Influence of moisture content on NO$_2$ sorption ability of cedar wood

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Abstract
This study examined the influence of moisture content (MC) on NO$_2$ sorption ability of cedar (Cryptomeria japonica) wood using the specimens with different MCs. Four MC conditions were applied, namely 0%, 8.6%, 12.7% and 16.5%, based on the differences of bonding condition of water molecule in the wood substances. As a result, the NO$_2$ sorption volume increased drastically due to the presence of water, and as the MC increased, the NO$_2$ sorption volume increased slightly. The NO generation volume and nitric acid concentration in the specimens were evaluated after an aeration test, and it was strongly indicated that the NO$_2$ reacted with water in the specimen, and that the NO generation volume increased as the content of multilayer adsorbed water increased. The nitric acid seemed to be formed by the reaction between NO$_2$ and water, and it was then held in the specimen. Based on the NO$_2$ sorption volume calculated using the NO generation volume, it was shown that the NO$_2$ sorption after 12-h aeration was caused mainly by the reaction between NO$_2$ and water. Furthermore, the presence of both water and extractives contributed to the NO$_2$ sorption in the initial period, and the presence of water contributed greatly to the NO$_2$ sorption over the long term.

Keywords: Japanese cedar wood, Nitrogen dioxide (NO$_2$) sorption, Moisture content

Introduction
Nitrogen dioxide (NO$_2$) is emitted mainly in the combustion of fossil fuels in industrial plants and vehicles, and in biological activity. Because NO$_2$ affects human health, environmental standards regarding NO$_2$ have been set by many countries as well as World Health Organization [1–4]. In many urban and industrial areas, the standards are not satisfied. There have been many reports about the monitoring of NO$_2$ concentrations in Asia and Europe [2, 5–7]. Also, there have been many reports that NO$_2$ concentration is related to respiratory symptoms and allergy [8, 9]. Recently, the number of asthma and allergy patients has increased, mainly in younger generations [10]. For the patients and their families, it is important to remove the indoor NO$_2$. For others, especially in urban areas, it is valuable to keep the living space healthy and comfortable.

It is known that cedar (Cryptomeria japonica) wood has a high NO$_2$ sorption ability. The structural features of tissues, moisture content (MC), and extractives are considered to be the main factors contributing to the NO$_2$ sorption ability. These factors have been evaluated roughly in previous studies [11, 12]. We reported precisely the influence of structural features of tissues and extractives using a measuring system to evaluate the NO$_2$ sorption volume [13, 14]. The structure of the tracheids in cedar wood contributed to increase of the interface area, which influenced the NO$_2$ sorption volume, and the specimen that had been dried naturally had greater NO$_2$ sorption ability than that dried in a kiln because it had a large amount of extractives. In addition, Abietadiene seemed to be one of the extractives that contributed to the NO$_2$ sorption ability. With regard to the contribution of MC, we clarified that the presence of water in the specimen enhances the contribution of the extractives...
to the NO\textsubscript{2} sorption ability [12]. Tsujino et al. reported that the NO\textsubscript{2} sorption volume increased when water was added to the specimen in which the NO\textsubscript{2} sorption volume was low [15]. In these previous studies, the NO\textsubscript{2} sorption ability was only evaluated either with or without moisture. However, under a condition with moisture, the bonding between the wood tissues and water takes different forms depending on the MC [16]. It is known that the NO\textsubscript{2} reacts with water [17]. Thus, the reactivity between NO\textsubscript{2} and water would be different depending on the MC. In addition, the MC of cedar wood changes according to the temperature and relative humidity in its usage environment. Therefore, to apply cedar wood as an NO\textsubscript{2} removal material, it is important to obtain the detailed data about the influence of MC. The purpose of this study was to clarify the influence of MC by comparing the NO\textsubscript{2} sorption volume among the four levels of MC. Also, we tried to evaluate the contributions of water and extractives on the NO\textsubscript{2} sorption.

