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INFLUENCE OF TEMPERATURE AND PRESSURE ON SUPERCRITICAL CO_2 DEWATERING OF BAMBOO STRIPS

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ABSTRACT

In this study, therepressure (15, 22.5, 30 MPa) and two temperature (45, 60°C) of $ScCO_2$dewatering were tested on Moso bamboo (*Phyllostachys edulis*) strips. The aim was to research the effects of these conditions on the dewatering rate, moisture distribution, and shrinkage of bamboo. The results showed that: *1)*The first cycle discharges the most water of all drying conditions. The most effective dewatering time consisted of a 15 min depressurization period and a 5 min discharge period. *2)*The ScCO2 dewatering rate of bamboo strips decreased with decreasing MC, with a maximum decrease of 78%.The maximum and minimum dewatering rates were 37.04%/h and 4.41%/h, respectively. The dewatering rate was synergistically affected by temperature and pressure, which increased significantly with pressure at 45°C, but was minimized at 60°C at 22.5 MPa. *3)*After dewatering, the moisture distribution in the bamboo strips shows a trend of higher moisture content(MC) in the middle and lower MC on both sides in the tangential and radial directions. *4)*Most of the bamboo strips produced shrinkage after the 1^{st} cycle of dewatering, and the overall shrinkage in the tangential direction was greater than that in the radial direction. The maximum tangential and radial shrinkage rations are 3.06% (22.5 MPa/45°C) and 0.94% (15 MPa/60°C), respectively.

KEYWORDS:Bamboo, supercritical CO_2 dewatering, temperature, pressure, dewatering rate, shrinkage ratio.

INTRODUCTION

As a green, fast-growing, high-quality non-timber forest resource, bamboo has won the favor of the industry and consumers because of the economical, ecological and cultural values. Currently, the global bamboo is mainly distributed in the regions of Asia, South America, Africa and Oceania(Fei et al. 2022; Wang et al. 2014; Zhang et al. 2017).Fresh bamboo has a high MC, and it needs to be dewatered promptly after harvesting to prevent mold, discoloration, and other issues caused by microbial attack. This preparation is crucial for storage, transportation, and subsequent processing(Lv et al. 2019; Singhal et al. 2022; Lv et al. 2018). Bamboo is usually dried by airdrying and conventional kiln drying, but the drying period is long and prone to deformation, cracking and discoloration, etc.(Liu and Yang 2024).Thus, exploring rapid and efficient drying methods for fresh bamboo has been of interest to industry.

Supercritical CO_2 ($ScCO_2$) is widely used as a non-toxic and environmentally friendly solvent in chemical extraction industry and research. Supercritical CO_2 ($ScCO_2$) has excellent diffusivity, allowing it to effectively penetrate porous materials. As pressure is lowered, the CO_2 phase shifts from supercritical fluid to gas, causing a significant volume change that expels liquid water in the wood cell lumina enabling rapid dewatering.

Foreign scholars have carried out experiments on the dewatering of wood using $ScCO_2$(Franich et al. 2020; Zhang et al. 2019; Aggarwal et al. 2019; Franich et al. 2019; Newman et al. 2016; Dawson et al. 2015). This technique mainly utilizes the excellent diffusion properties of $ScCO_2$, which can rapidly penetrate the wood and dissolve the water and solutes under the supercritical state.When the pressure of $ScCO_2$ drops below the critical point, $ScCO_2$ becomes gases and the volume becomes larger to discharge theliquid water in the wood cell cavities. Studies have shown that $ScCO_2$dewatering rate is fast and can improve the permeability of the wood and maintain the integrity of the cell structure. But this method can only remove the free water in the wood, and the wood moisture content (MC)can be reduced to near the saturation point of the fiber(FSP)(Franich et al. 2020; Zhang et al. 2019; Dawson et al. 2015). Regarding the study of bamboo $ScCO_2$dewatering, only Silviana et al. (2014) and Zhang et al. (2022) have carried out experiments on the dewatering of bamboo fibers.In addition, Liu et al. (2023)studied the dewatering characteristics of bamboo strips of different lengths in a $ScCO_2$single cycle and compared them with conventional kiln drying methods. They found that bamboo strips were more rapidly dewatered by $ScCO_2$(Liu et al. 2024).

