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ORIGINAL ARTICLE

Predicting oven-dry density of Sugi (*Cryptomeria japonica*) using near infrared (NIR) spectroscopy and its effect on performance of wood moisture meter

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Abstract We propose a non-destructive method to predict the oven-dry density of Sugi (*Cryptomeria japonica* D. Don) using near infrared (NIR) spectroscopy so as to calibrate a commercial moisture meter. A prediction model for oven-dry density was developed using NIR spectra obtained from Sugi samples with a known density. The density of air-dried Sugi boards was predicted with the developed model. Then, the moisture content (MC) of the boards was measured by a hand-held capacitance-type and an in-line microwave moisture meters. For each board, the moisture meters were calibrated by the predicted density. The predicted density was correlated with the measured one with an R^2 of 0.81 and a standard error of prediction (SEP) of 15.3 kg/m^3 within the measured density of 279.2–436.4 kg/m^3, indicating that the developed model was applicable for predicting oven-dry density of Sugi. The MC readings of both moisture meters showed a good correlation with the oven-dry MC that ranged from 12.1 to 28.9 %. For both moisture meters, the density calibration with the NIR-predicted density gave a higher R^2 and a lower SEP than with the conventional calibration with the mean density. These results demonstrate that the present density calibration using NIR spectroscopy could improve the performance of the moisture meters for the air-dried Sugi boards with varying densities.

Keywords Moisture meter · Density calibration · Near infrared spectroscopy · Sugi (*Cryptomeria japonica*) · Oven-dry density

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Introduction

Inspection of the moisture content (MC) in dried lumber is essential for wood industry to ensure dimensional stability in service. Several types of commercial moisture meters authorized by Japan Housing and Wood Technology Center (HOWTEC) (e.g. resistance-type, capacitance-type, microwave and neutron moisture meters) have been used to check MC of lumber in sawmills. These moisture meters are designed to perform a calibration based on wood density, because density has a significant influence on MC readings obtained with these moisture meters [1]. For instance, Milota [2] used a capacitance-type moisture meter to measure MC of samples of eight species conditioned sequentially at 22, 18, 12 and 8 % equilibrium MC, and demonstrated that the performance of the moisture meter could be improved by density calibration. Using a permeable neutron moisture meter, Moriya et al. [3] recently examined the effect of density on thermal neutron by which the MC of lumber is measured, and concluded that density calibration is critical for MC measurement with high accuracy. It is, however, difficult to measure the density of each lumber in sawmills, because density determination is traditionally done by a volumetric method that is accurate but destructive and time consuming. As a consequence, a commercial moisture meter is calibrated approximately by setting known, constant density values to each species, resulting in an error in MC reading. Thus, a method for swiftly and non-destructively measuring the density of lumber is being required.

An alternative for measuring wood density is near infrared (NIR) spectroscopy, which offers a rapid, non-destructive method for predicting many wood properties [4]. Bands in the NIR spectra of wood arise from the vibrations of chemical bonds in various components, such

as cellulose, hemicellulose, lignin and extractives. Relative amount of these components largely determines cell wall density and could influence overall density. For solid wood, the macro characteristic that affects density is lumen diameter and cell wall thickness. Having more cell wall material and less lumen diameter implies that more material is available for absorbance across all wavelengths in the NIR region [5, 6]. Thus, NIR spectroscopy could successfully predict the density of solid wood based on NIR spectra collected from different species, sample size, grain orientation and MC range in a large number of studies listed in Table 1 [4–36]. Density is expressed in the scientific literature as basic density (oven-dry mass per green volume), air-dry density (moist mass per moist volume) or oven-dry density (oven-dry mass per oven-dry volume). Among them, oven-dry density has been often used as a parameter of the density calibration for commercial moisture meters, but the prediction of oven-dry density using NIR spectroscopy has been seldom examined in the past studies (Table 1). In addition, few studies have reported the

