

# Comparative Study of the Hydrothermal Softening Characteristics of Heartwood and Sapwood

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## Abstract

This study was conducted to investigate the hydrothermal softening characteristics of heartwood and sapwood from *Catalpa bungei*. The viscoelastic properties were investigated by dynamic mechanical analysis (DMA Q800) with a tension and submersion mode. Heartwood and sapwood specimens were tested under water-saturated conditions in the radial and tangential directions. The measured temperature ranged from 25°C to 79°C with three heating rates: 0.5°C/min, 1°C/min, and 2°C/min. The results show that heartwood specimens presented a higher storage modulus ( $E'$ ) and a lower reduction of wood rigidity ( $\Delta E'$ ) than sapwood specimens. A difference was also observed in loss modulus ( $E''$ ), and the glass transition temperature of lignin in heartwood specimens was nearly 2°C to 3°C lower than that in sapwood specimens. This could be caused by lower lignin content and higher levels of extractives of sapwood in comparison with heartwood. Additionally, a circular arc curve of  $E'$  versus  $E''$  was formed in heartwood specimens, irrespective of heating rate. These findings suggest that heartwood presented better hydrothermal softening characteristics than sapwood. Compared with the tangential specimens, the radial specimens revealed higher  $E'$  and lower  $\Delta E'$ , indicating that the radial specimens were less influenced by hydrothermal treatment.

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An understanding of the hydrothermal softening characteristics of wood is essential in the processing and manufacturing operations in the wood industry. In the course of hydrothermal treatment, the viscoelasticity of wet wood is essential in many sectors of the wood industry, such as veneer peeling, bending, drying, panel pressing, and so on (Sandberg et al. 2013; Zhan et al. 2018a, 2018b). Thus, it is of practical importance to understand the concomitant changes of viscoelastic properties during the hydrothermal treatment, which could be helpful for improving utilization in the form of higher value-added products as well as for optimizing manufacturing operations and increasing the production efficiency of the wood industry.

As water plays a role of plasticizer, its entrance into wood cell walls may cause a decrement of wood stiffness and increment of viscosity (Engelund and Salmén 2012, Zhan et al. 2018a). Thus, water-saturated wood has less stiffness and more damping than dry and moist wood. Under water-saturated conditions, the viscoelastic properties of wet wood reflect to a large extent the properties of lignin (Salmén

1984, Placet et al. 2007, Furuta et al. 2010, Navi and Sandberg 2012). The softening temperature of water-

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Forest Prod. J. 70(3):243–248.

doi:10.13073/FPJ-D-20-00013

saturated wood, appearing between 50°C and 100°C, corresponds to the glass transition temperature ( $T_g$ ) of in situ lignin (Salmén 1984, Placet et al. 2007, Furuta et al. 2014, Song et al. 2014). In general, the  $T_g$  of lignin could characterize the softening point of wet wood. Therefore, precise information on the  $T_g$  of water-saturated wood is helpful to reduce unnecessary energy consumption and increase the production efficiency of the wood industry. Also, the viscoelastic properties of wet wood depend on the soaking time in water, so the heating rate becomes more and more important in the same measured temperature range.

In most tree species, after cambial initials are differentiated to xylem cells, mature sapwood is transformed into heartwood (Kampe and Magel 2013, Song et al. 2014). Due to the different chemical compositions and anatomical structures in heartwood and sapwood, these wood tissues also differ in their reaction to hydrothermal treatment. Nevertheless, most research on the viscoelastic properties in hydrothermal conditions focuses only on heartwood; less work has been done with sapwood. Song et al. (2014) reported the viscoelastic properties of heartwood and sapwood from Chinese fir (*Cunninghamia lanceolata*), and pointed out that sapwood had a lower softening temperature and required less activation energy than heartwood, indicating that heartwood of Chinese fir had more extractives. In addition, many studies have shown that the viscoelastic properties of wood differ in the radial and tangential directions (Backman and Lindberg 2001; Reiterer et al. 2002; Placet et al. 2007, 2008; Redman et al. 2011; Li et al. 2018). Backman and Lindberg (2001) measured the viscoelastic properties of Scots pine (*Pinus sylvestris*) in the transverse directions and found that the tangential direction showed a lower storage modulus and a higher loss peak temperature than the radial direction. A similar observation was made for hardwood, including oak (*Quercus sessiliflora*), beech (*Fagus sylvatica*), and poplar (*Populus sp.*), and for softwood spruce (*Picea abies*; Placet et al. 2007).