Bamboo and wood components are similar, but there are obvious density and MC differences along the radial direction of bamboo(Lv et al. 2018). In addition, the above studies did not carry out a systematic study on the effects of temperature, pressure and emissiontime on the $ScCO_2$dewatering process, moisture transfer, distribution and deformation of bamboo. Therefore, this study conducts seven dewatering cycles on bamboo strips under two temperature gradients (45°C and 60°C) and three pressure gradients (15 MPa, 22.5 MPa, and 30 MPa). The effects of these dewatering conditions on the dewatering rate, moisture distribution, and deformation of bamboo are comparatively analyzed. This provides technical support and reference for developing rapid dewatering technology for fresh bamboo.

MATERIALS AND METHODS

Experiment material

Moso bamboo (*Phyllostachys edulis*), wasobtained from Shunchang, Fujian, China, has an average initial MC of about 100%. The raw bamboo was split into bamboo strips, and then were cut into uniform size of 150 mm (L) × 50 mm (T), totaling 24 strips, and numbered into 6 groups for the $ScCO_2$dewatering test. Each bamboo strip was marked at the middle(a), sub-middle(b), and end(c) to determine the cutting positions. Lines and dots were drawn along the tangential and radial directions on the right ends to measure dimensions(Fig. 1b).

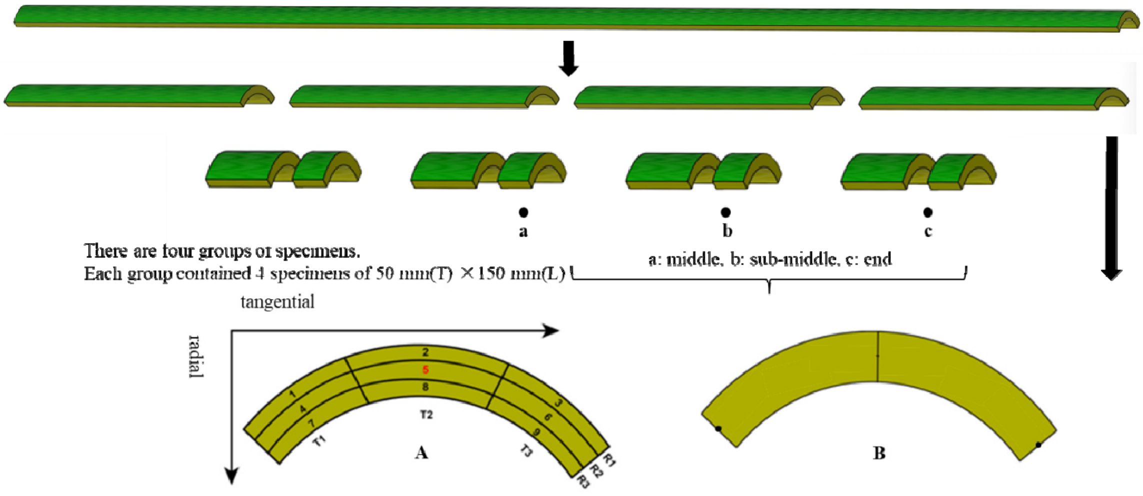

Fig. 1: Schematic diagram of bamboo sampling.

Equipment and instruments

The equipment used in this study is a $ScCO_2$ dewatering plant (HM120-50-025 Haian Hongmai Machinery Co., Ltd, Nantong, Jiangsu, China). The $ScCO_2$dewatering plant is shown in Fig. 2.The plant is composed of a CO_2 storage bottle (1), a cooling exchanger (2), a pressure pump (3), a 5L (ϕ100) dewatering vessel (4), and two adsorption vessels (5 and 6).The liquid CO_2(food grade,> 99.9%; sourced from http://www. cygyqt.cn/) was supplied to the dewatering plant from the storage bottle.The pressure in the dewatering vessel was controlled by the pressure pump from atmospheric (0.1MPa) to 30MPa, and the temperature of the dewatering vessel was regulated by a heated mantle from normal temperature to 60°C. Other equipments are electronic balance (JA21002, Shanghai Liangping Instrumentation, 1200g/10mg), vernier caliper (CD-20CPX, Mitutoyo, Japan, 0-200mm, 0.01mm).