### Materials and methods

#### Materials

Cedar heartwoods harvested in Kumamoto Prefecture in Japan were used. This wood was roughly 40 years old. In total, 160 discoid specimens with a longitudinal thickness of 1.5 mm and a diameter of 10 mm were prepared [12]. There were four levels of MC in the specimens, namely 0%, 8.6%, 12.7%, and 16.5%. The specimens at 0% MC were adjusted by drying with a freeze-drying device for more than 48 h [18]. The specimens at the other MCs were adjusted for at least 3 weeks in each desiccator at 20 °C and 34 ± 5.0%, 59 ± 5.0%, and 74 ± 5.0% relative humidity, respectively, by using saturated salt. The details of each MC condition are shown in Table 1. The proportion of late wood and the mean annual ring width were 17.2% and 5.56 mm, respectively.

#### NO\textsubscript{2} sorption test

The experimental system for the NO\textsubscript{2} sorption test is shown in Fig. 1. The NO\textsubscript{2} sorption test was conducted based on our previous study [11]. The conditions were as follows: NO\textsubscript{2} gas was aerated under a temperature of 20 ± 1.0 °C. The concentration of the NO\textsubscript{2} gas was adjusted to 1000 ppb, and the flow rate was adjusted to 560 ml/min [10]. The relative humidity was controlled by mixing damped air and dry air. The relative humidity was

### Table 1: The conditions of relative humidity and moisture content

| Relative humidity (%) | 0  | 34 | 59 | 74 |
|-----------------------|----|----|----|----|
| Moisture content (%)  | 0  | 8.6| 12.7|16.5|

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*Fig. 1. Experimental system for NO\textsubscript{2} sorption test. MFC refers to the mass flow controller which controls the flow rate. MFM refers to the mass flow meter which monitors the flow rate.*
The variation over time in NO concentration after NO2 gas passed through the specimen was monitored. The NO generation volume per minute at each aeration time $G$ (µmol) was calculated using Eq. (3). Then the total NO generation volume at aeration time $G_{NO}$ (µmol) was calculated using Eq. (4). A slight amount of NO that was generated by the reaction between NO2 and water in the gas and was included in the NO2 gas was detected in the blank test. Therefore, the average NO concentration monitored with the instrument only, before and after the experiment involving each specimen, was taken to be the reference value for the NO concentration:

$$G = ([NO] - [NO]_0) \times 10^{-9} \times \frac{f}{22.4} \times 10^6,$$

where $[NO]_0$ is the reference value for the NO concentration (ppb), $[NO]$ is the NO concentration after NO2 gas passed through the specimen (ppb), $f$ is the NO2 flow rate (560 mL/min), 22.4 is the volume (L) of 1 mol of the gas in the standard state, and $t$ is the aeration time (min).

In some conditions of this study, water was present in the aeration gas and specimens. It is known that NO2 reacts with water as shown in Eq. (a) [17]:

$$2NO_2 + H_2O \rightarrow HNO_2 + HNO_3$$

Nitrous acid (HNO2) decomposes easily as shown in Eq. (b) under the temperature higher than room temperature or under a high concentration because it is unstable [19, 20].

$$3HNO_2 + HNO_3 + H_2O + 2NO$$

Consequently, Eq. (c) results from the combination of Eqs. (a) and (b) [17]:

$$3NO_2 + H_2O \rightarrow 2HNO_3 + \ NO$$

To evaluate the reaction in Eq. (c), the variation over time in NO concentration after NO2 gas passed through the specimen was monitored. The NO2 sorption volume per minute at each aeration time $Q$ (µmol) was calculated using Eq. (1). Then the total NO2 sorption volume at aeration time $Q_{NO2}$ (µmol) was calculated using Eq. (2). The average NO2 concentration monitored with the instrument only, before and after the experiment involving each specimen, was taken to be the reference value for the NO2 concentration:

$$Q = ([NO2]_0 - [NO2]) \times 10^{-9} \times \frac{f}{22.4} \times 10^6,$$

$$Q_{NO2} = \int \{([NO2]_0 - [NO2]) \times 10^{-9} \times \frac{f}{22.4} \times 10^6 \} dt,$$

where $[NO2]_0$ is the reference value for the NO2 concentration (ppb), $[NO2]$ is the NO2 concentration after NO2 gas passed through the specimen (ppb), $f$ is the NO2 flow rate (560 mL/min), 22.4 is the volume (L) of 1 mol of the gas in the standard state, and $t$ is the aeration time (min).