*Catalpa bungei* is an economically important native hardwood in China and is widely employed in solid wood furniture, musical instruments, interior decorations, and so on. To date, little to no research has been conducted on the hydrothermal softening characteristics of heartwood and sapwood in this species. Therefore, information on wood softening characteristics of heartwood and sapwood from *C. bungei* is of particular interest. The objective of this study was to investigate the differences in the response of heartwood and sapwood across the grain to the hydrothermal conditions in terms of their viscoelastic properties. Results could contribute to a further understanding of the hydrothermal softening characteristics of *C. bungei* and provide a scientific basis for better utilization of precious hardwood.

## Materials and Methods

### Wood materials

Specimens were obtained from a 44-year-old *C. bungei* tree. Heartwood and sapwood zones were identified through visual inspection, and the average air-dry densities were about 0.49 and 0.42 g/cm<sup>3</sup>, respectively. Clear wood specimens without any visible defects and knots were cut from the heartwood and sapwood parts along the two wood directions across the grain, i.e., the radial and tangential directions. Wood specimens were rectangular, with a cross-

section of 6 by 1.4 mm<sup>2</sup> and a length of 35 mm. Specimen nomenclature indicates the location and direction, with heartwood specimens in the radial and tangential directions named as H-R and H-T specimens, and sapwood specimens across the grain named as S-R and S-T specimens, respectively. All wood specimens were stored in distilled water at 25°C for more than 3 months until a constant mass was achieved.

### Experimental methods

Tests were performed in tensile mode on a dynamic mechanical analyzer (DMA Q800; TA Instruments) equipped with submersible configuration. The submersion clamp was fixed in such a way that water-saturated specimens were put into a stainless-steel cup that maintained the specimens immersed and tested directly in water. During the DMA test, the tensile stress was applied along the length of wood specimens. The parameters of the storage modulus ( $E'$ ) and loss modulus ( $E''$ ) were automatically recorded, and the  $T_g$  of the specimen was defined as the temperature corresponding to the maximum  $E''$ . Temperature scans were performed in the range from 25°C to 79°C with heating rates of 0.5°C/min, 1°C/min, and 2°C/min. The span between the supporting points was fixed at 15 mm. A preload force of 0.01 N and a force track of 125 percent were applied. To ensure that all tests were carried out within the linear viscoelastic region, a sinusoidal displacement was applied with an amplitude of 15 μm at a frequency of 1 Hz. Three replicates were performed for each condition.

## Results and Discussion

### Storage modulus in heartwood and sapwood

Figure 1 shows  $E'$  as function of temperature in heartwood and sapwood specimens across the grain at different heating rates. It shows that the  $E'$  appears to decline gradually with increasing temperature in heartwood and sapwood specimens, regardless of the grain orientation. The wood cell wall polymers are provided with heat energy for segmental motion, resulting in the decrement of wood stiffness (Engelund and Salmén 2012, Li et al. 2018). In addition, the reduction of wood rigidity may be explained by the partial degradation of hemicelluloses during the hydrothermal treatment (Assor et al. 2009, Furuta et al. 2010, Salmén et al. 2016).

Table 1 provides the initial and final values of  $E'$  in heartwood and sapwood specimens across the grain at different heating rates. For each wood specimen, the initial value of  $E'$  decreased slightly with increasing heating rate, while the final value of  $E'$  was very close at any heating rate. Under hydrothermal conditions, the various changes in the amorphous polymers led to substantial reorganization within wood cell walls, so that the rigidity of wood was eventually almost restored to its initial value (Assor et al. 2009). As expected, the  $E'$  of specimens showed a clear difference in the radial and tangential directions, irrespective of the sampling location and heating rate. These findings were similar to previous reports (Backman and Lindberg 2001, Placet et al. 2007, Redman et al. 2011) in which results for the tangential direction revealed lower  $E'$  values than the radial direction.

