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Dynamic viscoelastic properties of wood and acetylated wood in nonequilibrium states swollen by water or organic liquids

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Abstract

To discuss the factors that greatly affect the stabilization and destabilization of the conformation of lignin caused by changes in temperature and/or swelling, the temperature dependence of the loss tangent ($\tan\delta$) of the sample given the different cooling rates and the changes in the storage elastic modulus (E') and the loss elastic modulus (E'') over time during the swelling process were measured using the untreated or the acetylated wood samples swollen by water or organic liquids. The difference in the glass transition temperature of lignin mainly affects the degree of the destabilization of the conformation of lignin caused by quenching in the untreated and acetylated wood samples. The changes in E' and E'' during swelling process were significantly different between the liquids in the untreated wood sample. The differences of E' and E'' in the different liquids were considered to be caused by the balance of the stabilization depending on the swelling amount and passage of time and the destabilization due to the rapid swelling.

Keywords: Organic liquid, Swelling rate, Destabilization, Conformation of lignin, Quenching

Introduction

Wood subjected to short-term changes in temperature and/or swelling shows lower elastic modulus and greater fluidity compared with stable wood that has been exposed to a constant temperature and humidity for a long time [1–8]. The thermal-softening properties of wood are also changed by histories such as quenching or drying before measurement [9–14]. It has been reported that the glass transition temperature of lignin in the water-swollen wood with quenching and/or drying was lower than that in green wood [9, 10]. The glass transition temperature of lignin that was decreased by these histories was increased over time by water soaking [10]. These changes in the mechanical properties of wood

are interpreted to be caused by the localized stress in the amorphous region of wood cell walls. In addition, based on the studies of micropores, dynamic viscoelastic properties, and endothermic and exothermic behaviors, it is considered that these stresses are mainly introduced in lignin in the amorphous region of the wood constituents [15–18]. Therefore, changes in the mechanical properties with histories are closely related to the thermodynamic state of the conformation of lignin.

From these studies, wood after being subjected to short-term change in the environment is interpreted as in the unstable state that has localized stresses in amorphous region of wood cell walls, which were caused by the dimensional change in the microstructure according to changes in temperature and/or swelling. The instability generated in wood is gradually eliminated over time. On the other hand, the rate of the stabilization would depend on the temperature and the swelling amount. Therefore, the stabilization and destabilization occur simultaneously during the process of changes in temperature and swelling, and the degree of the instability of wood

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is considered to be changed depending on the balance between the stabilization and destabilization. Similar phenomena are generally known in the field of polymers and metals, and new materials are developed with this in mind [19, 20]. Also, in the field of wood, molecular conditions should be considered to further technological development.

The thermal-softening properties of wood change significantly depending on the type of swelling liquid. In the previous study, we measured the temperature dependence of the dynamic viscoelasticity of wood and acetylated wood swollen by various organic liquids [21]. The results show that the glass transition temperature of lignin is greatly changed by the swelling liquids and the chemical treatment of wood. The change in the glass transition temperature of lignin is thought to be caused by a change in the interaction between the molecules of the wood constituents and the swelling liquids. Therefore, the conformational state of lignin subjected to changes in temperature and/or swelling is expected to be different depending on the swelling liquid or the chemical structure of the wood constituents.

In this study, two types of measurements were performed to discuss the factors that greatly affect the stabilization and destabilization of the conformation of lignin in detail. Firstly, to examine the degree of the destabilization caused by quenching in the swollen sample, the dynamic viscoelastic properties of annealed and quenched samples were compared in the untreated and acetylated wood swelled by water or various organic liquids. Secondly, to examine the change in the

conformation of lignin over time during the swelling process, the changes in the dimension and dynamic viscoelastic properties of the untreated sample were measured.