To evaluate the reaction in Eq. (5) [22] is held in the specimen.
compare the nitrate ion (NO$_3^-$) concentration derived from HNO$_3$ between the different MCs, 5 discoid specimens after the aeration test were soaked in 5 ml ion-exchanged water at 20 °C for 24 h, then the nitrate ion concentration of the purified water was measured using the nitrate ion meter (LAQUA twin-NO3-11, HORIBA Ltd., Kyoto, Japan). The value of the specimen that was not used for the aeration test was taken to be the blank value, and then the value of the nitrate ion concentration was calculated by subtracting the blank value from the measured value.

Data analysis
For the NO$_2$ sorption volume and NO generation volume, values were obtained for 24 h [11], and then the average of three trials was taken. For the nitrate ion concentration, the average of three trials was taken. For the obtained results, one-way analysis of variance (ANOVA) was conducted as a test of the differences between the conditions. In one-way ANOVA, if a significant difference was found, then a multiple comparison test was conducted using the Tukey method. In all cases, significance level was 5%.

Results and discussion
The influence of the different MCs on the NO$_2$ sorption ability
Figure 2 shows the variation over time in NO$_2$ sorption volume, with the four different MCs. The NO$_2$ sorption volume was high soon after the aeration test started and then gradually decreased. After around 12 h, the reduction was small in all conditions. The behavior was confirmed to be almost the same even under different MC conditions. The specimen at 0% MC had the lowest NO$_2$ sorption volume, as a whole. Soon after the aeration test started, the NO$_2$ sorption volume at 8.6% MC was about twice as greater as that at 0% MC. The NO$_2$ sorption volume at 12.7% MC showed almost the same value as that at 8.6% MC, and the NO$_2$ sorption volume at 16.5% MC was the greatest. However, the differences between the NO$_2$ sorption volumes at 8.6% MC, 12.7% MC, and 16.5% MC were very small. The reductions of the NO$_2$ sorption volume at 0% MC and above 8.6% MC were small after around 6 and 12 h, respectively. The behavior of NO$_2$ sorption volume was different between before and after the reduction of the NO$_2$ sorption volume was small. Because the reduction of the NO$_2$ sorption volume of all conditions was small at around 12 h, it was evaluated by dividing the aeration time into two periods, namely, the periods from the start of aeration to 12 h (0–12 h), and from 12 to 24 h (12–24 h). Figure 3 shows the NO$_2$ sorption volume per unit weight and time of the specimens with different MCs for each aeration period. At 0–12 h, the NO$_2$ sorption volume at 0% MC was the lowest (0.48 µmol), and that at 8.6% MC (1.28 µmol) was 2.7 times greater than that at 0% MC. This shows that the NO$_2$ sorption volume increased drastically with the presence of water. The NO$_2$ sorption volume at 12.7% MC was 1.37 µmol, and that at 16.5% MC was 1.50 µmol, which showed that the NO$_2$ sorption volume increased slightly as the MC increased. However, the increase was less than the increase between the NO$_2$ sorption volume at 0% MC and that at 8.6% MC. To evaluate the influence of the MC on NO$_2$ sorption volume, one-way ANOVA was conducted. The results showed significant differences in the NO$_2$ sorption volume between MC conditions ($p < 0.01$). Multiple comparisons showed that the NO$_2$ sorption volume under the conditions with moisture was significantly greater than that at 0% MC. At 12–24 h, the NO$_2$ sorption volume at 0% MC and 8.6% MC showed almost the same value (0.15 µmol), and those at 12.7% MC and 16.5% MC were 0.20 µmol and 0.28 µmol, respectively. Under the conditions with moisture, the NO$_2$ sorption volume increased slightly as the MC increased. The result of one-way ANOVA showed significant differences in the NO$_2$ sorption volume between MC conditions ($p < 0.01$). Multiple comparisons showed that the NO$_2$ sorption volume at 16.5% MC was significantly greater than that at 0% MC and 8.6% MC. From the results above, it was suggested that the NO$_2$ sorption volume at 0–12 h was greater than that at 12–24 h under each MC condition. Furthermore, at 0–12 h, it was shown that the presence of water contributed greatly to the NO$_2$ sorption ability.