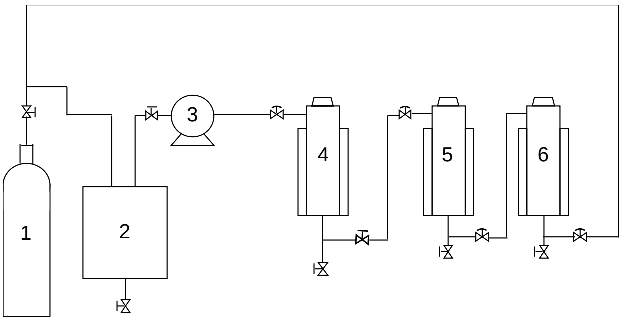

Fig. 2:Scheme of apparatus used for bamboo strip supercritical CO_2 dewatering.

Experimental method

Supercritical CO_2dewatering of bamboo

Firstly,the mass and tangential and radial linear dimensions of the specimen were measured, and then 4 bamboo pieces were put into the dewatering vessel (4), and temperature and pressurewere set (Tab. 1) after sealing the dewatering vessel.Then CO_2 gas was added into the dewatering vessel.Utilized the pressurized pump to elevate the pressure of CO_2, so as to make it into a supercritical state and reach the set temperature and pressure.Held the temperature and pressure of $ScCO_2$ for 15 min, so that it can fully contact with the bamboo(holding time). After the end of the pressure preservation, slowly decompression for about 15 min to 0.1 MPa, and then took out the specimens from the dewatering vessel, and measured the mass, tangential and radial dimensions at once.After that, the specimens were put into a small tray with a lid that was relatively sealed, and then the specimens were rest for 20 min at ambient temperature to make the CO_2 in the specimens fully discharged.During the emission period, the mass, tangential and radial dimensions of the specimen were measured every 5 min, and a dewatering cycle was completed. The above cycle was repeated seven times. After completing the seventh dewatering cycle, samples were cut at positions a, b, and c as shown in Fig. 1, with each position yielding a 10 mm (L) small bamboo strip. These strips were then quickly split into nine small bamboo blocks (Fig. 1a) and immediately placed in sealed bags to prevent moisture evaporation. The MC of each small bamboo block was measured using the drying method (GB/T 1927.4: 2021), which was used to analyze the moisture distribution and transfer regularity after dewatering.

Tab. 1: $ScCO_2$ dewatering parameters in each cycle.

Experimental group	1	2	3	4	5	6
Pressure (MPa)	15	15	22.5	22.5	30	30
Temperature (°C)	45	60	45	60	45	60
Pressurization time (min)	8	8	8	8	8	8
Decompression time (min)	15	15	15	15	15	15
Holding time (min)	15	15	15	15	15	15
Emission time (min)	20	20	20	20	20	20
Cycle number (times)	7	7	7	7	7	7

Determination of bamboo dewatering rate

The rate of bamboo dewatering was calculated according to Eq. 1:

$$R = \frac{MC_i - MC_f}{t} \quad (1)$$

where:$R$is dewatering rate (%/h), MC_i is initial MC of the specimen (%); $MC_f$is final MC of the specimen (%),t is dewatering time of the specimen (h).

Determination of dry shrinkage of bamboo

The tangential and radial dimensions of the right end of the sample(Fig. 1b) were measured at the end of each dewatering cycle. The tangential and radial shrinkage rates were calculated according to Eq. 2:

$$\beta = \frac{L_i - L_f}{L_i} \times 100\% \quad (2)$$

where:βistangential and radial drying shrinkage rate (%),$L_i$istangential and radial dimensions before dewatering(mm),$L_f$istangential and radial dimensions after dewatering(mm).

RESULTS AND DISCUSSION

Changes in specimen mass during dewatering

Fig. 3 shows the mass change curves of the specimens during the dewatering process for six temperature and pressure conditions. Among them, initial to 0 min is the decompression process in the dewateringvessel, and 0 to 20 min is the emission process after the specimens were removed.