In order to compare the changes of  $E'$  in heartwood and sapwood specimens, the parameters of  $\Delta E'$  and  $\Delta E'/\Delta t$  were calculated as

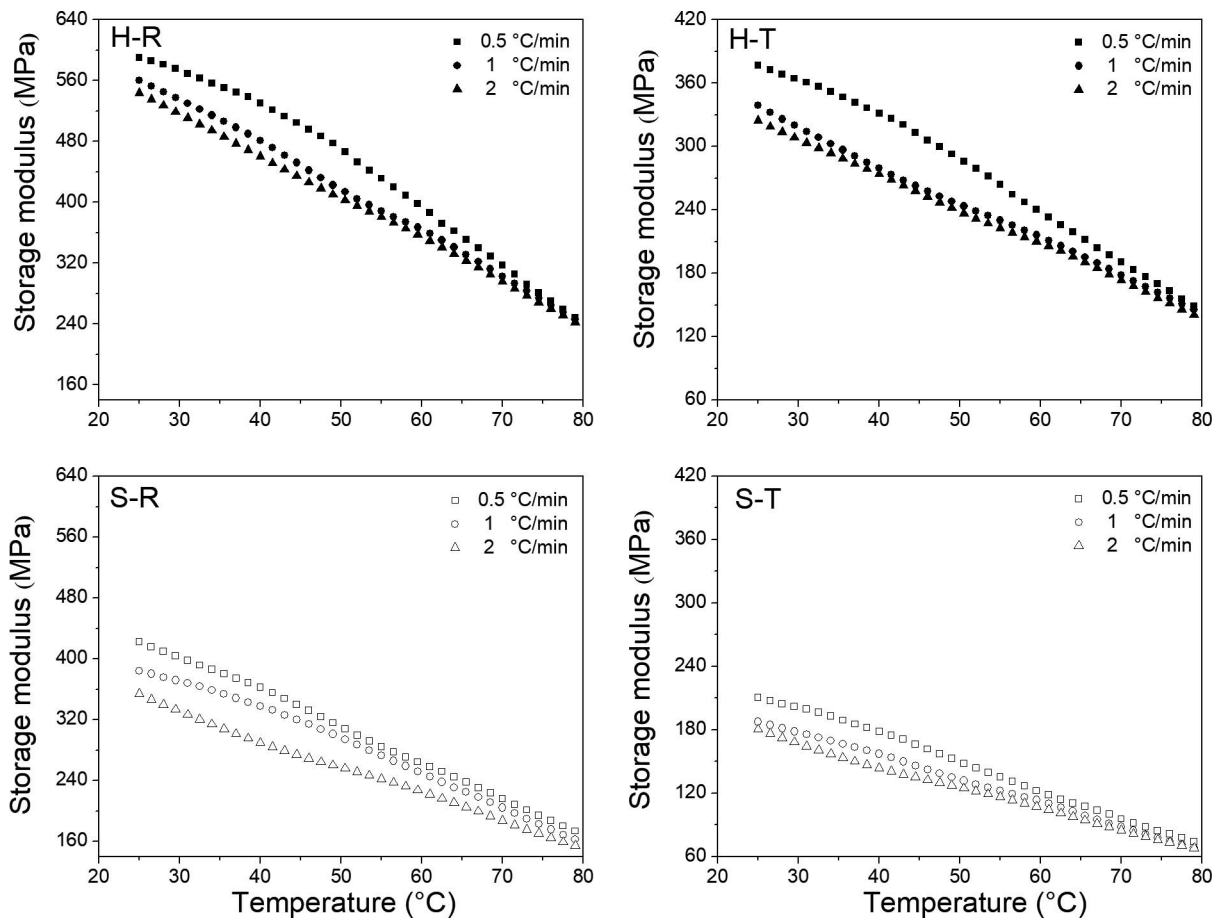


Figure 1.—Temperature dependency of the storage modulus in heartwood and sapwood specimens in the radial and tangential directions at different heating rates. H-R = heartwood specimen in the radial direction; H-T = heartwood specimen in the tangential direction; S-R = sapwood specimen in the radial direction; S-T = sapwood specimen in the tangential direction.

