

Article

Changes in Vibrational Characteristics of Dry-Thermal-Treated Japanese Cedar During Cyclic Moisture Change Tests

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Abstract

Changes in wood moisture content significantly affect its dimensions, mechanical properties, and vibrational characteristics. Thermal treatment is one of the most convenient approaches for improving the moisture resistance of wood; however, the effects of treatment conditions on moisture content and vibrational characteristics after short-term cyclic moisture absorption have not been clearly investigated. In this study, dry thermal treatment at 160–220 °C for three different durations was applied to Japanese cedar specimens. Higher thermal treatment temperatures and longer treatment times decreased the equilibrium moisture content (EMC). The fundamental resonant frequency of the free–free flexural vibration (f_1) increased with increasing treatment temperature, whereas it decreased over a longer duration. All specimens were subjected to three cycles of moisture change tests from 60%RH to 98%RH at 40 °C to track the change in moisture content, f_1 and its loss tangent ($\tan\delta$). The specimens treated at higher temperatures maintained a lower moisture content and higher f_1 . Under most treatment conditions, the moisture content at 98%RH increased from the first to the second cycle and remained constant in the third cycle. On the other hand, the resonant frequency at 98%RH remained unchanged from the first to the second cycle but increased in the third cycle. This indicates that the moisture surface became saturated in the second cycle, and moisture diffusion from the surface to the inside of the specimen increased with the number of cycles. Near-infrared absorption revealed that high-temperature treatment caused thermal decomposition of hemicellulose and an increase in apparent crystallinity due to a reduction in the amorphous region of cellulose. These changes enhance the hydrophobicity of the cell wall, contributing to moisture resistance and vibrational stability.

Keywords: thermal treatment; flexural vibration; moisture resistance; near-infrared spectroscopy

Academic Editor: Christian Brischke

Received: 13 May 2026

Revised: 10 June 2026

Accepted: 16 June 2026

Published: 23 June 2026

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1. Introduction

Solid wood is used for various purposes, such as construction, furniture and musical instruments. One of the most notable characteristics of wood is moisture absorption–desorption, along with changes in humidity, because cellulose, a dominant chemical component of wood, is highly hydrophilic. This characteristic acts as humidity conditioning for the interior or preserving the container; however, dimensional changes due to

absorption–desorption are a critical issue for construction and musical instruments. Furthermore, higher moisture absorption is associated with lower decay resistance. Therefore, in certain cases, wood must be treated to improve its resistance to humidity.

Thermal treatment is one of the easiest methods to improve the dimensional stability of wood. By increasing the crystalline region of cellulose, acetylation of the hydroxy groups and the network structure in lignin via thermal treatment result in a lower EMC and enhance dimensional stability [1–3]. In addition, the formation of a network between lignin and furfural, the production of acetic acid from pyrolyzed hemicellulose, and the esterification of cellulose during thermal treatment enhance decay resistance [4,5]. Thermal denaturation of lignin and phenolic extracts darkens the wood color; higher treatment temperatures and longer treatment durations decrease brightness. These color changes are related to chemical reactions in the wood components, which affect its mechanical properties and dimensional stability [6,7].

Thermal treatment also affects acoustic vibration properties. A treatment of 210 °C under a nitrogen atmosphere increased the crystallinity of cellulose and decreased sound resistance [8]. Vibrational characteristics, such as the fundamental resonant frequency (f_1) of flexural vibration and its loss tangent ($\tan\delta$), are also influenced by thermal treatment [9]. The frequency characteristics affect the sound characteristics of musical instruments. Furthermore, f_1 of flexural vibration is directly related to the dynamic Young's modulus (E_d); therefore, quantitative evaluation of the relationship between treatment conditions and changes in these characteristics is important for musical instruments or interior/exterior purposes.

Denaturation during thermal treatment is influenced by treatment conditions, such as time, temperature, the rate of temperature increase, moisture content, and atmosphere. As mentioned above, thermal treatment improves the water resistance of wood; however, the characteristics of wood change after long-term changes in humidity. According to a previous study, the EMC of thermally treated *Picea abies* increased after 90 cycles in a humid absorption–desorption test [10]. In addition, the moisture content during the absorption process increased during the second cycle of the cyclic humid absorption–desorption test [11]. Karami et al. conducted periodic moisture absorption tests after heat treatment under various humidity conditions [12]. The results showed reversible changes in the specific Young's modulus and $\tan\delta$, but irreversible changes due to heat treatment were also observed. These irreversible changes are thought to result from the annealing of the amorphous polymer during heat treatment.

Generally, cyclic absorption tests are designed to be sufficiently long for the specimens to reach their equilibrium moisture content. However, when using heat-treated wood for musical instruments, furniture, and interior purposes, not only long-term moisture absorption and desorption due to seasonal variations but also short-term temperature and humidity changes within a day, as well as atmospheric changes during transport, become important. This short-term moisture absorption and desorption do not cause the wood to reach its EMC. However, moisture absorption and desorption on the wood surface and moisture diffusion from the surface to the interior of the wood occur simultaneously. That is, even if the apparent moisture content is the same, different moisture gradients can be created, resulting in different physical properties.

This study aimed to clarify the changes in the vibrational characteristics of thermally treated wood during short-term cyclic moisture absorption. Nine different dry thermal treatment conditions (three temperature levels for three durations) were applied to Japanese cedar. Changes in f_1 and its $\tan\delta$ for flexural vibration were traced during three cycles of cyclic moisture change tests. Near-infrared diffuse reflectance spectra were also acquired to discuss the relationship between the chemical changes and vibrational characteristics.