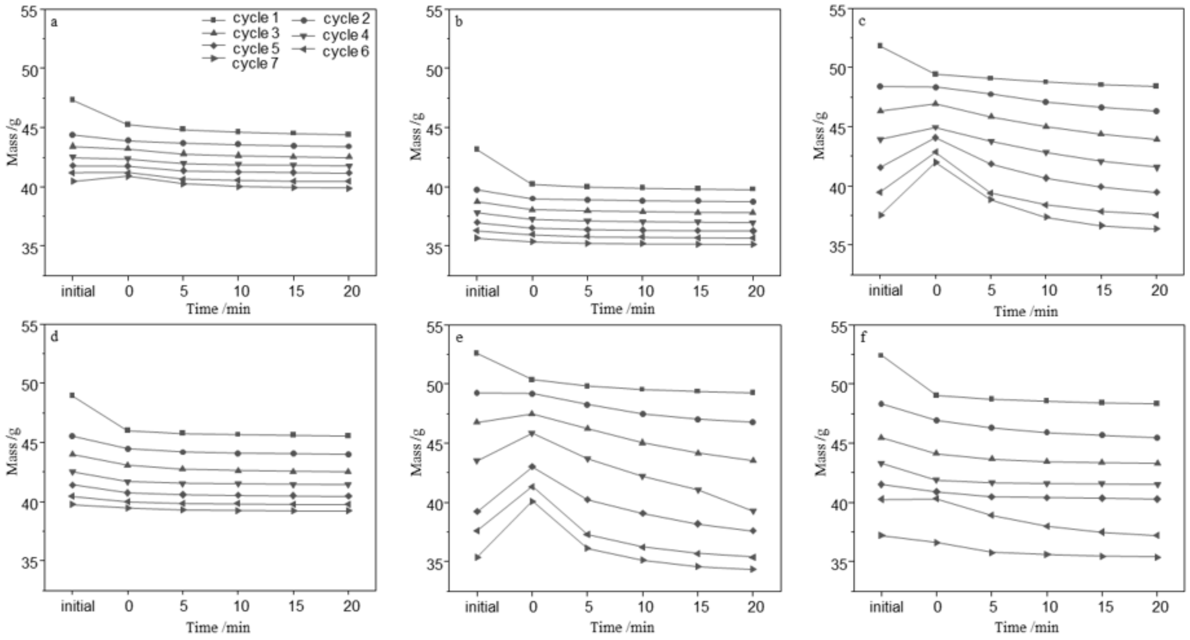

Fig. 3:Change curves of specimen mass during dewatering: a)15MPa/45°C, b) 15MPa/60°C,c) 22.5MPa/45°C,d)22.5MPa/60°C,e)30MPa/45°C,f)30MPa/60°C. Initial to 0 -decompressionprocess; 0 to 20 min - CO_2restingprocess.

The greatest mass loss of bamboo strips was observed during decompression in the first dewatering cycle, with the maximum mass loss was 4.07 g at 30 MPa/45°C and the minimum was 2.91 g at 15 MPa/45°C. Since the CO_2 within the specimens is not completely expelled during the decompression process, a small amount of water continues to be carried out by the CO_2 during the emission period, causing the specimens mass to decrease slowly. Comprehensively analyzing the dewatering cycle of each test condition, it was found that the mass of the specimens decreased the most in the first 5 min of the emission process, which accounted for about 50% of the whole emission process. The supercritical CO_2 penetrates the bamboo and forms bubbles as the pressure decreases. Under the pressure gradient and the force of the bubbles, the water in the bamboo is expelled from the cell cavities. Starting from the 2nd dewatering cycle, the mass reduction of specimens during the decompression process became slower but still higher than that of the emission process, so most of the water in the bamboo was discharged during the decompression process in each dewatering cycle. However, under the conditions of 22.5 MPa/45°C (Fig. 3c) and 30 MPa/45°C (Fig. 3e), the mass of the specimens became larger after the decompression process starting from the 3rd dewatering cycle, which was supposed to be due to the fact that the CO_2 penetrate into the interior of the bamboo was not discharged in time, which indicated that the 15 min decompression was not enough, and the water in the bamboo was discharged in the subsequent emission process. Therefore, 15 min of decompression and 5 min of emission time (20 min) is the most effective dewatering time in this study.