The following assumptions were made regarding the conditions of water in the specimens at each MC [14]. At 0% MC, it was assumed that water was not present. At 8.6% MC, it was assumed that water was present as monolayer and multilayer adsorbed water. In monolayer adsorbed water, hydrogen bonding between the water molecules and the hydroxyl of amorphous cellulose, hemicellulose, and lignin, which have hydrophilic property was formed on the surface of the tissue.
In multilayer adsorbed water, bonding by the van der Waals force and electrostatic attraction was formed onto the surface of the monolayer absorbed water. At 12.7% MC, the amount of multilayer adsorbed water increased to more than that was assumed at 8.6% MC. At 16.5% MC, the water was present as multilayer adsorbed water in a layer thicker than that was assumed at 12.7% MC. Based on the above, under the conditions in which water was present, it seems that the water molecules at 8.6% MC did not react easily with the NO₂ gas because some water molecules were present as a monolayer, which was adsorbed to the amorphous cellulose, hemicellulose, and lignin by a strong hydrogen bond. On the other hand, it seems that the water molecules at 12.7% MC and 16.5% MC reacted more easily than at 8.6% MC because the water molecules were adsorbed by the van der Waals force, which is weaker than the hydrogen bond. Therefore, it was considered that the NO₂ sorption volume increased as the MC increased, although the increase was slight. The behavior seemed to be different under the conditions with much higher MC due to capillary condensation. However, the experiment in this study was limited to conditions in which the bound water was present. Thus, a condition above 16.5% MC is needed to be examined in the future. In Japan, the equilibrium MC is about 15% [23], and it decreases to 10–12% in an air-conditioned environment. Therefore, it was elucidated that a high NO₂ sorption ability is observed in the range of humidity in real-life situations.

The reaction between NO₂ and moisture at different MCs

To evaluate the reaction between NO₂ and water (Eq. 5), the NO generation volumes at the four different MCs were compared. The results of the variation over time in NO generation volume are shown in Fig. 4. Under the condition at 0% MC, the NO generation volume was high soon after the aeration started and then decreased drastically. This behavior seemed to be caused by the NO that was generated due to the reaction between NO₂ and the surface of the tissue, and the polyphenol [17, 24]. After decreasing drastically, NO generation volume then decreased gradually. In this period, it was possible that water could not be removed from the specimen by the freeze-drying then remained slightly in the specimen. Therefore, NO seems to have been generated by the
reaction between NO₂ and the water. In addition, it was inferred that NO₂ reacted with the amorphous hydroxyl group on the surface of the tissue [17]. Under the conditions in which moisture was present, the NO generation volume was at its highest at about 3 h after the start of the aeration. With regard to the quantitative relationship, the NO generation increased as the MC increased. The greatest value at 16.5% MC was 1.4 times and 1.6 times greater than those at 12.7% MC and 8.6% MC, respectively. Compared with the behavior of the NO₂ sorption volume shown in Fig. 2, it took longer until the NO generation volume reached its greatest value. This suggested that NO₂ reacts with water on the surface of the tissue immediately after the start of aeration, and then takes time to diffuse. Also, it was presumed that the reaction between NO₂ and water continues for a long time because the NO generation volume did not reach equilibrium in 24 h under the conditions with moisture. The NO generation volume was also evaluated by dividing the aeration time into two periods as with the evaluation of the NO₂ sorption volume. Figure 5 shows the NO generation volume per unit weight and time of the specimens with the different MCs in each aeration period. At both 0–12 h and 12–24 h, the NO generation volume increased as the MC increased. To evaluate the influence of MC on NO generation volume, one-way ANOVA was conducted. The result showed significant differences in the NO generation volume at different MC conditions (p < 0.01). At 0–12 h, multiple comparisons showed that the NO generation volume of the specimen at 16.5% MC was significantly greater than the others (p < 0.01), and the NO generation volume of the specimen at 9.5% MC was significantly greater than that at 0% MC (p < 0.05). At 12–24 h, multiple comparisons showed that the NO generation volume of the specimen at 16.5% MC was significantly greater than those at 0% MC and 3.8% MC (p < 0.01 and p < 0.05, respectively). Considering that the MC in monolayer adsorbed water is in the range below 5–6% [16], the NO generation volume increased greatly, namely the reactivity between NO₂ and water would be enhanced by the presence of the multilayer adsorbed water. As a result, it was strongly indicated that the water in the specimen reacted with NO₂, and the NO generation volume increased as the content of multilayer adsorbed water increased.