2. Materials and Methods

2.1. Sample Preparation

Seventy Japanese cedar (*Cryptomeria japonica*) specimens, 150 mm in length, 20 mm in radius, and 10 mm in tangent, were prepared. Specimens were dried at 105 °C for 24 h to measure the initial dry weight and then placed at 20 °C and 65%RH for over three weeks to measure the initial airdried density. The average airdried density of 70 specimens was 0.383 g/cm³. The statistical characteristics of the specimens before thermal treatment are summarized in Table 1.

Table 1. Statistical characteristics of the specimens before thermal treatment.

Airdry Density g/cm ³	Moisture Content %	f_1 Hz	$\tan\delta$
0.383 (0.015)	8.86 (0.67)	2207 (76)	$5.71 (0.63) \times 10^{-3}$

() indicate standard deviation.

2.2. Flexural Vibration Test

The f_1 and its loss $\tan\delta$ for the flexural free vibration of each specimen were measured. The specimens were supported by polyurethane foam (33.6 mm) at both ends. Flexural vibrations were excited by tapping the center of the specimen with a small hammer. The excited vibration signals were captured using a condenser microphone (M4261, NTi Audio Co.) placed at the end of the specimen (Figure 1). The signal captured by a microphone was recorded by a field recorder (HS- P82, TEAC Co.) with 44.1 kHz and 24-bit WAV format. The obtained flexural signal was converted into a power spectrum using fast Fourier transform to determine f_1 . In addition, a Hilbert transform was performed to obtain the envelope signal on f_1 ; $\tan\delta$ was calculated from the envelope signal as follows.

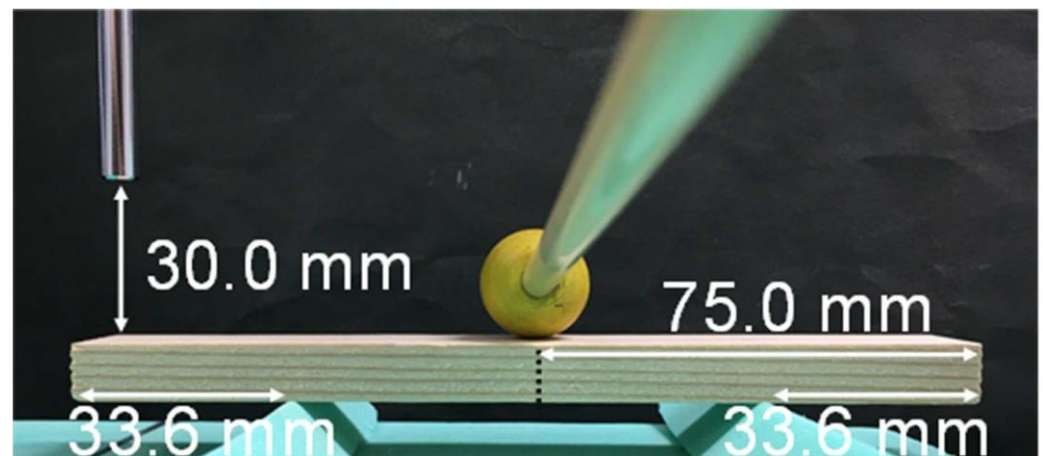


Figure 1. A schematic diagram of a flexural vibration test.

$$\tan\delta = \frac{\Delta \log A}{f\pi\Delta t} \quad (1)$$

where A and t are the resonance amplitude and time, respectively, of a logarithmic envelope signal. The dynamic flexural modulus (E_d) was determined from f_1 as follows:

$$E_d = \frac{48\pi^2 \rho l^4 f_1^2}{500.6h^2} \quad (2)$$

where ρ , l and h are density, length and thickness of the specimen, respectively.

The measurements were repeated three times and averaged. MATLAB (R2018a; MathWorks Inc., Natick, MA, USA) was used for this procedure. Flexural vibration tests were performed before and after the thermal treatment and during the cyclic moisture change test; f_1 , $\tan\delta$, and E_d were evaluated by a rate of change as follows:

$$\text{Rate of change in } x = \frac{X_a - X_b}{X_b} \times 100 \quad (3)$$

where X_b and X_a are vibrational characteristics such as f_1 and $\tan\delta$ before and after thermal treatment, respectively.

2.3. Dry Thermal Treatment

Nine different dry thermal treatments comprising three temperatures (160, 190 and 220 °C) and three treatment durations (short, medium and long) were applied in this study. Table 2 shows the combination of treatment temperature and duration. Dry thermal treatments were performed in an oven (SOFW- 450, ASONE Co.) under an air atmosphere. The specimens were placed in an oven prior to heating. Note that the treatment duration in this study is defined as the duration after the chamber reaches 100 °C. After thermal treatment, all specimens were immediately weighed then incubated at 20 °C and 65%RH for at least 3 weeks. Seven replications were used for each treatment condition, and seven untreated specimens were kept at 20 °C and 65%RH for control. Mass loss and EMC were calculated as shown below.

Table 2. Dry thermal treatment conditions.

Temperature (°C)	Treatment Duration (min.)		
	s*	m*	l*
160	800	1200	1600
190	200	300	400
220	50	75	100

*s: short, m: medium, l: long.

$$\text{Mass loss} = \frac{DW_b - DW_a}{DW_b} \times 100 \quad (4)$$

$$\text{EMC} = \frac{W - DW_a}{DW_a} \times 100 \quad (5)$$

where DW_b , DW_a , and W are the dry weight before thermal treatment, dry weight after thermal treatment, and weight after conditioning at 20 °C and 65%RH, respectively.

2.4. Near-Infrared (NIR) Spectroscopy

NIR diffuse reflectance spectra ranging from 1000 to 2600 nm at 1 nm intervals were measured using a spectrophotometer (UV-3600 Plus, Shimadzu Co.). The radial–longitudinal surface of each specimen was measured three times and averaged. The acquired NIR spectra were transformed into second-derivative spectra using the gap segment method (21-point segment with 0-point gap). MATLAB (R2018a, Mathworks Inc.) was used for the spectral transformation.