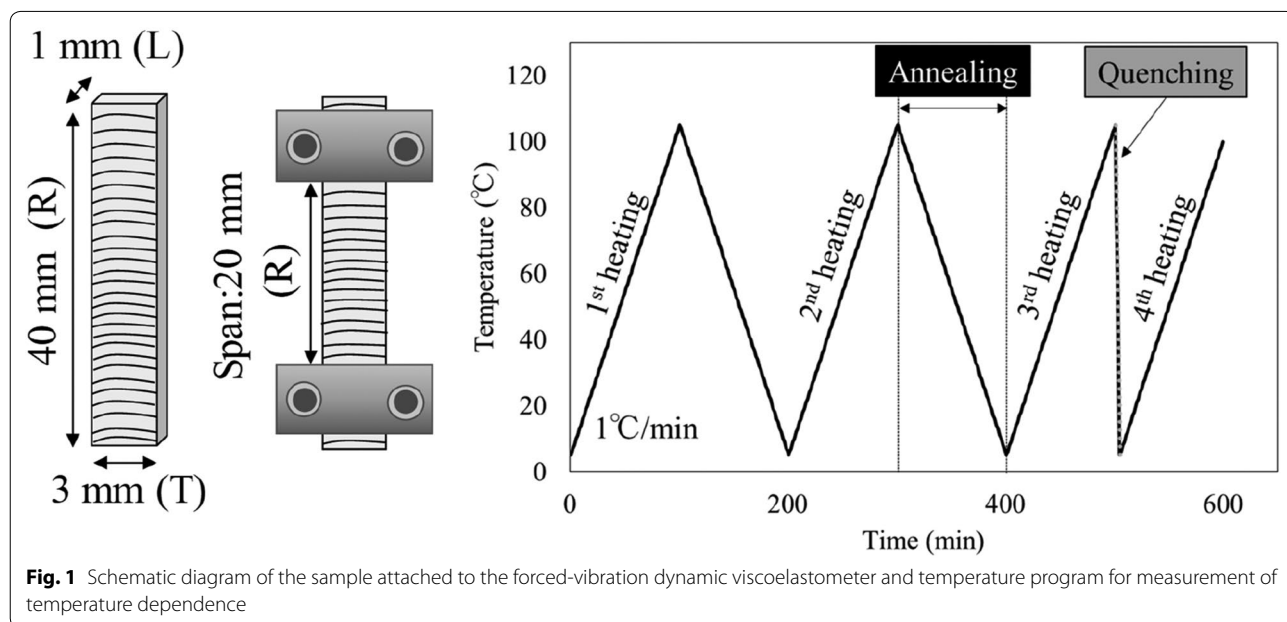
Materials and methods

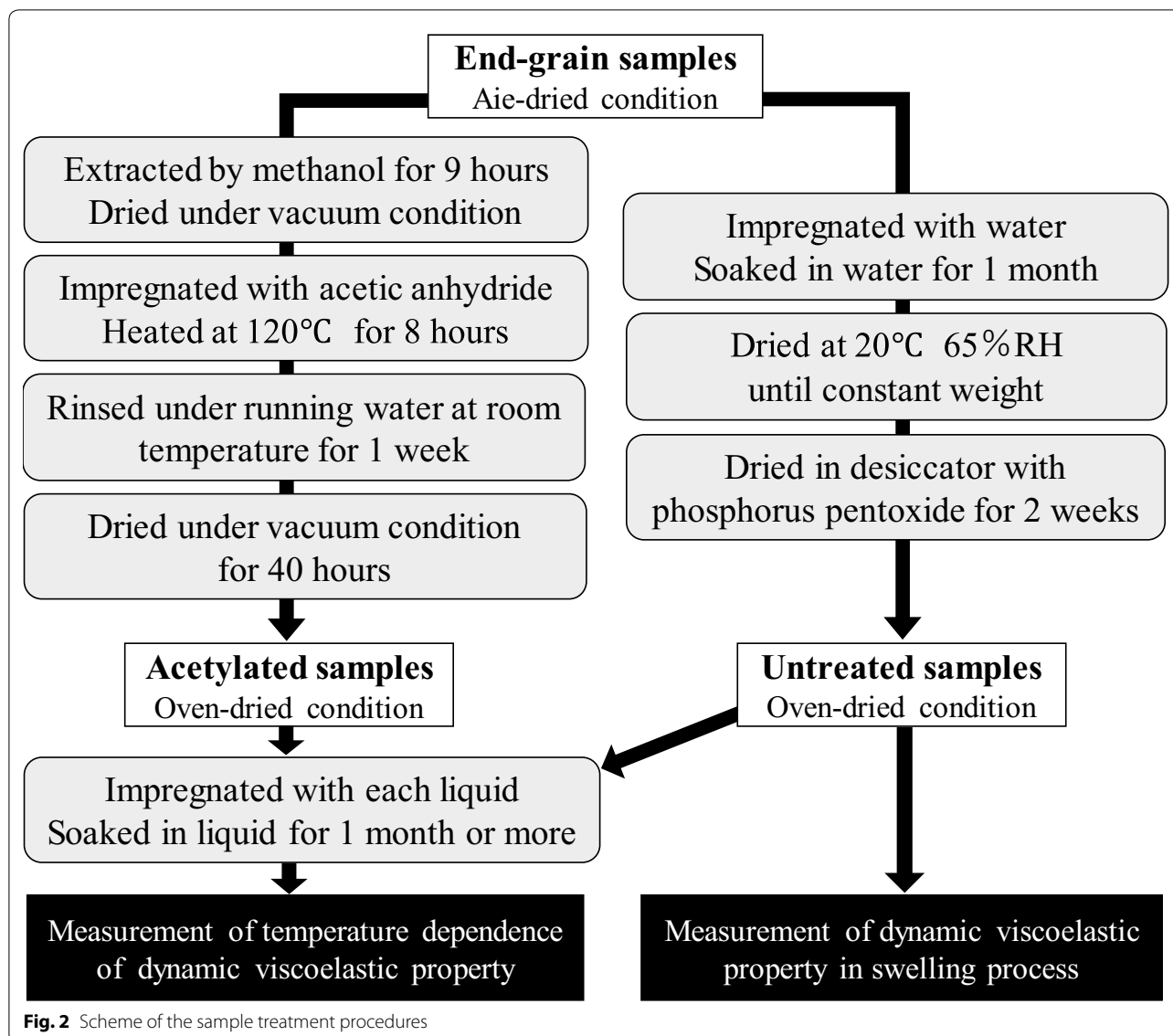
Samples

The samples used for the measurements were air-dried hinoki (*Chamaecyparis obtusa*). Blocks (40 and 3 mm in the radial and tangential direction, respectively) were cut from almost the same growth ring band in the outer area of the heartwood. From each block, end-grain samples with 1 mm thickness were continuously obtained in the longitudinal direction. The average of the annual ring width was 0.73 mm. The shape of the sample is shown in Fig. 1. The samples were extracted for 9 h using methanol, oven-dried under vacuum conditions and impregnated with acetic anhydride under reduced pressure, and then treated for 8 h in an oil bath at 120 °C. After treatment, the samples were rinsed for 1 week under running water at room temperature, and oven-dried under the vacuum condition for 40 h at 25 °C. Weight increase due to acetylation was 24%. The untreated samples were dried for 2 weeks in a desiccator with phosphorus pentoxide until they were at a constant weight. The schematic diagram of the sample treatment procedure is shown in Fig. 2.

Organic liquids

Distilled water and 10 organic liquids were used. The basic properties for ethanol (EtOH), 2-propanol (PrOH), 1-butanol (BuOH), acetone (Act), methyl ethyl ketone (MEK), ethylene glycol (EG), formamide (FA), dimethyl





sulfoxide (DMSO), methanol (MeOH), and toluene (Tol) are listed in Table 1.

Dynamic viscoelastic measurement

A forced-vibration dynamic viscoelastometer (DMS6100, Seiko Instruments, Chiba, Japan) was used for two types of measurements.