$$\Delta E' = (E'_{25} - E'_{79}) / E'_{25} \times 100\% \quad (1)$$

$$\Delta E' / \Delta t = \Delta E' / (54 / \nu) \quad (2)$$

Table 1.—Values of the storage modulus ( $E'$ ) in heartwood and sapwood specimens in the radial and tangential directions at different heating rates.<sup>a</sup>

| Heating rate<br>(°C/min) | Specimen | $E'$ (MPa) |       | $\Delta E'$ (%) | $\Delta E' / \Delta t$ (%) |
|--------------------------|----------|------------|-------|-----------------|----------------------------|
|                          |          | 25°C       | 79°C  |                 |                            |
| 0.5                      | H-R      | 590.1      | 248.3 | 57.9            | 0.54                       |
|                          | H-T      | 377.0      | 149.0 | 60.5            | 0.56                       |
|                          | S-R      | 422.5      | 173.7 | 58.9            | 0.55                       |
|                          | S-T      | 210.6      | 74.2  | 64.8            | 0.60                       |
| 1                        | H-R      | 560.3      | 245.9 | 56.1            | 1.04                       |
|                          | H-T      | 338.9      | 145.6 | 57.0            | 1.06                       |
|                          | S-R      | 384.5      | 162.7 | 57.7            | 1.07                       |
|                          | S-T      | 187.5      | 68.4  | 63.5            | 1.08                       |
| 2                        | H-R      | 543.8      | 242.2 | 55.5            | 2.06                       |
|                          | H-T      | 324.7      | 140.7 | 56.7            | 2.10                       |
|                          | S-R      | 354.0      | 154.7 | 56.3            | 2.09                       |
|                          | S-T      | 180.4      | 67.8  | 62.4            | 2.31                       |

<sup>a</sup> H-R = heartwood specimen in the radial direction; H-T = heartwood specimen in the tangential direction; S-R = sapwood specimen in the radial direction; S-T = sapwood specimen in the tangential direction;  $\Delta E'$  = the decrement of storage modulus;  $\Delta t$  = the time of experimentation.

where  $E'_{25}$  and  $E'_{79}$  indicate  $E'$  at the beginning and end of the measured temperature range, respectively;  $t$  represents the time of experimentation; and  $\nu$  represents the heating rate: 0.5°C/min, 1°C/min, and 2°C/min. As can be seen in Table 1, at a heating rate of 0.5°C/min, the  $\Delta E'$  values of H-R, H-T, S-R, and S-T specimens were 57.9, 60.5, 58.9, and 64.8 percent, respectively, and the  $\Delta E'$  values of H-R, H-T, S-R, and S-T specimens were 56.1, 57.0, 57.7, and 63.5 percent at 1°C/min, respectively. Clearly, the  $\Delta E'$  of heartwood and sapwood specimens decreased with increasing heating rate, regardless of the grain orientation. In the same range of measured temperatures, the lower the heating rate, the longer the heating time. The values of  $E'$  in wood specimens decreased with the prolonged heating time in water (Assor et al. 2009, Jiang et al. 2009, Engelund and Salmén 2012), resulting in the reduction of  $\Delta E'$  with an increasing heating rate. Interestingly, at each heating rate, the  $\Delta E' / \Delta t$  values of heartwood and sapwood specimens were very similar, regardless of the grain orientation. Also, the value of  $\Delta E' / \Delta t$  was directly proportional to heating rate. The greater the provided heating energy for segmental motions per unit time, the greater the value of  $\Delta E' / \Delta t$ .