2.5. Cyclic Moisture Change Test

Cyclic moisture change tests were performed on the treated and control specimens. Initially, the constant-temperature humidity chamber (PH-2KT, Espec Co.) was set to 40 °C and 60%RH. The RH was changed to 73, 86, 98, 86, 73 and 60% at 48 h intervals. This

procedure was repeated three times. An illustration of the target RH during the cyclic moisture change test is shown in Figure 2. The weight measurements and flexural vibration tests were performed every 48 h. This test was designed to simulate short-term variations in moisture change; therefore, the specimens did not reach equilibrium moisture content at every stage.

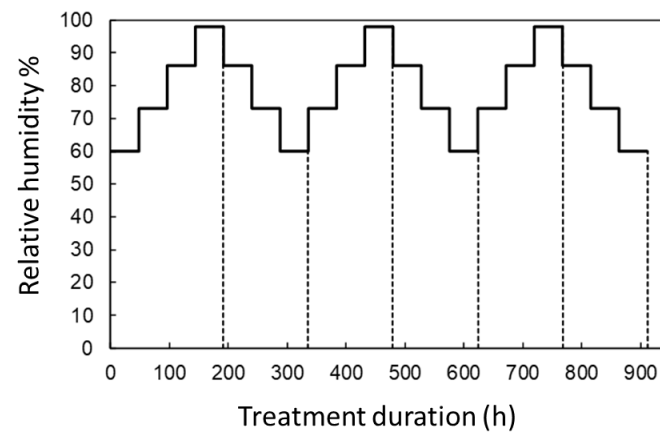


Figure 2. Design of relative humidity transition during cyclic moisture change tests.

3. Results and Discussion

3.1. Effect of Dry-Thermal Treatment on the Physical Properties

Figure 3 shows the mass loss and EMC under various thermal treatment conditions. As shown in Figure 3a, a greater mass loss occurred with longer treatment durations and higher temperatures. The maximum mass loss was approximately 5% under the harshest conditions (200 °C, 100 min). Generally, mass loss increased linearly with increasing treatment duration at the same temperature; however, there was almost no increase between 50 and 75 min under 220 °C.

The EMC decreased under all thermal treatment conditions (see Figure 3b). A higher treatment temperature resulted in a lower EMC. A longer treatment duration tended to lower the EMC at the same temperature, but this tendency was not pronounced compared with the mass loss. As with mass loss, EMC did not change between 50 and 75 min under 220 °C. As mentioned above, treatment duration is defined as the time after the chamber reached 100 °C; therefore, the strict duration when the specimens were heated at the treatment temperature was shorter. Almost the same mass loss and EMC at shorter and medium duration under the 220 °C treatment indicate that the 50 and 75 min treatment durations were too short to heat the specimen to the exact treatment temperature.

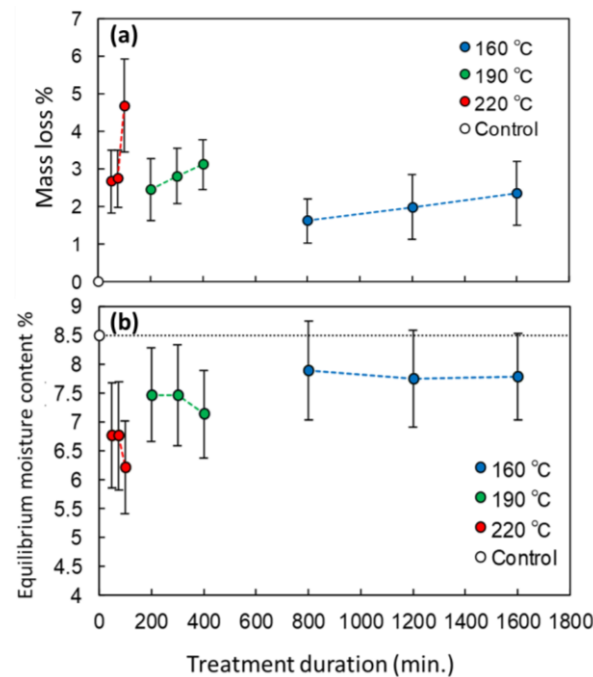


Figure 3. Relationship between thermal treatment duration (a) and mass loss (b). Equilibrium moisture content. Error bars indicate the standard deviation.

The relationship between the treatment conditions and flexural vibrational characteristics is shown in Figure 4. Under all conditions, f_1 increased after thermal treatment. The rate of change of f_1 increased with increasing treatment temperature; f_1 is inversely proportional to the square root of the density. Both higher mass loss and lower EMC due to the higher treatment temperature resulted in a lower airdried density. This is one of the main reasons why higher temperatures led to a higher rate of change in f_1 . Under the same temperature conditions (190 and 220 °C), f_1 decreased from the short to medium duration. Between 50 and 75 min under the 220 °C treatment condition, neither mass loss nor EMC changed. In addition, EMC did not decrease for the short or medium durations under the 190 °C treatment condition; f_1 is inversely proportional to the square root of flexural modulus. Therefore, the decrease in f_1 may be due to a decrease in the flexural modulus caused by the relatively higher treatment temperature.

Figure 4b shows the relationship between treatment conditions and $\tan\delta$. $\tan\delta$ slightly increased after 220 °C of thermal treatment; however, there was no clear relationship between treatment duration and $\tan\delta$ at each treatment temperature. There was a large variation in $\tan\delta$ relative to the mean rate of change, making it difficult to evaluate the effect of thermal treatment conditions on $\tan\delta$ in this study.