Concentration of nitrate ion

In the reaction between NO₂ and water, HNO₃ is generated in addition to NO (Eq. 5). To evaluate the generated HNO₃, the concentration of NO₃⁻ was measured using the specimen after the aeration test. The results are shown in Fig. 6. The concentration of NO₃⁻ of the specimen at 0% MC was 4.9 ppm. This seemed to be formed by the reaction between NO₂ and the water that could not be removed from the specimen by freeze-drying and then remained in the specimen in slight amounts and that was present on the surface of the amorphous hydroxyl group [17]. The concentration of NO₃⁻ in the specimen at 8.6% MC was 8.78 ppm, which was about twice as high as that at 0% MC. It was suggested that HNO₃ was formed by the reaction between NO₂ and water under the 8.6% MC condition. Furthermore, the concentration increased linearly in the range from 0% MC to 12.7% MC, and then it decreased slightly in the specimen at 16.5% MC. To evaluate the influence of MC on the HNO₃ generation, one-way ANOVA was conducted. The result did not show significant differences in the concentration of NO₃⁻ between different MC conditions. It was expected that

![Fig. 5 NO generation volume per unit weight and time (0–12 h and 12–24 h), G_{GWNO}, with different MCs. The error bars show the standard deviation (n = 3). *:0.05 > p, **:0.01 > p (Tukey method)
the concentration of NO$_3^-$ would increase as the MC increased as in the case of NO generation. However, the concentration of the specimen at 16.5% MC was almost the same as that at 12.7% MC. Based on the discussion of the NO$_2$ sorption volume and NO generation volume, it was shown that the reaction between NO$_2$ and water occurred mainly in the 12–24 h. Because the results shown in Fig. 6 included the values in the whole aeration time of 0–24 h, it seemed that the relationship between the MC and NO$_3^-$ was not shown clearly. In addition, it was reported that nitrous acid (HNO$_2$) is also formed in the reaction between NO$_2$ and water (Eq. 3). Therefore, it is possible that HNO$_2$ was held in the specimen in addition to HNO$_3$ and the condition was different depending on each moisture content. From the above results, we need to examine the reaction between NO$_2$ and water in more detail. However, it was indicated strongly that HNO$_3$ was formed by the reaction between NO$_2$ and water, and then held in the specimen.

**Contribution of water**

According to Eq. (5), the NO$_2$ sorption volume can be calculated theoretically using the result of the NO generation volume shown in Fig. 4. The calculated value was compared with the actual measured value of the NO$_2$ sorption volume shown in Fig. 2. In addition, the NO$_2$ sorption volume results for the extracted specimens were compared. An evaluation of the contributions of water and extractives to NO$_2$ sorption was attempted by comparing these values. The results are shown in Fig. 7. In 0–12 h aeration, the calculated value was much lower than the measured value in every MC condition and showed different behavior from the measured value (extracted specimen). It was expected that the factors other than water would make large contributions to the NO$_3$ sorption volume. After 12 h aeration, the calculated value was almost the same as both measured values at 8.6% MC. As the MC increased, it took longer duration of aeration for the calculated value to match the measured values. The calculated values seemed to show the NO$_3$ sorption volume consumed by the reaction between NO$_3$ and water. Thus, it was implied strongly that the NO$_3$ sorption after 12 h aeration was caused by the reaction between NO$_2$ and water. To compare them in detail, the NO$_2$ sorption volume per unit weight and time of the specimen for each MC at 0–12 h and 12–24 h aeration are shown in Fig. 8. To evaluate the reaction between NO$_2$ and H$_2$O on NO$_2$ sorption volume at each aeration time, one-way ANOVA was conducted. The results at 0–12 h, showed significant differences between the measured values and the calculated value at every MC condition ($p<0.01$). Multiple comparisons showed that the NO$_2$ sorption volume of the measured value was significantly greater, five times greater, than that of the measured value (extracted specimen) at every MC condition ($p<0.01$). This seems to show the contribution of the extractives [12]. Also, it was considered that water contributed mostly to the NO$_2$ sorption of the extracted specimen, because there was no significant difference between the calculated value and the measured value (extracted specimen) of the NO$_2$ sorption volume at 8.6% MC and 12.7% MC. However, there was a significant difference between them at 16.5% MC ($p<0.05$). In the case of 16.5% MC, it is possible that the NO$_2$ sorption volume that was calculated from the NO generation volume was found to be greater than it actually was. To elucidate the reason for this, the NO$_3$ sorption ability under the higher MC conditions needs to be evaluated. At 12–24 h, because the result of one-way ANOVA did not show significant differences between the measured values and the calculated value, it was strongly implied that the NO$_2$ sorption was caused mainly by the reaction with water at every MC condition as discussed above.