Bamboo ScCO$_2$dewatering rate

Fig. 4a shows the curves of the MC of bamboo strips with the dewatering cycle under six dewatering conditions, and Fig. 4b shows the relationship between dewatering rate and temperature and pressure. Regression processing was performed on each curve in Fig. 4a, and the dewatering rates (Tab. 2) for different MC ranges of the six dewatering conditions could be calculated based on the corresponding regression equations.

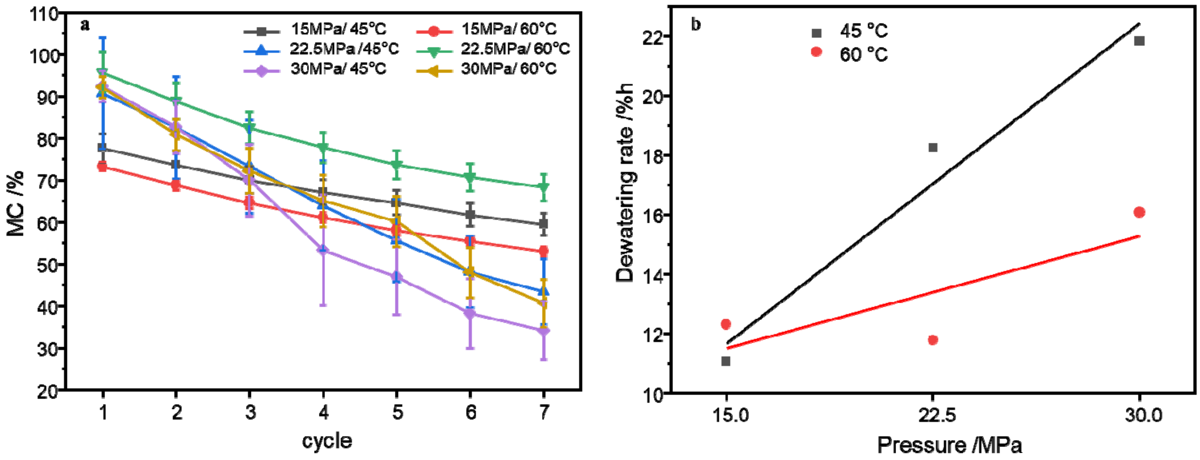

Fig. 4: a) MC curves of bamboo strips during dewatering process, b)relationship of average dewatering rate and pressure and temperature.

Tab.2:Dewatering rate of bamboo under six dewatering conditions.

MC range	Dewatering or drying rate (%/h)					
	15MPa/ 45□	15MPa/ 60□	22.5MPa/ 45□	22.5MPa/ 60□	30MPa/ 45□	30MPa/ 60□
110-100	/	/	28.25	20.83	37.04	21.51
100-90	/	/	23.04	16.58	30.30	19.05
90-80	20.83	25.57	19.46	9.59	25.64	17.09
80-70	7.83	12.72	16.84	6.74	22.22	15.50
70-60	4.59	6.55	14.84	5.20	19.61	14.18
60-50	/	4.41	13.26	/	17.54	13.07
50-40	/	/	11.99	/	15.87	12.12
40-30	/	/	/	/	14.49	/
Mean	11.08	12.31	18.24	11.79	22.84	16.07