The temperature dependence of the dynamic viscoelastic property of each sample swollen by each liquid was measured. Before the measurement, the oven-dried samples were sealed in sample jars and soaked in liquid under vacuum conditions. The sealed samples were left for 1 month or longer. The swollen sample was attached to the DMS and the measurement was performed at a temperature range of -20 °C to 95 °C, for each liquid.

If the freezing or boiling point was within this temperature range, measurements were taken in a range of 10 °C above or below the freezing and boiling points of the liquid. Figure 1 shows the temperature schedule used for measurement. The results of the third and fourth heating process after cooling at 1 °C/min and after the rapid cooling, respectively, were compared to the results of annealed and quenched samples.

The dynamic viscoelasticity of untreated samples during the swelling process was measured over time. The oven-dried sample was attached to the DMS, and the attached sample was enclosed in a plastic bag with silica gel and phosphorus pentoxide. After the viscoelasticity fluctuations were reduced over approximately 2 h, the attached sample was soaked in each liquid at 30 °C. The

Table 1 Properties of the liquids and the relative swelling ratio of the samples swollen by those liquids

Liquids		Structural formula	Molar volumes [cm ³ /mol]	P.A.Δv ^a [cm ⁻¹]	H-bonding properties	C.E. ^d [cal/cm ³]	Relative swelling ^e [%]
Methanol	MeOH	CH ₃ OH	40.7	187	AD ^b	204	89.7
Ethanol	EtOH	C ₂ H ₅ OH	58.5	187	AD	166	82.5
2-Propanol	PrOH	(CH ₃) ₂ CHOH	75.0	187	AD	144	73.6
1-Butanol	BuOH	CH ₃ CH ₂ CH ₂ CH ₂ OH	91.8	187	AD	128	63.0
Acetone	Act	CH ₃ COCH ₃	74.0	64	A ^c	96	68.0
Methyl ethyl ketone	MEK	CH ₃ CH ₂ COCH ₃	90.2	57	A	86	56.0
Toluene	Tol	C ₆ H ₅ CH ₃	106.4	45	A	79	0.9
Ethylene glycol	EG	HOCH ₂ CH ₂ OH	55.8	206	AD	266	111.2
Formamide	FA	HCONH ₂	39.7		AD	317	116.8
Dimethyl sulfoxide	DMSO	(CH ₃) ₂ SO	70.9	141	A	166	133.0
Water	Water	H ₂ O	18.0	390	AD	552	100.0

^a Proton accepting powers: The data are cited from a table of Kagiya et al. [22], Crowley et al. [23], and Ishimaru et al. [24]. ^b Liquid having both proton accepting and donating properties for hydrogen bonding. ^c Liquid having only proton accepting properties for hydrogen bonding. ^d Cohesion energy: the data are calculated from the solubility parameter values in a table by Hansen [25]. ^e Relative swelling compared with that in water: The data are cited from a table of Sakai et al. [26] and Ishimaru et al. [27]

change in the dynamic viscoelastic properties was measured over 6 h during the swelling process.

Those measurements were performed using a sine wave and the measurement frequency was 0.05, 0.5, 1, 5, 10, and 50 Hz. The span was 20 mm in the radial direction, and the tension displacement amplitude was 5 μm. The results measured at 0.05 Hz are discussed in this study.

Dimensional change in the swelling process

A thermomechanical analyzer (TMA/SS6100, Seiko Instruments, Chiba, Japan) was used to measure the dimensional change of the untreated sample during the swelling process. The dried sample was attached to the TMA, and the attached sample was enclosed in a plastic bag with silica gel and phosphorus pentoxide. After the fluctuation of dimensional change was reduced over approximately 2 h, the attached sample was soaked in each liquid at 30 °C. The dimensional change of the sample was measured for 6 h. The span was 20 mm in the radial direction and the constant tension load was 5 g.

Results and discussion

The destabilization of the conformation of lignin caused by quenching

The temperature dependence of the loss tangent ($\tan\delta$) measured after the annealing and quenching processes is shown in Fig. 3. For almost all the quenched samples, the $\tan\delta$ was higher in the lower temperature range and the peak temperature of the $\tan\delta$ was slightly lower than that for each annealed sample. Similar results were observed in a study of the dynamic viscoelastic properties of the water-swollen hinoki in radial measurements

[1–5]. In this study, the $\tan\delta$ measured after quenching was higher than the $\tan\delta$ measured after annealing due to the decrease in the storage elastic modulus (E') and the increase in the loss elastic modulus (E'') in the temperature range below the peak temperature of the $\tan\delta$. These changes in the dynamic viscoelastic properties are thought to be caused by the destabilization of the conformation of lignin, which cannot be eliminated due to the inactive molecular motion at low temperatures. Similar phenomena are generally observed in the polymer material [19]. It was also observed that the $\tan\delta$ for the annealed and quenched samples swollen by each liquid crossed at a certain temperature. In the higher temperature range where the $\tan\delta$ of the quenched sample was lower than that of the annealed sample, the conformation of lignin in the quenched swollen samples may still be memorized in the active condition at higher temperatures.