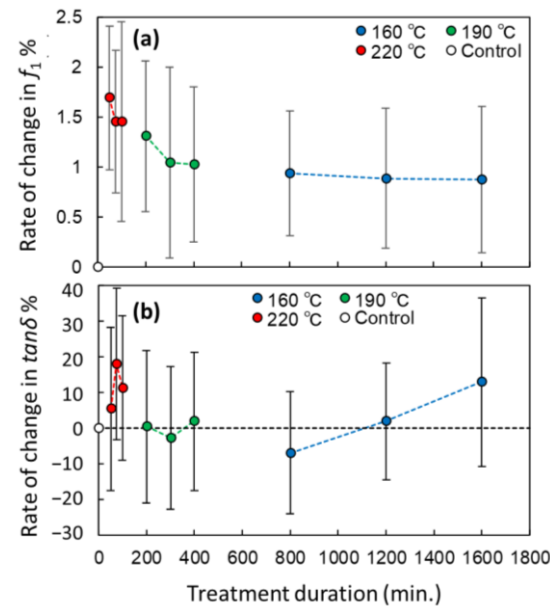


Figure 4. Relationship between thermal treatment duration and vibrational characteristics: (a) fundamental resonant frequency; (b) $\tan\delta$. Error bars indicate the standard deviation.

Figure 5a shows the rate of change in E_d . E_d decreased after thermal treatment under all conditions. The decrease in the E_d was greater with longer treatment durations. The decrease in E_d during the treatment period was greater at higher temperatures. As described in Equation (2), E_d is proportional to both the density and the square of f_1 . Specifically, E_d ($E_d/\text{density}$) increased under all treatment conditions, as shown in Figure 5b. Therefore, the decrease in E_d was primarily due to mass loss.

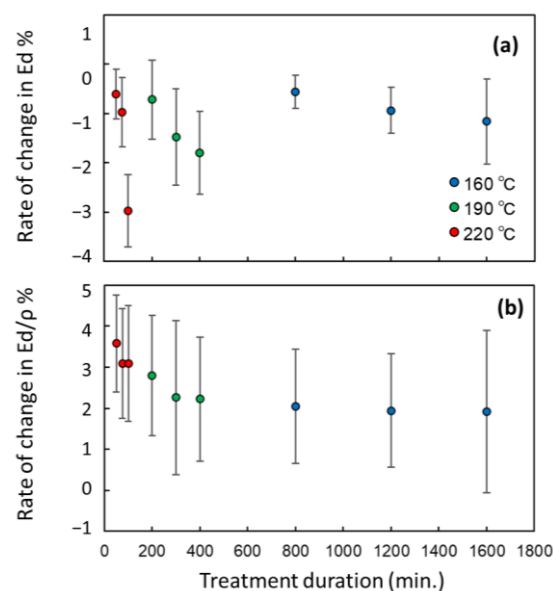


Figure 5. Relationship between thermal treatment duration and (a) E_d and (b) specific E_d . Error bars indicate the standard deviation.

3.2. NIR Spectra

The influence of the thermal treatment conditions on the chemical components of wood is discussed based on the notable absorption peaks in the NIR spectra assigned to the main wood components [13]. Note that the absorption peaks were extracted from the

second-derivative spectra; therefore, a lower second-derivative value indicates strong absorption.

The peak at 1153 nm was assigned to the second overtone of the C-H stretching vibration in the acetyl groups of hemicellulose [13]. As shown in Figure 6a, the absorption at 1153 nm was reduced across all treatment conditions compared to that of the control. Furthermore, the absorption mostly decreased with a longer duration and higher temperature. The decrease in absorbance with increasing treatment time was gradual at 160 °C but became more pronounced at higher temperatures. This indicated that hemicellulose deacetylation occurred at all treatment temperatures. In addition, partial pyrolysis occurred at 190 and 220 °C [14].