We tried to compare the contribution ratios, namely, those of extractives only, water only, both extractives and water, and the others, to the NO$_3$ sorption under each MC condition based on the measured values at 0–12 h. The NO$_3$ sorption value in the non-extracted specimen at 16.5% MC was taken to be the reference value because it had the largest MC and the greatest NO$_3$ sorption volume under all conditions. The contribution ratio of extractives only was calculated using the NO$_3$ sorption volume in the non-extracted and extracted specimens at 0% MC using the following equation.

![Fig. 6 The concentration of NO$_3^-$ of specimens with different MCs after NO$_2$ aeration test. The error bars show the standard deviation ($n=3$)](image)
The contribution ratio of water only was calculated using the NO₂ sorption volume of the extracted specimen at each MC using the following equation.

\[
\text{Contribution ratio of water only (\%)} = \frac{(Q_{W \, \text{NO}_2} \text{ of non-extracted specimen} \, \text{at } 16.5\% \, \text{MC} \, \times 100)}{(Q_{W \, \text{NO}_2} \text{ of non-extracted specimen at } 16.5\% \, \text{MC})} - (Q_{W \, \text{NO}_2} \text{ of extracted specimen at } 0\% \, \text{MC} \, \times 100)
\]

The contribution ratio of extractives only was calculated using the NO₂ sorption volume of the extracted specimen at each MC using the following equation.

\[
\text{Contribution ratio of extractives only (\%)} = \frac{(Q_{W \, \text{NO}_2} \text{ of non-extracted specimen})}{(Q_{W \, \text{NO}_2} \text{ of non-extracted specimen at } 16.5\% \, \text{MC})} \times 100.
\]

The contribution ratio of both water + extractives was calculated by subtracting the difference between the NO₂ sorption in the extracted specimen at each MC and that at 0% MC, namely the influence of water only (A), from the difference between the NO₂ sorption of the non-extracted specimen at each MC and that at 0% MC, namely the influences of water only and of both water and extractives.

\[
\text{Contribution ratio of both water + extractives (\%) } = \frac{(Q_{W \, \text{NO}_2} \text{ of non-extracted specimen at } 16.5\% \, \text{MC} \, \times 100)}{(Q_{W \, \text{NO}_2} \text{ of non-extracted specimen})} - (Q_{W \, \text{NO}_2} \text{ of extracted specimen at } 0\% \, \text{MC} \, \times 100).
\]

The left ratio was taken to be the contribution ratio of factors other than water and extractives. The results of the calculation are shown in Fig. 9. The contribution ratio of extractives only was 23.4%. Under the conditions with moisture, the contribution ratio of water only ranged from 4.1 to 6.7%, which was close to the value found in the previous study [12]. The contribution ratio of both water + extractives was the highest, and it increased slightly as the MC increased. The above results implied that the presence of water and extractives together contributed greatly to the NO₂ sorption.
ability. This might be because the extractives changed due to the presence of water, and the extractives could then make contact with NO₂ gas more easily [25]. The details need to be examined in the future.

**Conclusion**

NO₂ aeration test was conducted using specimens of cedar wood under different MCs, and the NO₂ sorption volume was then evaluated focusing on the status of water in the specimen. As a result, it was elucidated that the MC contributes greatly to the NO₂ sorption, and the NO₂ sorption volume increases as the MC increases above 8.6% MC. Also, it was implied that the presence of both water and extractives together contributes to NO₂ sorption in the initial period and water contributes greatly to the NO₂ sorption over the long term. Furthermore, it was implied strongly that the nitric acid that was formed by the reaction between NO₂ and water was held in the specimen.
Abbreviations
MC: Moisture content; ANOVA: Analysis of variance.

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Declarations
Competing interests
The authors declare that they have no competing interests.

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