On the other hand, the difference of the $\tan\delta$ between the annealed and quenched samples varied depending on the type of swelling liquid. To discuss the degree of the instability caused by quenching in all samples, $\tan\delta_Q/\tan\delta_A$ was defined by the following equation:

$$\tan\delta_Q/\tan\delta_A(\%) = 100 \times (\tan\delta \text{ measured after quenching}/\tan\delta \text{ measured after annealing}).$$

The above is a ratio of the $\tan\delta_Q$ measured after quenching to the $\tan\delta_A$ measured after annealing. The ratio is higher than 100% if the $\tan\delta$ measured after quenching is higher than the $\tan\delta$ measured after annealing, due to the destabilization of the conformation of lignin in the temperature range below the peak temperature of the

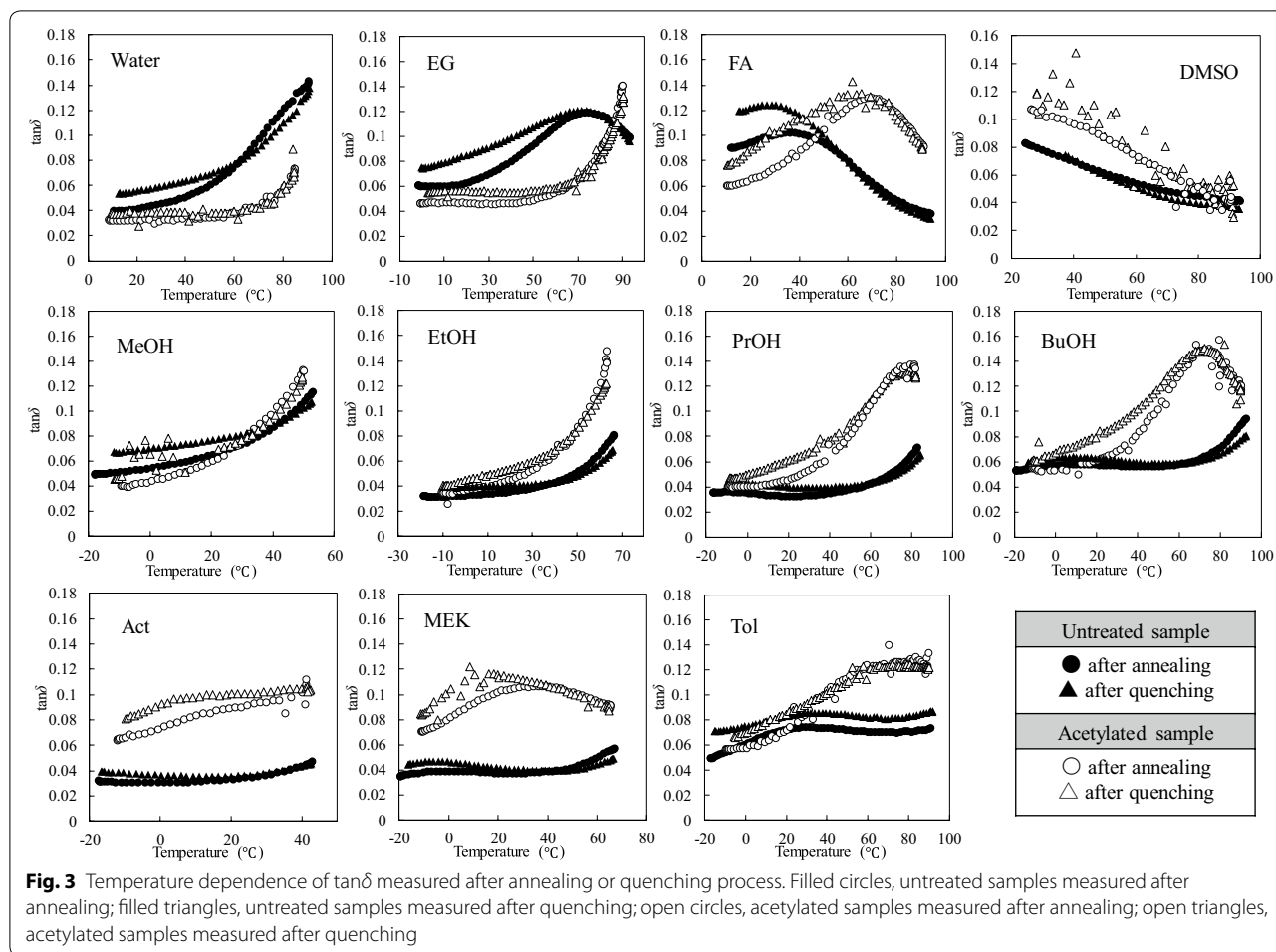
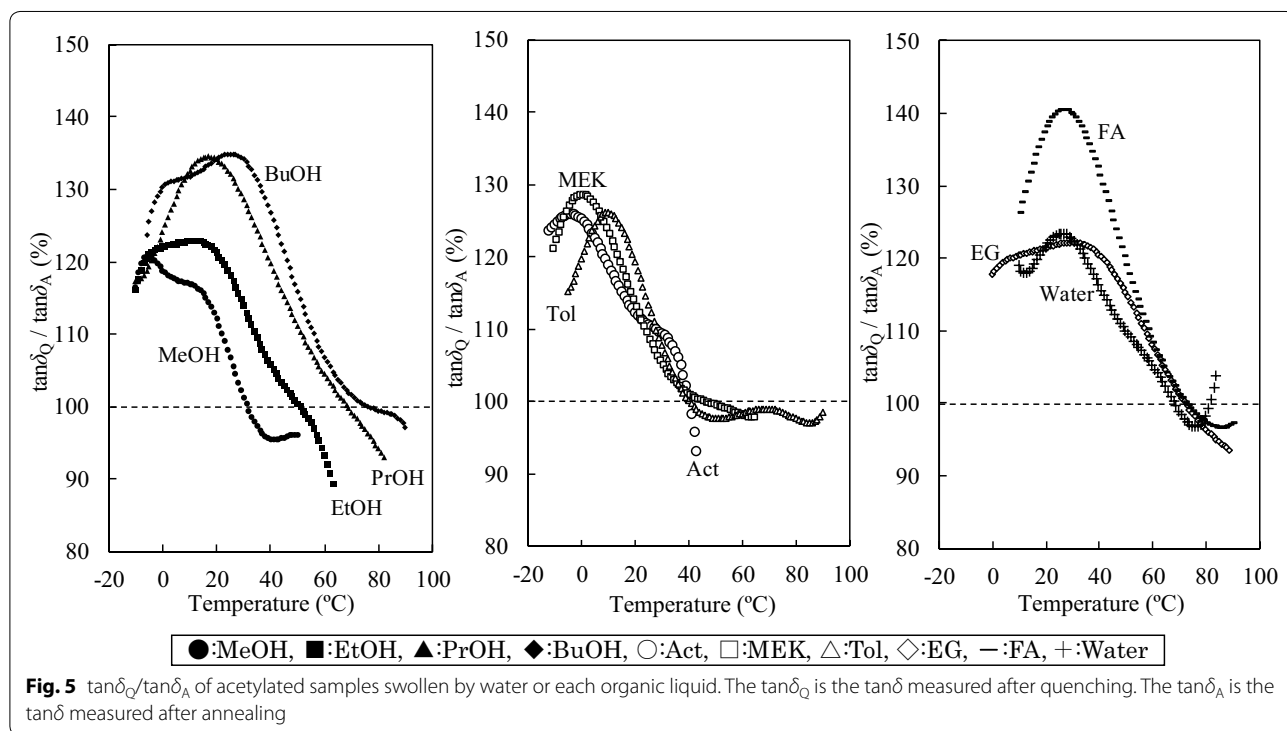
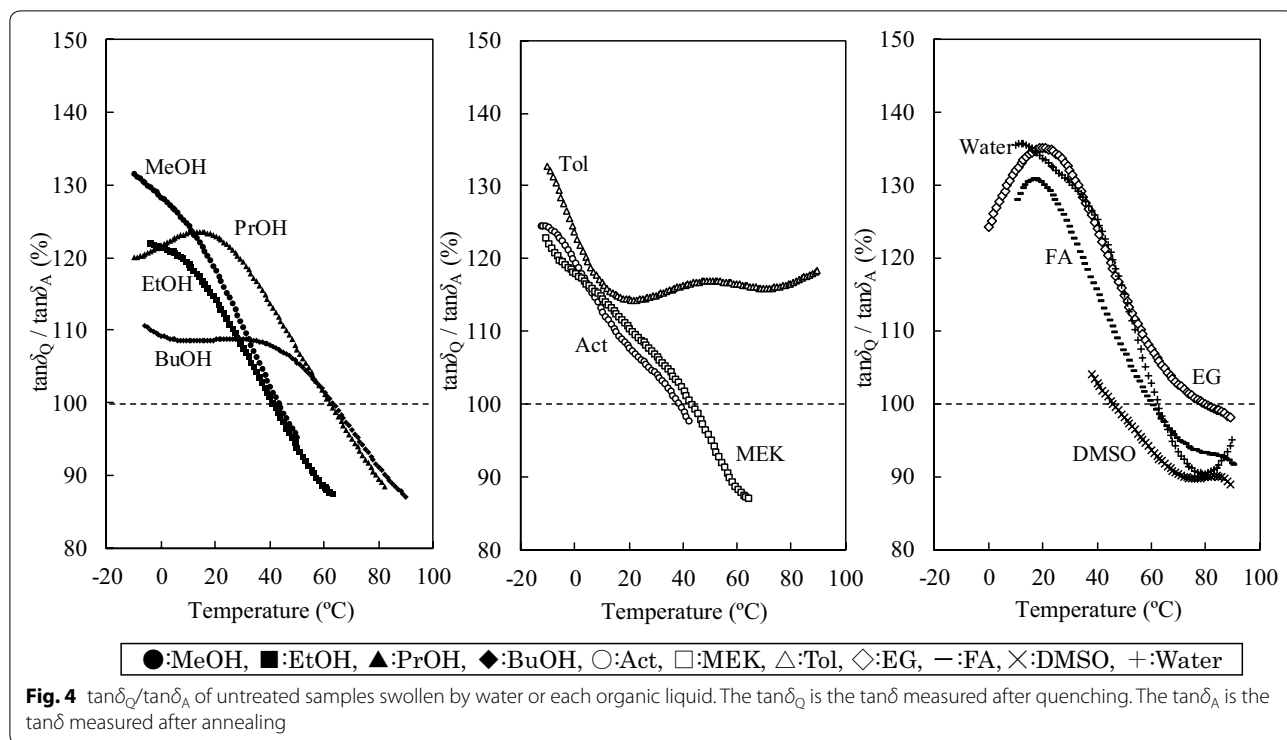