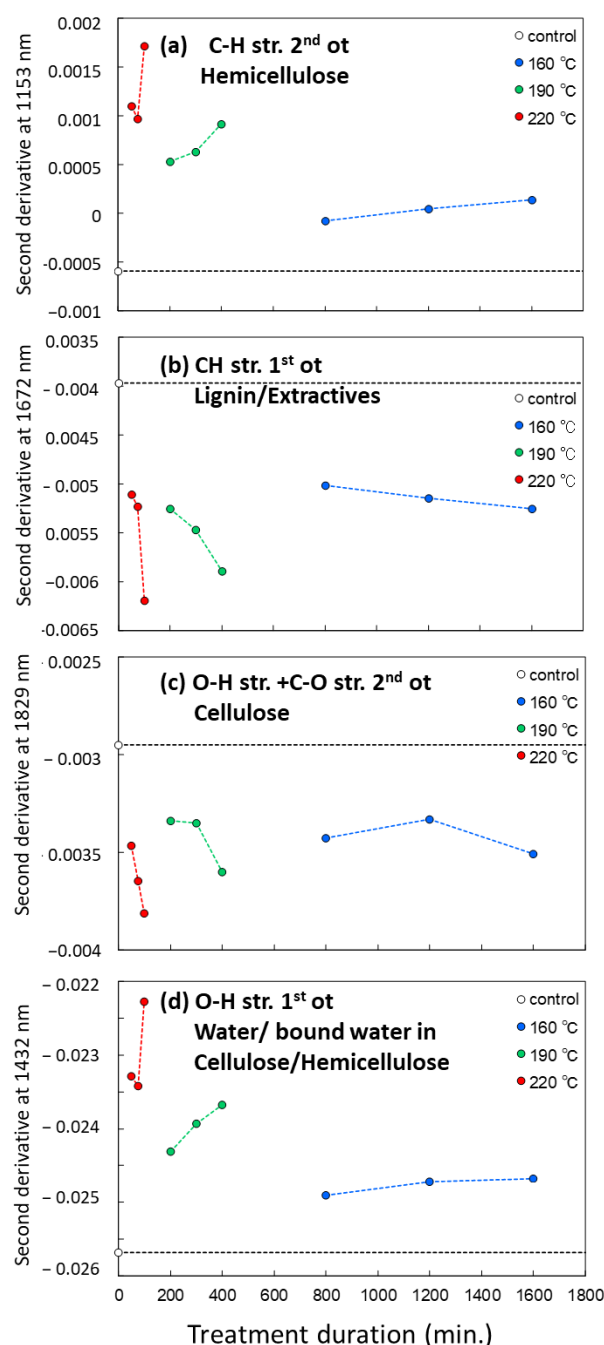


Figure 6. Relationship between thermal treatment duration and NIR absorbance at (a) 1153 nm, (b) 1672 nm, (c) 1829 nm and (d) 1432 nm.

Absorption at 1672 nm was assigned as the first overtone of the C-H stretching vibration due to the aromatic skeletons of lignin and wood extractives [13]. The absorption at this wavelength increased under all treatment conditions (Figure 6b). This increment is due to the relative increase in these components induced by the decrease in other components, such as hemicellulose and water. At 220 °C, absorbance was almost the same at 50 and 75 min; however, it was drastically increased at 100 min. Pyrolysis of lignin occurs gradually at lower temperatures, i.e., around 200 °C [15]. Also, 190–212 °C treatment induces the condensation polymerization of lignin [16]. This tendency also proves that the 50 and 75 min treatment durations in this study were too short to heat the specimen at the exact treatment temperature.

Figure 6c shows the second-derivative absorbance at 1829 nm, which was due to the combination of OH stretching and the second overtone of the CO stretching vibration assigned to the crystalline region of cellulose. The absorbance at 1829 nm increased after each thermal treatment. At 190 and 220 °C, stronger absorption was observed with increasing treatment duration. This indicated that the amorphous region of cellulose was thermally degraded by heat treatment at temperatures above 190 °C, resulting in a higher proportion of crystalline cellulose [17].

The absorption peak at 1432 nm, assigned to the first overtone of the OH stretching vibration due to water, decreased under all thermal treatment conditions (Figure 6d). Generally, the absorption decreased as both the duration and temperature increased. The absorption at 1432 nm is not only due to water but also due to the hydroxyl groups that form weak hydrogen bonds in the amorphous region of cellulose, glucomannan, and xylan [13]. The decrease in this absorption was mainly due to the loss of bonded water during thermal treatment. Furthermore, the decreased absorption at higher treatment temperatures indicates that thermal degradation of hemicellulose and dehydration of cellulose [18] also occurred under these conditions. Under anoxic conditions, cellulose microfibrils are reoriented in the middle layer of the secondary wall [19] and the microfibril angle changes [20]. Even our experiment cannot directly refer to that result since our thermal treatment was conducted under oxic conditions. However, it is highly likely that reorientation of cellulose microfibril occurred under conditions of 220 °C.

3.3. Moisture Content During Cyclic Moisture Change Test

Figure 7 shows the moisture content transition of the specimens during the three cycles of the moisture change tests. Note that the data at 73%RH in the first cycle are missing owing to errors in the experimental operation. Under all conditions, the moisture content during desorption was higher than that during the absorption process under the same relative humidity for the same number of cycles. This is due to sorption hysteresis [21]. The moisture content of the control specimens was consistently the highest throughout the test. There is a clear tendency for higher treatment temperatures and longer treatment durations to decrease moisture content. The moisture contents at 60 and 98%RH were extracted, as shown in Figure 8, and the effects of cyclic moisture changes were investigated. As shown in Figure 8a, the moisture content increased by 2%–3% after the first cycle of moisture change. As mentioned earlier, this phenomenon is known as sorption hysteresis. The increase in moisture content after the first cycle due to sorption hysteresis was greater at higher treatment temperatures and treatment durations. In contrast, the moisture content after the first cycle was lower at higher temperatures and longer durations. Focusing on the effect of the cycle number, the moisture content slightly increased with increasing cycles.

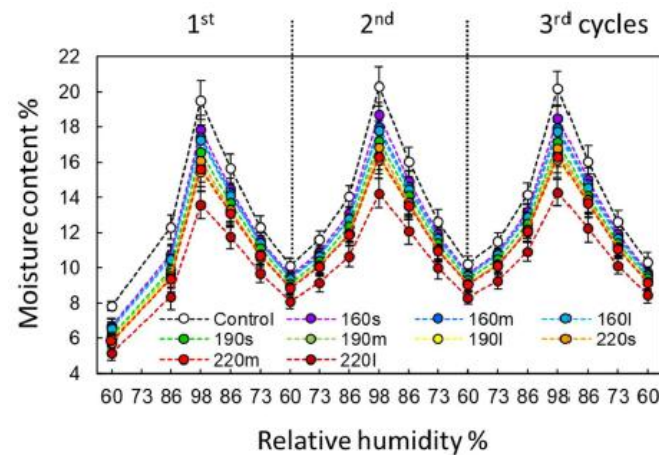


Figure 7. Changes in moisture content during the cyclic moisture change test. Error bars indicate the standard deviation.