At any heating rate, the  $\Delta E'$  values in heartwood specimens were slightly lower than those in sapwood specimens (Table 1). Accordingly, the  $\Delta E'$  values of H-R specimens were slightly lower than those of S-R specimens, and the  $\Delta E'$  values of H-T specimens were slightly lower than those of S-T specimens. The differences in  $\Delta E'$  between heartwood and sapwood might be explained by the different chemical compositions. Li et al. (2019a) pointed out that increasing amounts of tyloses were found from sapwood to heartwood in *C. bungei*. Therefore, abundant distribution of tyloses in heartwood might provide higher mechanical support and reduce wood permeability (De Micco et al. 2016), thus resulting in the lower  $\Delta E'$  of heartwood specimens.

The  $\Delta E'$  values of wood specimens in the tangential direction were different from the radial direction, regardless of the sampling location and heating rate. Obviously, the  $\Delta E'$  in the tangential direction was slightly higher than that in the radial direction, which is in agreement with the results of Placet et al. (2007) and Redman et al. (2011). The differences in  $\Delta E'$  of wood specimens across the grain might be related to variation in the chemical composition and anatomical structure (Backman and Lindberg 2001, Reiterer et al. 2002, Placet et al. 2007, Redman et al. 2011). It was assumed that greater amounts of tyloses were found in vessels in the radial plane of *C. bungei* wood studied here, resulting in a lower  $\Delta E'$  in the radial direction. Furthermore,

wood specimens in the tangential direction were found to be more sensitive to hydrothermal treatment compared with specimens in the radial direction (Placet et al. 2007, Redman et al. 2011, Li et al. 2019b).

### Loss modulus in heartwood and sapwood

The loss modulus ( $E''$ ) as function of temperature in heartwood and sapwood specimens in the radial and tangential directions at different heating rates is depicted in Figure 2. The results clearly show that water-saturated wood specimens were found to soften in the temperature range of 40.9°C to 60.2°C, corresponding to the relaxation of lignin (Salmén 1984, Olsson and Salmén 1997, Placet et al. 2007, Redman et al. 2011, Furuta et al. 2014). A relaxation of  $E''$  was observed in the H-R and H-T specimens at any heating rate, whereas relaxation of  $E''$  occurred in sapwood specimens only at 0.5°C/min. Yang and Li (2020) confirmed that sapwood has a lower lignin content and a higher benzyl alcohol extractive content as compared to heartwood of *C. bungei*, indicating that sapwood needed more energy to activate the softening process or the chain mobility of lignin. In addition, the prolonged heating time provides more heating energy for segmental motions in the wood cell wall, resulting in the observation of a relaxation in sapwood at lower heating rate.

Table 2 lists the  $T_g$  of lignin in heartwood and sapwood specimens across the grain at different heating rates. The