Fig. 3 Temperature dependence of $\tan\delta$ measured after annealing or quenching process. Filled circles, untreated samples measured after annealing; filled triangles, untreated samples measured after quenching; open circles, acetylated samples measured after annealing; open triangles, acetylated samples measured after quenching

$\tan\delta$. Figure 4 shows the $\tan\delta_Q/\tan\delta_A$ of untreated swollen samples. All samples, except for the sample swollen by Tol, showed that $\tan\delta_Q/\tan\delta_A$ was higher than 100% in the lower temperature range and lower than 100% in the higher temperature range. The maximum values of the $\tan\delta_Q/\tan\delta_A$ of the samples swollen by each alcohol were higher in the order of larger relative swelling of the wood, except in the sample swollen by PrOH. The samples swollen by water or EG showed the highest maximum value of $\tan\delta_Q/\tan\delta_A$. The samples swollen by water or EG were cooled from near the peak temperature of the $\tan\delta$. For these samples, the conformation of the lignin was disturbed at low temperatures because the activated molecular motion near the glass transition temperature of lignin was rapidly frozen by quenching. In addition, the unstable conformation of lignin could not be eliminated due to the inactive molecular motion at low temperatures. On the other hand, the $\tan\delta_Q/\tan\delta_A$ of the sample swollen by DMSO was less than 100% in almost all the measured temperature ranges. This sample probably has a peak temperature of $\tan\delta$ at lower temperatures below

the measurement program. Therefore, the destabilization of the conformation of lignin caused by quenching was quickly eliminated because the molecular motion of the sample swollen by DMSO was active even after being cooled to approximately 20 °C. In contrast, the sample swollen by Tol showed a high $\tan\delta_Q/\tan\delta_A$ in all temperature ranges. Tol barely swelled the sample. The state of the lignin might be unstable because the molecular motion is not active at higher temperatures.