Table 1 Studies that successfully predicted the density of solid wood based on NIR spectra from different species, sample size, wood surface and MC range

Density	Species	Sample	Wood surface	MC range	Reference
BD	*Picea abies*	Disk	Tr	89–211 %	[7]
AD	*Picea abies*	Small block	Tr	<30 %	[4]
AD	*Eucalyptus delegatensis*	Strip	R	Around 12 %	[5]
OD	*Larix decidua*	Small block	R	12.3 ± 0.3 %	[8]
AD	56 species from around the world	Strip	R or T	Around 12 %	[9]
AD	*Pinus radiata*	Strip	R	Air-dried state	[10]
AD	*Pinus palustris*	Strip	R	Air-dried state	[6]
AD	*Pinus radiata*	Strip	R	Air-dried state	[11]
AD	*Pinus taeda*	Small block	R, Tr	Around 7 %, 100–154 %	[12]
AD	*Pinus taeda*	Strip	R	Air-dried state	[13]
AD	*Pinus taeda*	Small block	R, T	100–154 %	[14]
AD	*Pinus taeda*	Strip	R	Air-dried state	[15]
AD	*Pinus taeda*	Strip	R	Air-dried state	[16]
SG	*Pinus taeda*	Small block	R, Tr	12 %	[17]
AD	*Pinus densiflora, Zelkova serrata*	Small block	R	12 % on average	[18]
AD	*Pinus palustris*	Strip	R	Air-dried state	[19]
AD	*Eucalyptus nitens*	Strip	R	Air-dried state	[20]
AD	*Pinus taeda*	Strip	R	Air-dried state	[21]
AD	*Larix gmelinii Larix kaempferi*	Small block	R	13.1 % on average	[22]
BD	*Quercus* spp.	Small block	R, T, Tr	68–100 %	[23]
SG	*Pinus taeda*	Increment core	R	Green state, air-dried state	[24]
AD, BD	*Pinus taeda*	Strip	R	Air-dried state	[25]
AD	*Larix gmelinii, Larix kaempferi*	Lumber	T	13.1 % on average	[26]
BD	*Eucalyptus urophylla*	Small block	R, T, Tr	12 %	[27]
SG	*Pinus taeda*	Strip, log	R for strip, Tr for log	100–154 % for strip, 70–200 % for log	[28]
AD	*Pinus taeda*	Strip	R	Air-dried state	[29]
BD	*Eucalyptus nitens*	Increment core	–	Green state	[30]
AD	*Larix kaempferi*	Lumber	T	8.6–9.5 %	[31]
AD	*Larix kaempferi*	Lumber	T	10.6 % on average	[32]
AD	*Abies balsamea, Picea mariana*	Stick	R	Green state, air-dried state	[33]
SG	*Eucalyptus tereticornis*	Small block	R, T	7–16 %	[34]
AD	*Eucalyptus camaldulensis*	Strip	–	Air-dried state	[35]
AD	*Pinus pinaster Aiton, Larix × eurolepis*	Strip	T	Air-dried state	[36]

AD air-dry density, *BD* basic density, *SG* specific gravity (the ratio of oven-dry mass per moist volume to the density of water), *OD* oven-dry density, *Tr* transverse surface, *R* radial surface, *T* tangential surface

applicability of NIR spectroscopy for Sugi (*Cryptomeria japonica* D. Don), which has more variable wood properties than other species [37].

The first objective of this study is to investigate the applicability of NIR spectroscopy for predicting the oven-dry density of Sugi. A prediction model for oven-dry density was developed using NIR spectra obtained from Sugi samples with known density and MC in a hygroscopic range. The second objective is to investigate the effect of the density calibration using NIR spectroscopy on the performance of moisture meters. The oven-dry density of air-dried Sugi boards was predicted using the developed model. Then, MC of the boards was measured by a hand-held capacitance-type moisture meter and an in-line microwave moisture meter, respectively. For each board, the moisture meters were calibrated by the predicted density, and the MC readings were compared with those approximately calibrated by the known mean density of the boards.