From Fig. 4a and Tab. 2, it can be seen that the dewatering rate of bamboo is affected by the MC, and the dewatering rate decreases with the decrease of MC under the same condition (Yang 2021; Yang et al. 2021). For example, under the condition of 30 MPa/45°C, the dewatering rate of bamboo strips is as high as 37.04%/h when the MC is between 110% and 100%. As the MC decreases to 40%-30%, the dewatering rate drops to 14.49%/h, a reduction of 61%. The maximum decrease of dewatering rate with decreasing MC was 78% (15 MPa/45°C) and the minimum decrease was 44% (30 MPa/60°C). The dewatering rate was synergistically affected by temperature and pressure. At 45°C, the dewatering rate of each MC stage increased with the increase of pressure, but at 60°C, the dewatering rate was the minimum at 22.5 MPa. The average dewatering rate of bamboo strips showed the same regular (Fig. 4b). Increasing the pressure can increase the solubility of CO_2 in water and improve the permeability of bamboo, which is favorable to increase the dewatering rate (Franich et al. 2014). Increasing the temperature promotes the diffusion ability of CO_2 in water, but decreases its solubility in water (Hou et al. 2012), which in turn is unfavorable for water removal. Therefore, the rate of dewatering is synergistically affected by pressure and temperature on the diffusion and solubility capacities of CO_2 in water (Franich et al. 2014).

Moisture content distribution

Fig. 5 shows the distribution of MC along the tangential and radial directions in the middle, sub-middle and end of the bamboo strips after ScCO$_2$dewatering. From Fig. 5a-c, the trend of tangential moisture distribution after ScCO$_2$dewatering was similar, generally showing highMC in the middle and slightly lower on both sides.

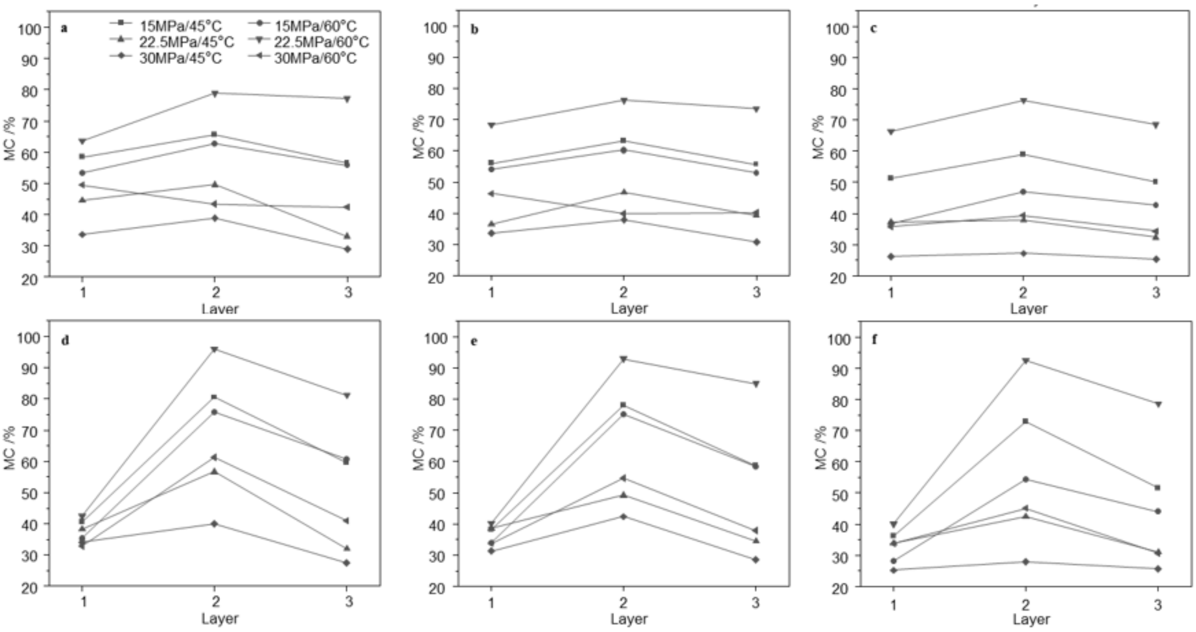

Fig. 5: Moisture contentalong bamboo strips tangential direction after ScCO$_2$ dewatering in the middle(a), sub-middle(b), and end(c);along radial direction in the middle(d),sub-middle(e); and end(f).