Figure 5 shows the $\tan\delta_Q/\tan\delta_A$ of acetylated samples swollen by water or organic liquid. The $\tan\delta_Q/\tan\delta_A$ of the sample swollen by DMSO could not be calculated because the data of the $\tan\delta$ contained too much noise. Compared with the result obtained from untreated wood, the maximum value of the $\tan\delta_Q/\tan\delta_A$ of the samples swollen by alcohol, except MeOH, was greater, while the values in samples swollen by EG or water were less at lower temperatures. The maximum value of the $\tan\delta_Q/\tan\delta_A$ of the sample swollen by FA was the highest in all of the acetylated samples. These results indicate that the degree of instability caused by quenching is closely



related to the glass transition temperature of lignin. In Fig. 3, the peak temperature of the $\tan\delta$ was lower in the acetylated samples swollen by alcohols and higher

in the acetylated samples swollen by water or EG, when compared with the untreated samples. That is, a sample cooled from a temperature near the peak temperature of

the $\tan\delta$ to a lower temperature has a greater large value of $\tan\delta_Q/\tan\delta_A$. For the acetylated sample swollen by Tol, the $\tan\delta_Q/\tan\delta_A$ decreased at higher temperatures. It is known that Tol swelling of acetylated wood is greater than that of the untreated wood [28]. Therefore, as the molecular motion is activated by swelling, some of the disturbed conformation of lignin caused by quenching may be stabilized.

From the results obtained, it is considered that the destabilization of the conformation of lignin is mainly affected by the glass transition temperature of lignin, whereas the value of the $\tan\delta_Q/\tan\delta_A$ of the untreated sample swollen by MeOH or FA could not be explained only by the glass transition temperature. Liquid properties, such as proton accepting power and cohesive energy, should also be considered in further studies to explain in detail the behavior of $\tan\delta_Q/\tan\delta_A$.

The change in the conformation of lignin in the swelling process

Changes in dimension and the viscoelasticity of untreated samples after soaking in water or organic liquid were measured. In this study, viscoelasticity was measured at 0.05 Hz. There are reasons why the measurement was performed at 0.05 Hz. Much data on the viscoelastic properties of water-swollen wood with various histories have been reported at 0.05 Hz. In addition, we wanted to measure the response of the relatively large molecules, in which case it takes some time to eliminate the instability. In the swelling process, the time required for the stabilization and the destabilization of the conformation of lignin was expected to be different between the liquids. However, as this experiment was being performed for the first time, we were unable to predict an appropriate sampling interval for the measurement. Therefore, we discuss the results measured at 0.05 Hz in this section.

The dimensional change during swelling process is shown in Fig. 6. The swelling behavior was different between the liquids. The samples swollen by DMSO, water, EG, FA, or MeOH reached their swelling equilibrium at around 100 min, whereas other samples were still in the swelling process after 360 min. The samples swollen by DMSO, water, EG, or FA that reached their swelling equilibrium showed high swelling at 360 min. The higher the swelling rate of each alcohol, the smaller the molar volume. When dry wood constituents adsorb molecules of liquid, energy for scission of the hydrogen bonds between the wood constituents is needed. The large amount of energy for scission is required if the volume of the molecule of liquid is large [29]; therefore, the molar volume affects the swelling rate in a sample swollen in alcohol.

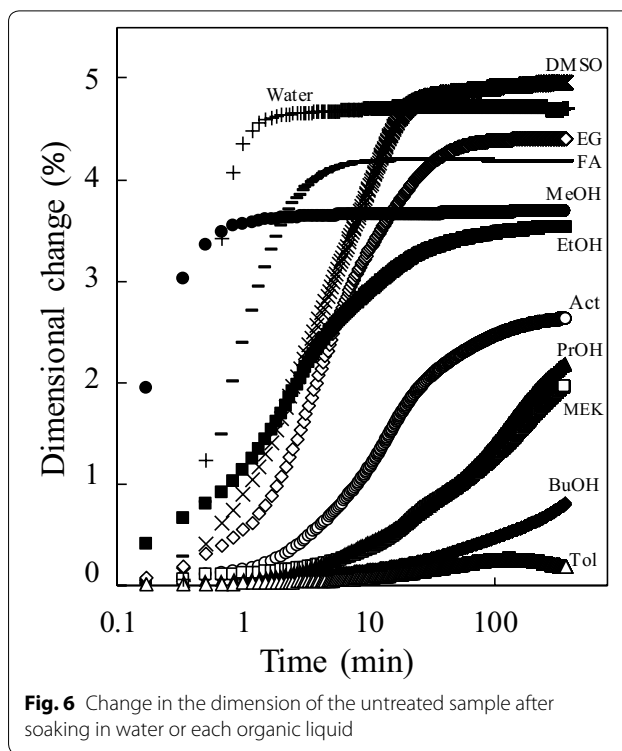
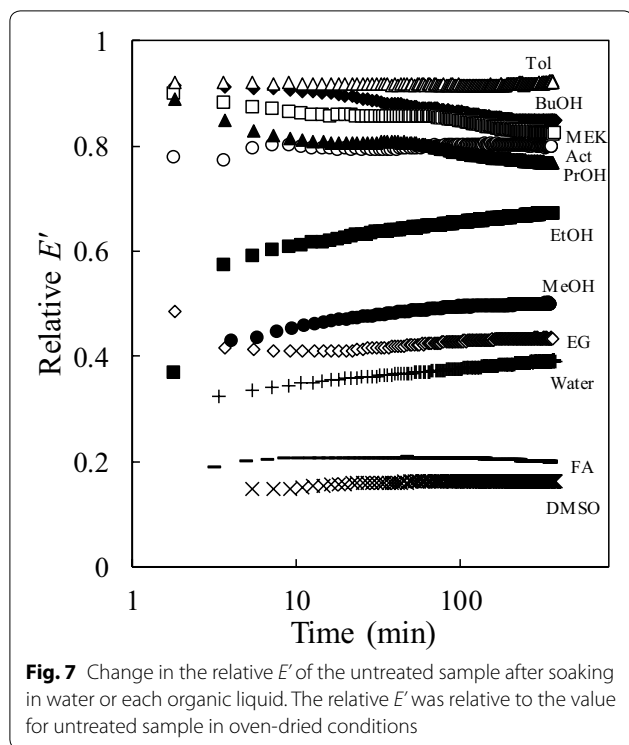