Materials and methods

Development of density prediction model

The density prediction method employed in this study consisted of mainly 4 steps; sample preparation, NIR measurement, development of prediction model and model validation. The procedure of the density prediction method is shown in Fig. 1.

Thirty green logs of Sugi were obtained from Ibaraki Prefecture in Japan. Three sticks (40 × 40 mm^2 in cross-section and 300 mm long) were cut from each log: one from juvenile wood, one from mature heartwood and one from sapwood (Fig. 2). The sticks were dried in an oven at a temperature of 50 °C for no less than 5 days, and then five small samples with dimensions of 33 × 33 mm^2 in cross-section and 3.5 mm in the longitudinal direction were cut from each stick to obtain a total of 450 samples. The samples did not contain any knots, while large color variation was observed within and between samples mainly due to black-colored heartwood observed in 55 samples from juvenile wood and mature heartwood. Thereafter, the samples were conditioned in two conditioning rooms sequentially at temperature and relative humidity of 20 °C/65 %RH and 20 °C/75 %RH until the constant weight was attained.

NIR spectra were acquired before and after each conditioning. Diffuse reflectance spectra on a spot diameter of approximately 2 cm were collected at 5-nm intervals over the range of 715–2500 nm using a Fourier transform NIR spectrometer (MATRIX-F, Bruker Optics Inc., Ettlingen, Germany) equipped with a NIR fiber optic probe and four

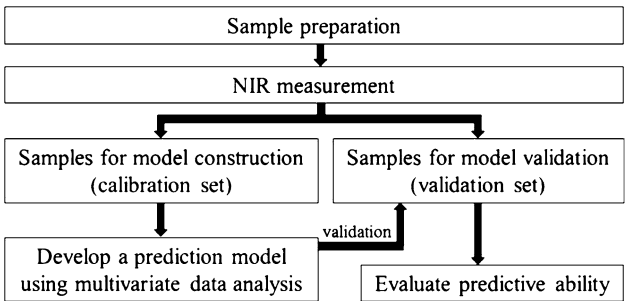

Fig. 1 Procedure of density prediction method based on NIR spectroscopy

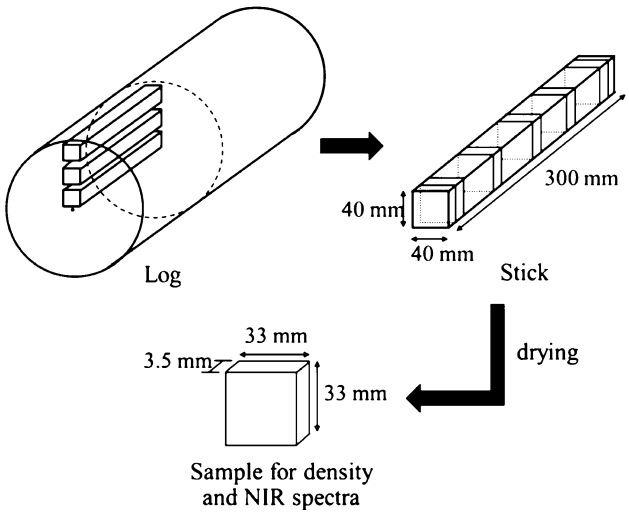

Fig. 2 Sample preparation procedure

tungsten light sources. A piece of commercial resin Spectralon was used as the reference material. Although it is common to collect NIR spectra from radial or tangential surfaces of wood (Table 1), NIR spectra were collected from a cross-sectional surface of the sample. This is because, in a real situation, a cross-sectional surface in a lumber's end is available for NIR measurement, whereas radial and tangential surfaces cannot always appear on the surface of lumber. To reduce the acquisition time, only five scans were accumulated and averaged into a single spectrum, while, in general, more scans were acquired for the prediction of density [4–6, 8–16, 19–21, 23, 26, 27, 31–36]. A total of 1350 spectra were collected from the 450 samples with varying MC.