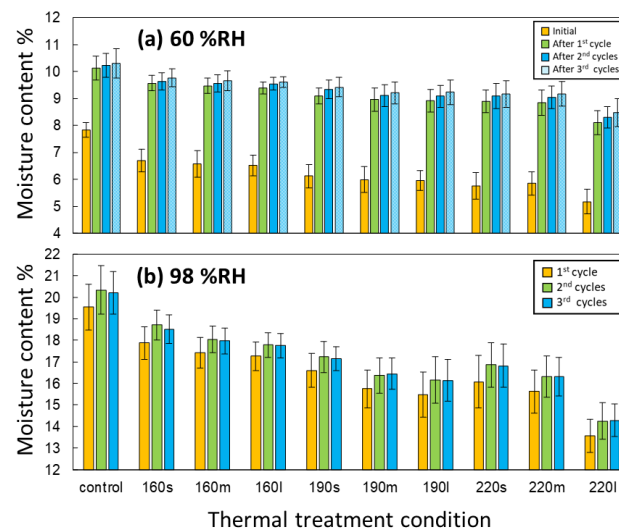


Figure 8. Changes in moisture content during cyclic moisture changes test at (a) 60%RH and (b) 98%RH. Error bars indicate the standard deviation.

Figure 8b summarizes the moisture content at 98%RH. In general, the moisture content decreased with increasing treatment temperature and duration. However, the moisture contents at 50 (s) and 75 (m) minutes at 220 °C were higher than that at 190 °C. As indicated by mass loss, EMC and NIR absorption, this phenomenon also proves that 50 and 75 min of treatment duration on 220 °C was too short to heat the specimen at the exact treatment temperature. When focusing on the changes in the cycle, there was no increase in moisture content from the second to the third cycle for any condition. This phenomenon differed from that observed for the moisture content at 60%RH. Moisture absorption occurs on the wood surface, which then diffuses. Therefore, at 98%RH, the surface of the specimen became saturated during the second cycle. However, as mentioned previously, the moisture content at 60%RH increased with the number of cycles. This suggests that diffusion from the surface to the interior occurs during desorption.

3.4. Vibrational Characteristics During Cyclic Moisture Change Test

Figure 9 shows the transition of f_1 and its $\tan\delta$ during cyclic moisture change tests. All data were expressed as rates of change relative to the values obtained before thermal treatment. As shown in Figure 9, data for the first absorption at 73%RH were missing. As shown in Figure 9a, f_1 decreases owing to moisture absorption. At the same relative

humidity under the same cycle, f_1 was higher during absorption than during desorption. This is because the moisture content during absorption was lower than during desorption, owing to sorption hysteresis. Higher treatment temperatures tended to result in lower f_1 . Further discussion on the effect of treatment conditions on f_1 will be presented later.

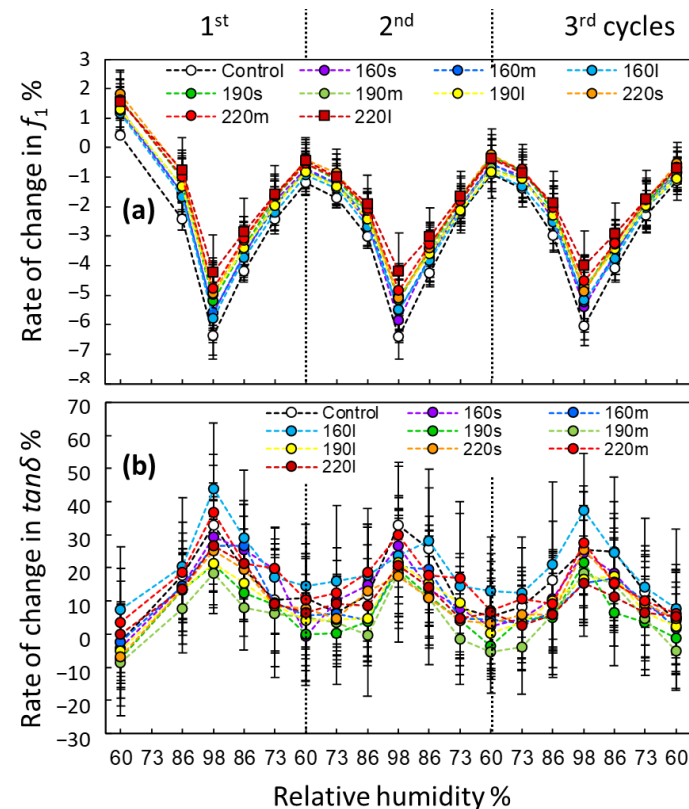


Figure 9. Changes in vibrational characteristics during cyclic moisture changes test: (a) rate of change in fundamental resonant frequency and (b) rate of change in $\tan\delta$. Error bars indicate the standard deviation.

Figure 9b shows the transition of $\tan\delta$. For all conditions, $\tan\delta$ decreased with increasing relative humidity. Increasing the wood moisture content means increasing the bound water in the cell wall, which results in a lower elastic modulus and increased internal friction. Unlike moisture content and f_1 , no clear trend was observed between treatment conditions and $\tan\delta$, and the difference between the control and treated groups was unclear. Essentially, f_1 did not change with the tapping method or strength. However, vibration damping is highly influenced by tapping conditions, as wood is a high-damping material. In this study, the specimens were manually tapped using a mallet. Therefore, $\tan\delta$ at each tapping was largely deviated, resulting in an unclear tendency across the treatment conditions. Hereafter, we focus on changes in f_1 .

The rates of change in f_1 at 60% and 98%RH during the cyclic moisture change tests are shown in Fig. 10. The rate of change of f_1 at the beginning of the tests was positive under all conditions. This is because f_1 increased with thermal treatment (see Figure 4a), and the specimens were slightly dried by changing the conditions from 20 °C and 65%RH to 40 °C 60%RH. As shown in Figure 10a, f_1 at 60%RH generally increased with increasing treatment temperature, but no clear trend was observed with respect to treatment time at the same temperature. At 65%RH, f_1 increased slightly from the first to the second cycle and then decreased slightly in the third cycle.