Fig. 6 Change in the dimension of the untreated sample after soaking in water or each organic liquid

The change in E' over time, during the swelling process, is shown in Fig. 7. Relative E' was relative to the value for untreated samples in oven-dried condition. Relative E' was largely decreased immediately after soaking in each liquid and gradually changed over time. The behavior of relative E' was different between the swollen liquids. Generally, the elastic modulus of wood swollen by water or organic liquid tends to decrease with an increase in the swelling amount, and we have also obtained the similar results in previous studies [30, 31]. It is also known that the elastic modulus of the water-swollen wood is decreased or increased by a destabilization or stabilization in the conformation of lignin, respectively [11]. Therefore, in this study, the decrease in relative E' was not solely caused by an increase in the swelling amount.

In Fig. 7, the decrease in relative E' immediately after soaking in each liquid was great in the samples swollen by DMSO, FA, water, or EG, which indicates a high swelling rate and swelling amount. The decrease in relative E' immediately after soaking in each liquid was small in the samples swollen by Tol, BuOH, MEK, Act, or PrOH, which indicates a low swelling rate and swelling amount. However, the degree of the reduction in relative E' did not necessarily correspond to the swelling amount in all the samples. The decrease in relative E' of the sample swollen by MeOH was much larger than that of the sample swollen by EtOH. If the degree of the reduction in the relative E' is mainly affected by the amount of swelling,



then the difference in relative E' between the samples swollen by EtOH and MeOH is small. However, the difference in relative E' between the samples was large. The sample swollen by MeOH showed extremely rapid swelling, as shown in Fig. 6. Therefore, it is considered that the reduction in relative E' immediately after soaking in each liquid is increased by the destabilization of the conformation of lignin caused by the rapid swelling.

For the change in relative E' over time, the samples that reached swelling equilibrium at 100 min after soaking in each liquid, such as the samples swollen by DMSO, water, EG, FA, or MeOH, showed a slight increase or a constant value. The destabilization caused by swelling did not occur after the samples reached their swelling equilibrium. Therefore, these samples were considered to be in the process of the stabilization. In particular, the samples swollen by DMSO, FA, or EG, which showed a constant value of relative E' , had high swelling and low glass transition temperature of lignin. The change in relative E' over time during the swelling process was measured at 30 °C. The stabilization of the conformation of lignin rapidly proceeded as the molecular motion was relatively activated at the measured temperature. That is, the destabilization in the conformation of lignin caused by swelling was almost eliminated in the samples swollen by DMSO, FA, or EG. However, relative E' of the samples swollen by BuOH, MEK, or PrOH showed small decrease immediately after soaking in each liquid and slight decrease over

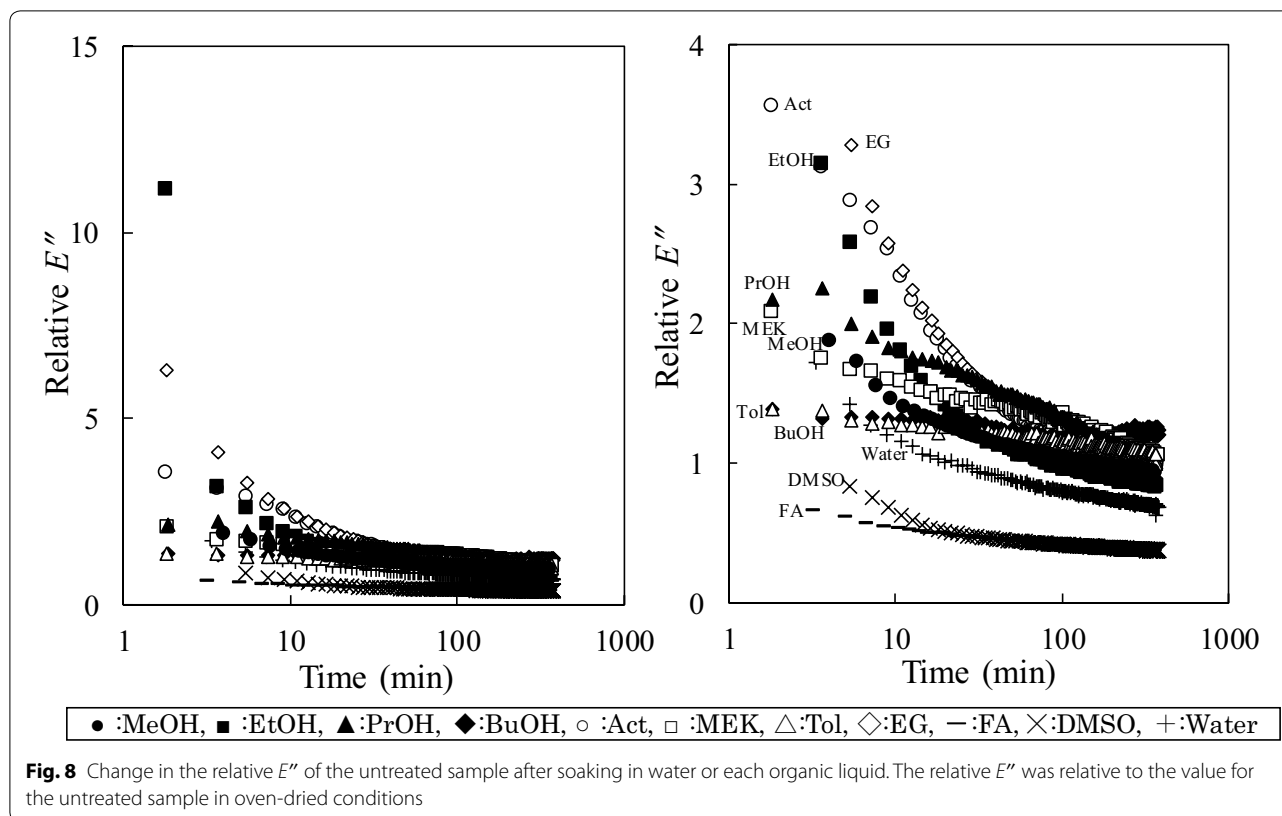
time during swelling process. Swelling rate of these samples was slow in Fig. 6. Therefore, it is considered that the reduction of relative E' was small because the destabilization caused by swelling was small in early stages of swelling. After that, the destabilization is caused during the swelling process; however, the stabilization does not occur sufficiently during measurement because the lignin is not much softened due to low swelling amount.