After NIR measurement, the samples were oven-dried at a temperature of 103 ± 2 °C. The dimensions and weight of the samples were measured to obtain oven-dry density and MC. Since the cross-section of the oven-dry sample was distorted anisotropically, the area of the cross-section was calculated from a cross-sectional image taken by a commercial image scanner, while the thickness was

Table 2 Oven-dry density and MC of the samples used for the calibration and prediction sets

Constituent	Sample set	*n*	Mean	SD	Max	Min
Oven-dry density (kg/m^3)	Calibration	270	332.1	45.4	559.0	243.0
	Validation	180	328.4	44.9	567.1	243.8
MC before conditioning (%)	Calibration	270	7.5	0.9	12.5	5.6
	Validation	180	7.4	0.9	12.7	5.9
MC at 20 °C/65 %RH (%)	Calibration	270	11.8	0.8	16.4	10.0
	Validation	180	11.8	0.8	16.4	10.0
MC at 20 °C/75 %RH (%)	Calibration	270	14.0	0.7	17.8	11.9
	Validation	180	13.9	0.7	17.9	12.2

SD standard deviation

measured using a digital caliper with a sensitivity of 0.01 mm. Each pixel of the image corresponded to an area of 0.127 × 0.127 mm^2, and the pixels of the cross-section were counted by means of ImageJ software [38]. From a total of 450 samples, 270 samples were randomly selected as a calibration set, and the remaining 180 samples were used as a validation set. Table 2 provides a summary of oven-dry density and MC of the samples used for each set.

A prediction model for oven-dry density was developed with the NIR spectra of the calibration set using Unscrambler software (CAMO, Corvallis, OR, USA). The raw reflectance spectra were converted to absorbance. Pre-processing techniques used to enhance the quality of prediction models, such as first and second derivatives or multiplicative scatter correction, were not used, because little improvement was observed in the models pre-processed by these techniques. Partial least squares (PLS) regression analysis in full cross-validation was used to construct a prediction model. The number of factors which corresponded to the first minimal residual variance was adopted as optimum. No outlier was removed from the model.

The oven-dry density in the validation set was predicted using the prediction model. The quality of the model was evaluated by comparing the predicted values with the measured ones. The coefficient of determination (R^2), the standard error of calibration (SEC), the standard error of prediction (SEP) and the ratio of performance to deviation (RPD) served as the statistical measures of predictive ability. SEP was used to measure how well the model predicts the parameter of interest for a set of unknown samples excluding the calibration set. RPD, the ratio of the standard deviation of the reference data to the SEP, allows comparison of models developed for wood density that have differing data ranges. An RPD value of >2.5 is considered satisfactory for screening [39], but an RPD value of approximately 1.5 is sufficient for initial screening [40].

MC measurement using moisture meters

Thirty green square lumbers of Sugi (115 × 115 × 3000 mm^3) were obtained from Ehime Prefecture in Japan. Each lumber was cut to yield 4 boards with the dimensions of 35 × 110 × 300 (longitudinal direction) mm^3. Both end surfaces of the boards were sealed with silicon sealant and air-dried for 32 days in a room temperature atmosphere. After air-drying, the boards were finished by a planer and their sealed end section was cut off. Consequently, a total of 120 boards with the final dimensions of 30 × 110 × 250 (longitudinal direction) mm^3 were prepared. The boards were placed for 1 day in a conditioning room at a temperature and relative humidity of around 20 °C and 30 %RH until just before NIR measurement.

NIR measurement was conducted in the same manner as above except that four NIR spectra were acquired from an end surface of each board and averaged. The oven-dry density was predicted from the NIR spectra using the developed model. Subsequently, the end surface of 4 mm in thickness cut from each board was oven-dried, and its oven-dry density was measured gravimetrically. The boards were allowed to contain checks in the end surface where NIR spectra were collected, whereas the surface did not contain any knot.