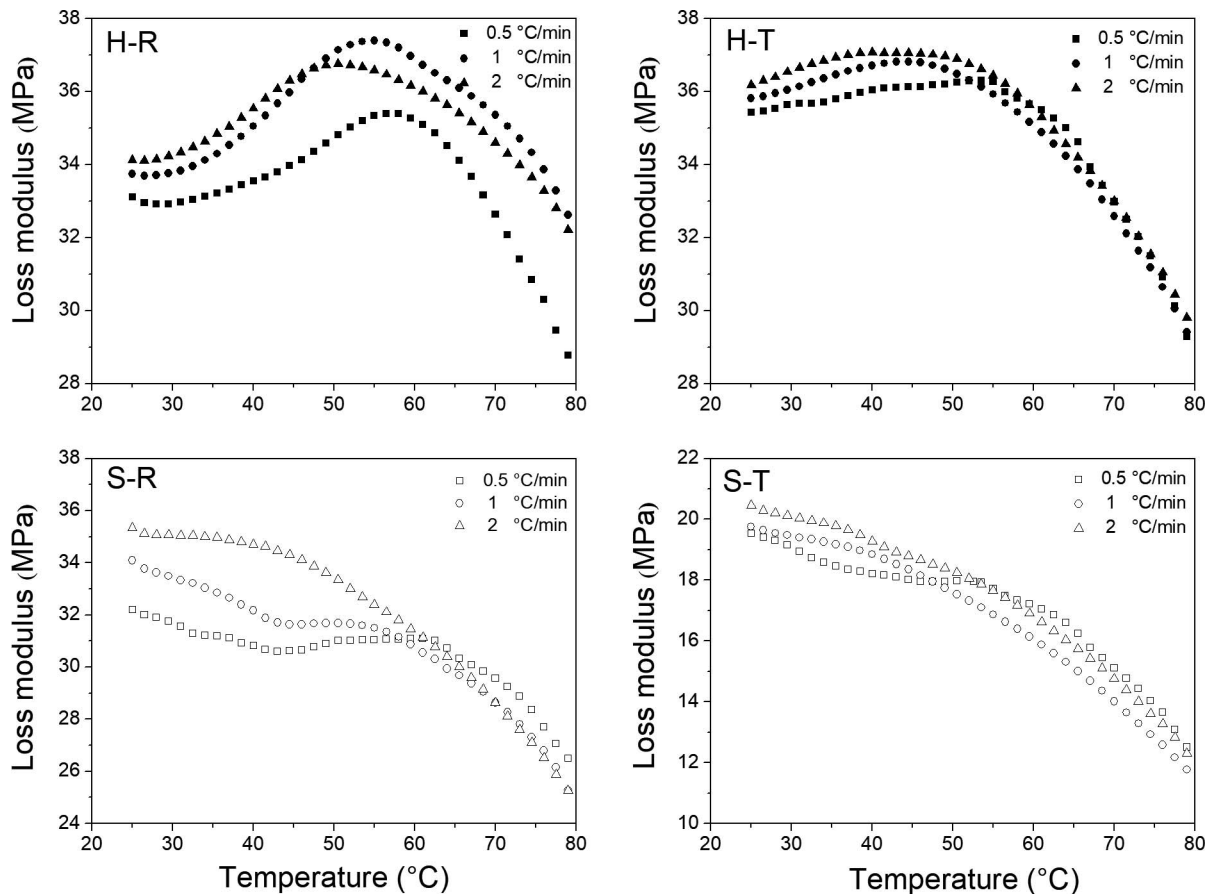


Figure 2.—Temperature dependency of the loss modulus in heartwood and sapwood specimens in the radial and tangential directions at different heating rates. H-R = heartwood specimen in the radial direction; H-T = heartwood specimen in the tangential direction; S-R = sapwood specimen in the radial direction; S-T = sapwood specimen in the tangential direction.

**Table 2.—The glass transition temperature of lignin in heartwood and sapwood specimens in the radial and tangential directions at different heating rates.<sup>a</sup>**