From the results in relative E' , it can be seen that the E' of the samples immediately after soaking in each liquid were decreased by swelling, and the degree of the reduction in E' was large in the samples with high swelling rate. Due to the balance between the stabilization and destabilization of the conformation of lignin, the change in the E' of the samples over time was different between the liquids. It was considered that the stabilization over time rapidly proceeded in the samples with high swelling amount because the degree of the thermal softening of lignin was large.

The change in E'' over time during the swelling process is shown in Fig. 8. Relative E'' was relative to the value for an untreated sample in oven-dried conditions. The result of relative E'' of 4 or less is also shown in addition to the whole result of relative E'' because the range of relative E'' changes significantly between the swelled liquids.

It is known that the E'' is decreased by the stabilization of the conformation of lignin [11]. Therefore, the rapid increase immediately after soaking in each liquid is thought to be caused by the destabilization of lignin. In all of the samples, the sample swollen by EtOH showed the largest relative E'' immediately after soaking in liquid. The sample swollen by EtOH did not show an extremely high swelling rate or swelling amount in Fig. 6; therefore, the destabilization would not be especially large. The sample swollen by EG resulted in a relatively large increase in E'' , which had a high swelling rate and swelling amount. However, the samples swollen by DMSO or FA with a high swelling rate and swelling amount, as well as the sample swollen by EG, indicated a low relative E'' and a slight change in relative E'' during all measurement times. The reason for the change in the relative E'' that could not be explained by the destabilization of lignin is considered below.

It is reported that the E'' of the sample swollen by various organic liquids that reached swelling equilibrium at 20 °C is decreased with increasing swelling amounts after the E'' reached a maximum at a relative swelling of approximately 30% [30]. The swelling amount and the swelling rate of the samples swollen by DMSO or FA were relatively high; therefore, these samples showed low relative E'' in the early stages of swelling. In addition, the sampling interval was relatively long in this measurement because the frequency was low. Therefore, the large



increase in relative E'' due to the destabilization of lignin possibly occurred in the short time after the sample was soaked in each liquid. Stabilization also proceeded immediately before the first sampling point. Therefore, the samples swollen by DMSO or FA that had low glass transition temperatures of lignin did not show large change in the relative E'' . In contrast, a large change in the relative E'' was found in samples swollen by EtOH, EG, or Act during this sampling interval.

The samples with a low swelling rate and swelling amount, such as the samples swollen by PrOH, MEK, Tol, or BuOH, were observed to have a low relative E'' and a small decrease in relative E'' over time. For these samples, the destabilization of the conformation of lignin was small due to the low swelling rate and swelling amount. In addition, the destabilization and stabilization of lignin simultaneously occurred during the swelling process. Therefore, the decrease in the relative E'' was small and the change in relative E'' over time was apparently little.

From the results in relative E'' , it can be seen that the E'' of the samples after soaking in each liquid rapidly increased and decreased over time. In addition, due to the swelling amount and swelling rate of each liquid, the rates of the destabilization and stabilization of the conformation of lignin are different. However, a rapid change in E'' could not be sufficiently observed in this

experiment. Therefore, further research is needed to discuss the destabilization and stabilization of lignin immediately after soaking the samples in each liquid, taking into account the measurement frequency and swelling time according to each swelling liquid.

Conclusion

The dynamic viscoelastic properties of the annealed and quenched samples were compared in untreated wood and acetylated wood swollen by water or various organic liquids. In addition, the changes in the dimension and the dynamic viscoelastic properties of the untreated sample were measured during the swelling process. The results obtained were as follows.

The destabilization of the conformation of lignin caused by quenching was mainly affected by the glass transition temperature, both in the untreated and acetylated samples swollen by water or organic liquid. The destabilization of the conformation of lignin caused by swelling was affected by the swelling rate, and the stabilization of the conformation of lignin over time was affected by the swelling amount. It was found that the destabilization caused by swelling is large in the sample with high swelling rate, but at the same time, the stabilization over time is also large due to the high swelling amount.

Abbreviations

tan δ : Loss tangent; E' : Storage elastic modulus; E'' : Loss elastic modulus; tan δ_q : tan δ measured after quenching; tan δ_a : tan δ measured after annealing; EtOH: Ethanol; PrOH: 2-Propanol; BuOH: 1-Butanol; Ac: Acetone; MEK: Methyl ethyl ketone; EG: Ethylene glycol; FA: Formamide; DMSO: Dimethyl sulfoxide; MeOH: Methanol; Tol: Toluene.

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Authors' contributions

YM: Writing the manuscript. YM, AS, NA: performed the experiment of the study and analyzed data. YM, KK, YF: conceived and designed the experiments. All authors read and approved the final manuscript.

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Availability of data and materials

The datasets used and/or analyzed during the current study are available from the corresponding author on reasonable request.

Competing interests

The authors declare that they have no competing interests.

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