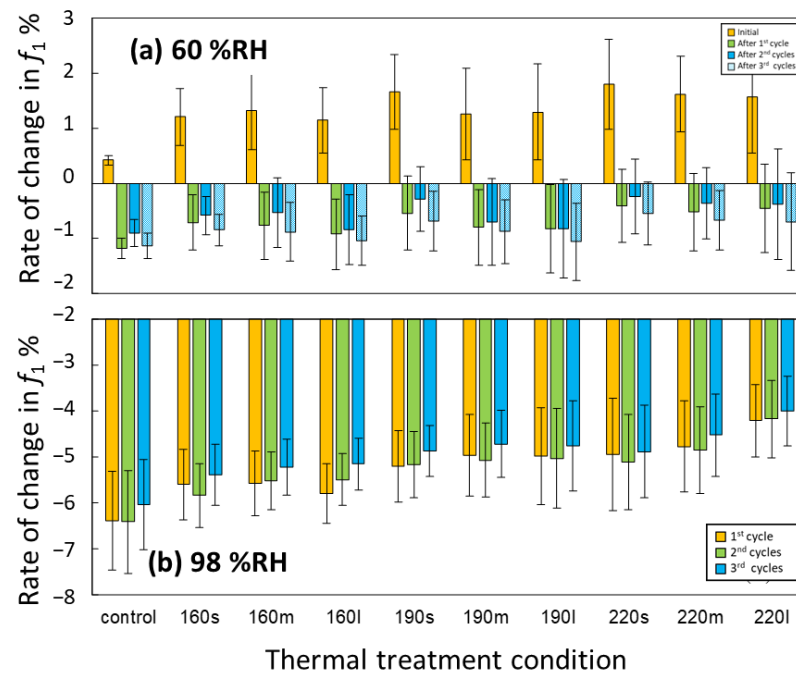


Figure 10. Changes in fundamental resonant frequency during cyclic moisture changes test at (a) 60%RH and (b) 98%RH. Error bars indicate the standard deviation.

As shown in Figure 10b, f_1 at 98%RH showed a similar tendency to that at 65%RH; a higher f_1 was observed at higher treatment temperatures. Furthermore, longer treatment duration resulted in a higher f_1 for the 220 °C treatment. Focusing on the changes in the number of cycles, f_1 did not change from the first to the second cycle, whereas it increased from the second to the third cycle under most conditions. Notably, this trend contrasts with the transition in the moisture content. As mentioned above, the moisture content at 98%RH increased from the first to the second cycle but did not change in the third cycle. Basically, an increasing moisture content decreases f_1 . In this study, the moisture content increased but f_1 did not change during the second cycle. In addition, the moisture content was constant, but f_1 increased during the third cycle. Nishino et al. reported that the moisture gradient in the thickness direction decreased the flexural resonant frequency of a free–free beam using the transfer matrix method [22]. Therefore, moisture diffusion from the surface to the core of the specimen increased with the number of cycles, mitigating the moisture gradient. As this phenomenon was observed under all treatment conditions, including the control, the thermal treatment in this study did not have a significant effect on moisture diffusion during cyclic moisture absorption.

From the f_1 transition during the cyclic moisture change tests, the thermal treatment conditions in this study did not significantly influence the moisture diffusion from the surface to the core of the specimen; however, a higher treatment temperature tended to maintain a higher f_1 during cyclic moisture changes. In particular, at the highest treatment temperature of 220 °C, longer treatment duration kept the higher f_1 of the specimen through the cyclic moisture change tests and decreased the difference between 60%RH and 98%RH. This is induced by mass loss during thermal treatment, and wood becomes more moisture-resistant during thermal treatment. Several absorption bands illustrated in Figure 6 show that the thermal treatment decreased the EMC and accelerated the degradation of hygroscopic pigments such as hemicellulose and the amorphous region of cellulose. The hemicellulose present between the cellulose microfibrils within the cell wall contributed to the viscosity and viscoelastic properties of wood. Thermal treatment reduces the free hydroxyl groups in hemicellulose, which not only causes hydrophobicity in the cell wall but also reduces shear slip between cellulose microfibrils, leading to

reorientation of the cellulose microfibrils [19] and increased crystallinity. These phenomena increase the stiffness of the cell wall, leading to higher f_1 values under hygroscopic conditions.

4. Conclusions

In this study, nine different dry thermal treatment conditions with three treatment temperatures and durations were applied to wood specimens. Higher treatment temperatures and longer treatment durations led to greater mass losses and lower EMC. The lowest EMC was obtained by the most severe thermal treatment conditions, 220 °C for 100 min. The fundamental resonant frequency of the free–free flexural vibration (f_1) increased with increasing treatment temperature; however, a longer treatment duration gradually decreased the resonant frequency. The E_d decreased under all conditions, mainly owing to mass loss, but the specific E_d increased as the temperature increased. The specimens were subjected to three cycles of moisture change tests to track the changes in the moisture content and vibrational characteristics. Similar to the EMC, the moisture content during the cyclic moisture change tests was lower, with higher thermal treatment temperatures and longer durations. The f_1 remained high at higher treatment temperatures. Under all conditions, the moisture content at 98%RH increased from the first to the second cycle; however, no change was observed from the second to the third cycle. However, f_1 did not change from the first to the second cycle, whereas it increased in the third cycle. Therefore, moisture diffusion from the surface to the core of the specimen was confirmed; however, there were no notable differences in the diffusion process under the thermal treatment conditions. The degradation of hemicellulose and the amorphous region of cellulose was suggested by the NIR spectra. Thus, chemical deformation by thermal treatment imparted hydrophobicity to the cell wall, resulting in a decrease in moisture content and changes in resonant frequency.

The moisture content and f_1 transition of the specimen at the 220 °C for 100 min condition were the most stable during the cyclic moisture change tests. Therefore, more severe treatments increase stability against moisture. This is a significant advantage when wood is used in furniture, flooring, and musical instruments.