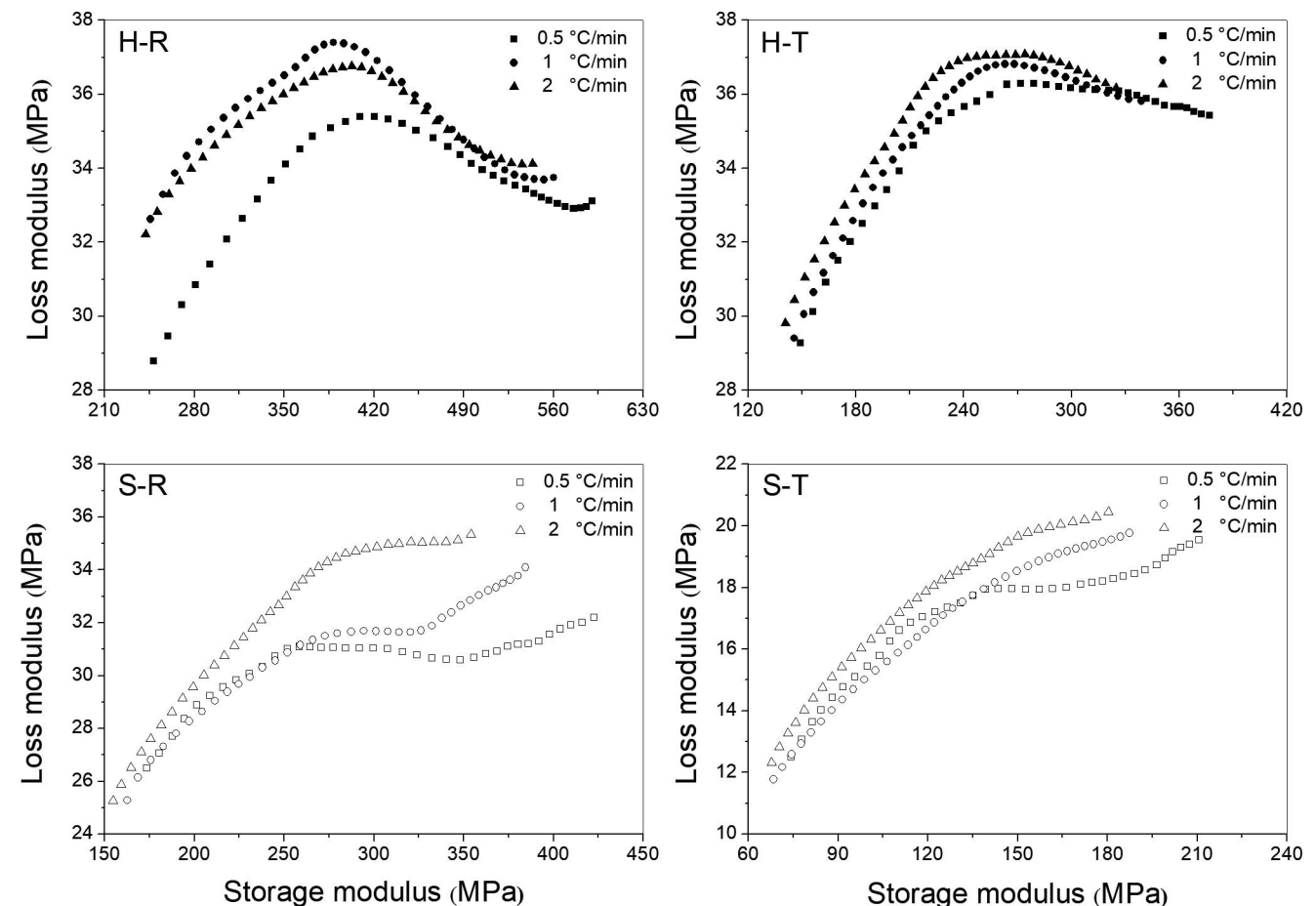
| Heating rate<br>(°C/min) | Glass transition temperature (°C) |            |            |            |
|--------------------------|-----------------------------------|------------|------------|------------|
|                          | H-R (SD)                          | H-T (SD)   | S-R (SD)   | S-T (SD)   |
| 0.5                      | 57.3 (2.5)                        | 52.2 (3.2) | 60.2 (0.4) | 54.1 (2.5) |
| 1                        | 54.8 (1.4)                        | 43.5 (3.6) | —          | —          |
| 2                        | 51.3 (1.8)                        | 40.9 (3.2) | —          | —          |

<sup>a</sup> H-R = heartwood specimen in the radial direction; H-T = heartwood specimen in the tangential direction; S-R = sapwood specimen in the radial direction; S-T = sapwood specimen in the tangential direction.

variability of results was calculated based on the standard deviations obtained from the average of three specimens for each test condition. The  $T_g$  values of lignin in the H-R and H-T specimens with heating rates of 0.5°C/min, 1°C/min, and 2°C/min were 57.3°C and 52.2°C; 54.8°C and 43.5°C; and 51.3°C and 40.9°C, respectively. Clearly, the  $T_g$  of lignin in heartwood decreased with increasing heating rate, irrespective of the grain orientation. With hemicelluloses partially removed, a more direct stress connection between cellulose and lignin becomes possible, and lignin was enabled to play a more direct role in the stress transfer in the matrix of the wood cell wall, resulting in a lower lignin

softening temperature (Salmén et al. 2016). In addition, the higher the heating rate, the greater the instability of internal structure of wood specimens (Zhan et al. 2018a, 2018b), resulting in wood softening in the lower temperature range.

