound treatment, which contributed to the collapsed cell's recovery. Therefore, ultrasound treatment may prevent wood collapse in varying degrees.

CONCLUSIONS

1. The drying rate increased after the ultrasound pre-treatment, and the 3-hour treatment tended to have the largest property improvement, followed by either the 1-hour or 6-hour treatment. Drying rate increased below the FSP.
2. The residual stress index Y of the treated specimens was greater than that of the untreated specimens during the initial period and they all decreased during the later period of the drying process. The residual stress index Y was smallest in the 3 h treatments. The modified wood micro-structures by ultrasound treatment affected the development of visco-elastic creep strain and mechano-sorptive strain and finally reduced residual stress of wood.
3. Total shrinkage of specimen treated by ultrasound decreased compared with the untreated specimen, and shrinkage decreased by 14.9% for the specimens after the 3 h treatments.
4. The pit membranes broke down, and there were some micro-channels on the pits. The micro-structure changes not only improved the permeability of the wood, the moisture migration paths, and the drying rate, but these changes also influenced the development of drying stress and cell collapse and recovery.

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