Author Contributions: Conceptualization, H.K., Y.K. and K.A.; Methodology, H.K., R.H., Y.K. and K.A.; Experiment, R.H. and H.K.; Data Processing, R.H. and H.K.; Sample Preparation, H.K.; Writing—Original Draft Preparation, H.K. and R.H.; Writing—Review and Editing, H.K.; Supervision, H.K., Y.K. and K.A.; Project Administration, H.K. All authors have read and agreed to the published version of the manuscript.

Funding: This study received no external funding.

Data Availability Statement: The data supporting the findings of this study are available from the corresponding author, H.K., upon reasonable request.

Conflicts of Interest: The authors declare no conflicts of interest.

Abbreviation

EMC	equilibrium moisture content
f_1	fundamental resonant frequency
$\tan\delta$	loss tangent
NIR	near infrared

References

- Wikberg, H.; Maunu, S.L. Characterization of thermally modified hard- and softwoods by ^{13}C CPMAS NMR. *Carbohydr. Polym.* **2004**, *58*, 461–466.
- Tjeerdsma, B.F.; Militz, H. Chemical changes in hydrothermal treated wood: FTIR analysis of combined hydrothermal and dry heat-treated wood. *Holz Alz Roh-Und Werkst.* **2005**, *63*, 102–111.
- Boonstra, M.J.; Acker, J.V.; Tjeerdsma, B.F.; Kegel, E.V. Strength properties of thermally modified softwoods and its relation to polymeric structural wood constituents. *Ann. For. Sci.* **2007**, *64*, 679–690.
- Weiland, J.J.; Guyonnet, R. Study of chemical modifications and fungi degradation of thermally modified wood using DRIFT spectroscopy. *Holz Alz Roh-Und Werkst.* **2003**, *61*, 210–220.
- Boonstra, M.J.; Tjeerdsma, B.F. Chemical analysis of heat-treated softwoods. *Holz Alz Roh-Und Werkst.* **2006**, *64*, 204–211.
- Bekhta, P.; Nimez, P. Effect of high temperature on the change in color, dimensional stability and mechanical properties of spruce wood. *Holzforschung* **2006**, *37*, 539–546.
- Johansson, D.; Moren, T. The potential of color measurement for strength prediction of thermally treated wood. *Holz Alz Roh-Und Werkst* **2006**, *64*, 104–110.
- Zhu, L.; Liu, Y.; Liu, Z. Effect of high-temperature heat treatment on the acoustic-vibration performance of *Picea jezoensis*. *BioResources* **2016**, *11*, 4921–4934.
- Inoue, M.; Norimoto, M.; Tanahachi, M.; Rowell, R. Steam or heat fixation of compressed wood. *Wood Fiber Sci.* **1993**, *25*, 224–235.
- Majka, J.; Czajkowski, L.; Olek, W. Effects of cyclic changes in relative humidity on the sorption hysteresis of thermally modified spruce wood. *BioResources* **2016**, *11*, 5265–5275.
- Hill, C.; Ramsay, J.; Keating, B.; Laine, K.; Rautkari, L.; Hughes, M.; Constant, B. The water vapour sorption properties thermally modified and densified wood. *J. Mater. Sci.* **2012**, *47*, 3191–3197.
- Karami, E.; Bardet, S.; Matsuo, M.; Bremaud, I.; Gaff, M.; Gril, J. Effects of mild hygrothermal treatment on the physical and vibrational properties of spruce wood. *Compos. Struct.* **2020**, *253*, 112736.
- Schwanninger, M.; Rodrigues, J.C.; Fackler, K. A Review of Band Assignments in near Infrared Spectra of Wood and Wood Components. *J. Near Infrared Spectrosc.* **2011**, *19*, 287–308.
- Tjeerdsma, B.F.; Boonstra, M.; Pizzi, A.; Tekely, P.; Militz, H. Characterisation of thermally modified wood: Molecular reasons for wood performance improvement. *Holz Alz Roh-Und Werkst.* **1998**, *56*, 149–153.
- Brebu, M.; Vasile, C. Thermal degradation of lignin -A review. *Cellul. Chem. Technol.* **2010**, *44*, 353–363.
- Özgenç, Ö.; Durmaz, S.; Boyaci, I.H.; Eksi-Kocak, H. Determination of chemical changes in heat-treated wood using ATR-FTIR and FT Raman spectrometry. *Spectrochim. Acta Part A Mol. Biomol. Spectrosc.* **2017**, *171*, 395–400.
- Esteves, B.M.; Pereira, H.M. wood modification by heat treatment: A review. *BioResources* **2009**, *4*, 370–404.
- Scheirs, J.; Camino, G.; Tumiatti, W. Overview of water evolution during the thermal degradation of cellulose. *Eur. Polym. J.* **2001**, *37*, 933–942.
- Wang, D.; Fu, F.; Lin, L. Molecular-level characterization of changes in the mechanical properties of wood in response to thermal treatment. *Cellulose* **2022**, *29*, 3131–3142.
- Deng, L.; Chen, F.; Ren, X.; Jiang, Z. An environmentally friendly and efficient method to improve the acoustic vibration performance of bamboo for musical instruments: Nitrogen-protected heat treatment. *Ind. Crops Prod.* **2022**, *188*, 115554.
- Chen, C.M.; Wangaard, F.F. Wettability and the hysteresis effect in the sorption of water vapor by wood. *Wood Sci. Technol.* **1968**, *2*, 177–187.
- Nishino, Y.; Nakao, T.; Tanaka, C.; Takahashi, A. Free-free beam vibrations of wood with moisture gradients IV. Simulations of changes in natural frequencies during moisture absorption using a three-dimensional diffusion model. *J. Jpn. Wood Res. Soc.* **1993**, *39*, 382–388. (In Japanese)

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