At the heating rate of 0.5°C/min, the  $T_g$  of lignin in the H-R, H-T, S-R, and S-T specimens were 57.3°C, 52.2°C, 60.2°C, and 54.1°C, respectively. Accordingly, the  $T_g$  of lignin in heartwood was slightly lower than that in sapwood, irrespective of the grain orientation. Differences in the lignin softening of heartwood and sapwood might be related to the chemical compositions of *C. bungei* wood. It is well known that the extractives are strongly associated with the wood cell wall components, which would restrict wood softening (Masahiro et al. 2000, Kampe and Magel 2013). As discussed previously, the greater extractive content in sapwood obstructed its access to the plasticizer of water, and therefore more energy was needed to activate the chain mobility of its lignin. Furthermore, the higher lignin content of heartwood compared with sapwood is another reason that the lignin  $T_g$  in heartwood occurred at a lower temperature range. Also, the  $T_g$  of lignin in wood specimens showed a minor difference in the radial and tangential directions, in agreement with previous studies (Backman and Lindberg 2001, Redman et al. 2011, Li et al. 2019b). More tyloses were found in vessels in the radial plane of *C. bungei* wood,



**Figure 3.—Cole-Cole plot in heartwood and sapwood specimens in the radial and tangential directions at different heating rates. H-R = heartwood specimen in the radial direction; H-T = heartwood specimen in the tangential direction; S-R = sapwood specimen in the radial direction; S-T = sapwood specimen in the tangential direction.**

which may be a plausible explanation a higher lignin  $T_g$  in the radial direction relative to the tangential direction.

## Loss modulus versus storage modulus in heartwood and sapwood

Cole-Cole diagrams represent a convenient way to summarize the viscoelastic properties of wood materials and to visualize the relaxation mechanisms (Placet et al. 2007, Zhan et al. 2015, Li et al. 2019b). The viscoelastic data of  $E'$  and  $E''$  for the water-saturated heartwood and sapwood specimens in the transverse directions were plotted as Cole-Cole diagrams to show the glass transitions of lignin (Fig. 3). Clearly, a circular arc was observed in heartwood specimens, irrespective of heating rate and the grain orientation, while only one circular arc was found in sapwood specimens, at  $0.5^\circ\text{C}/\text{min}$ , irrespective of the grain orientation. As for *C. bungei* wood, sapwood has a lower lignin content and a higher extractive content as compared with heartwood. Therefore, sapwood specimens needed more energy to activate the wood softening process and resulted the circular arc losing its integrity at higher heating rates.

## Conclusions

The  $E'$  of heartwood and sapwood decreased gradually with increasing measured temperature. Heartwood specimens presented a higher  $E'$  and a lower reduction of wood rigidity ( $\Delta E'$ ) than sapwood specimens, regardless of heating rate. In comparison with sapwood, heartwood contained a higher lignin content and a lower content of extractives. Correlated to the chemical changes, a higher  $T_g$  of lignin in sapwood specimens was observed, indicating the restricted mobility of lignin in sapwood. Differences in viscoelastic behavior were also observed in the radial and tangential directions. Compared with the tangential specimens, the radial specimens revealed a higher  $E'$  and a lower  $\Delta E'$ , and had a higher  $T_g$  at the heating rate of  $0.5^\circ\text{C}/\text{min}$ . Additionally, a circular arc curve of  $E'$  versus  $E''$  was formed in sapwood specimens at  $0.5^\circ\text{C}/\text{min}$  and in heartwood specimens irrespective of heating rate.

## Acknowledgment

This research was sponsored by the Fundamental Research Funds of Chinese Academy of Forestry (CA-FYBB2020MA001).

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