Under the condition of 30 MPa/60°C, the difference in tangential MC between the middle and the sub-middle was the smallest, which was 7.05% and 6.40%, respectively; and the difference in tangential MC between the ends was the smallest under the condition of 30 MPa/45°C, which was 1.91%. As can be seen from Fig. 5f, the difference of moisture in the radial direction was obvious, with the highest MC in the bamboo internal (R2) layer, followed by the bamboo yellow (R3) layer, and the lowest MC in the bamboo green layer (R1). The smallest difference in radial MC was found in the middle, sub-middle, and end under the condition of 30 MPa/45°C, which were significantly higher than that of the tangentialdirection, with the differences of 12.58%, 13.76%, and 2.63%, respectively. The radial moisture distribution was similar to the results of the previous study, which was attributed to the fact that bamboo has a special gradient organizational structure in the radial direction (Vorontsova et al. 2016; Chen et al. 2023). It can also be seen that the radial MC difference decreases with the decrease of MC.

Fig. 6 shows the MC of the middle, sub-middle and end of the bamboo strips after dewatering and the average values of the three positions. After $ScCO_2$ dewatering, there was no significant difference in moisture distribution between the middle and sub-middle parts of the bamboo strips in the length direction, but the MC at the end of the bamboo strips was significantly lower than that at the central part under the four dewatering conditions ($P<0.05$). Therefore, it was inferred that moisture was more easily discharged at the end along the fiber direction.

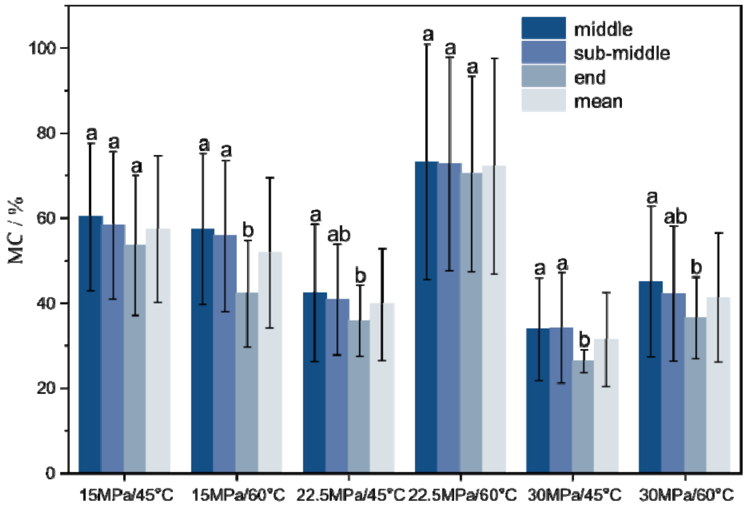

Fig. 6:MC in the middle, sub-middle and end of the bamboo strips and their average values after $ScCO_2$ dewatering (different letters represent significant differences according to Duncan's test, $P<0.05$).

Shrinkage

Wood drying shrinkage usually occurs below the FSP (Zheng et al. 2024; Peng et al. 2023). However, some woods also shrink at MC above the FSP, a phenomenon known as collapse, which is mainly caused by capillary tension due to free water migration exceeding the transverse tensile strength of the cell wall (Yang et al. 2022; Liu et al. 2021). Unlike wood, bamboo shrinks from the beginning of drying, and shrinkage occurs mainly in the radial direction when the MC drops below the FSP (Sun et al. 2006). The average FSP of Moso bamboo is 34.99% (Vetter et al. 2015). As can be seen from Fig. 4a, the MC of the specimens in the first six cycles of dewatering is higher than the FSP, and the MCof the specimens at 30 MPa/45°C is clearly close to the FSP after the seventh cycle. Figs. 7a,b show the drying shrinkage of bamboo strips in tangential direction and radial direction, respectively, at the end of each dewatering cycle. Most of the bamboo stripsshrinkage in the tangential direction after the first dewatering cycle, and the tangentialshrinkage of bamboo strips showed a slow increasing trend in the subsequent dewatering cycles (Fig. 7a). At the end of the 7th dewatering cycle, the largest shrinkage (3.06%) was observed for the 22.5 MPa/45 °C condition, followed by the 15 MPa/45°Ccondition (1.96%), and the smallest shrinkage (0.36%) was observed for the 30 MPa/60°C condition. However, the radial shrinkage of the specimen was significantly smaller than that in the tangential direction, with the maximum shrinkage of 0.94% (15 MPa/60°C), at which time the MC at the end was already lower than the FSP (Fig. 5f), and the bamboo strips might have produced dry shrinkage below the FSP, and the shrinkage at this time accumulated the collapse of the previous period, but the shrinkage of the other conditions were all less than 0.5%. In addition, unlike the tangential direction, the specimens produced expansion in the radial direction during dewatering at 15 MPa/45°C and 30 MPa/60°C, and the expansion rate was less than 0.3% at the end of dewatering. The shrinkage produced above the FSP during $ScCO_2$dewatering of bamboo strips is presumed to be the collapse of some cells (Wang et al. 2022; Sun et al. 2006), which is caused by the migration of free water to deform the cells. The swelling produced in the radial direction at the end of the bamboo strips may be due to the high pressure of $ScCO_2$, and the pressure resulted in radial deformation of cells or tissues.

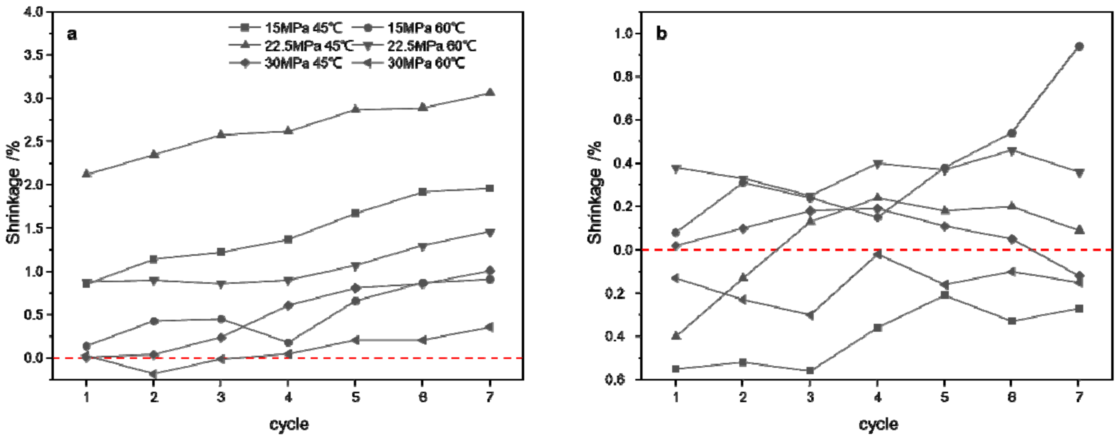

Fig. 7:a) Shrinkage in tangential direction, and b)radial direction of bamboo strips during $ScCO_2$ dewatering.

CONCLUSIONS

(1) During the decompression and emission process of $ScCO_2$dewatering, $ScCO_2$penetrated into the interior of the bamboo strips turned into bubbles, and water was discharged out of the bamboo strips after volume expansion. Under all conditions, the most water is expelled during the first cycle. The most effective dewatering time includes a 15 min decompression period and a 5 min emission period.*(2)*The bamboo strips were dewatered quickly by $ScCO_2$, with the maximum and minimum dewatering rates of 37.04%/h and 4.41%/h, respectively, and the dewatering rate decreased with the decrease of MC, with the maximum decrease of 78%. The dewatering rate was synergistically affected by temperature and pressure, and the dewatering rate increased significantly with pressure at 45°C, but the dewatering rate of 22.5 MPa was the smallest at 60°C.*(3)* The moisture distribution of bamboo strips after $ScCO_2$ dewatering was relatively uniform along the tangential and length

directions, with the MC in the middle of the tangential direction being slightly higher than that on both sides, and that in the end of the length direction being slightly lower than that in the middle. The distribution of radial MC varied significantly, with the bamboo internal layer being the highest, the bamboo yellow layer the next highest, and the bamboo green layer the lowest.*(4)* The maximum shrinkage of bamboo strips in the tangential and radial directions after ScCO$_2$dewatering was 3.06% (22.5 MPa/45°C) and 0.94% (15 MPa/60°C), respectively, with the tangential direction being larger than the radial direction. The radial direction of the bamboo strips expanded under some dewatering conditions, but the expansion ratiowas less than 0.3%.

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