A handheld capacitance-type moisture meter (Moco II, Kett Electric Laboratory, Tokyo, Japan) and an in-line microwave moisture meter (MM-94L, Kawasaki Kiko Co., Shizuoka, Japan) were used to measure MC of the boards. It should be noted that the moisture meters must be used correctly, and the calibration equation for the microwave moisture meter was obtained preliminarily as a function of microwave attenuation, moisture content and density in accordance with the manual of the manufacturer. First, meter readings were taken from the middle of the boards by setting the density value to the mean measured density of 355 kg/m^3. Second, meter readings were taken again by setting the density value to the NIR-predicted value obtained from the end surface of each board. Thereafter, the boards were oven-dried and their MC was calculated gravimetrically. Twelve boards had oven-dry MC above 30 % and were considered as "wet". Therefore, the wet boards were eliminated and the remaining 108 boards were used for MC evaluation.

The MC readings from the in-line microwave moisture meter were assessed if the moisture meter could fulfill the

performance requirement of in-line moisture meter for softwood lumber [41]. According to the performance requirement, the criterion required for the moisture meter is expressed as follows:

$$|M_1 - M_2| \leq 0.12 \times M_2$$

where M_1 is the moisture reading, M_2 is the oven-dry MC. Fifteen dried test specimens (at least 1 meter long) having oven-dry MC range of 10–25 % are prepared for the test. When more than 13 test specimens satisfy this criterion, the in-line moisture meter is approved by HOWTEC.

Results and discussion

Quality of density prediction model

Table 3 summarizes the statistics for the prediction model. The model had high R^2 of 0.85 for the calibration set and 0.85 for the validation set, low SEC of 17.4 kg/m^3 and low SEP of 17.6 kg/m^3. The RPD value of 2.54 shows that the model was satisfactory for screening. These results were comparable with those reported by Kothiyal and Raturi [34], who predicted the specific gravity of *Eucalyptus tereticornis* strips with various MC of 7–16 % and reported R^2 of 0.58–0.75, SEP of 20–22 kg/m^3 and RPD of 2.67–3.00. For oven-dry density, Gindle and Teischinger [8] developed a prediction model using NIR spectra taken from solid *Larix decidua* Mill. samples with MC of around 12 % and obtained an R^2 of 0.95 and a root mean square error of cross-validation (RMSECV) of 23 kg/m^3. Although their prediction model was not validated with external samples excluding the calibration set, the quality of the model is not significantly different from that obtained in this study, despite varying MC (5.9–17.9 %) and large color variation of the samples. These results demonstrate the applicability of NIR spectroscopy for predicting the oven-dry density of Sugi with various MC in a hygroscopic range.

The optimum number of factors of 13 used in the model was comparable with those reported by Galleguillos-Hart et al. [30], who modeled the basic density of green *Eucalyptus nitens* with transmitted NIR spectra from increment cores. However, the required number of factors in the present study was considerably more than those reported by most of other studies listed in Table 1 [4–27, 29–36]. This is probably due to the large spectral variation caused by a large variability in heartwood color peculiar to Sugi species [37], as well as varying MC of the samples.

To evaluate the quality of NIR models for predicting wood density, we validated the prediction model using the validation set excluded in the model construction [4–7, 9–17, 19–23, 25–36]. However, this evaluation is not adequate to simulate a real situation when the density of unknown samples should be predicted by developed NIR models [27]. Hence, the robustness of the prediction model was evaluated using boards obtained from a different site, totally independent from the samples used for the model construction. The laboratory-measured oven-dry density and MC of the board's end of 4 mm in thickness where NIR spectra were collected ranged as 279.2–436.4 kg/m^3 with a mean of 354.8 kg/m^3 and 10.5–22.5 % with a mean of 13.8 %, respectively. Relationship between the laboratory-measured oven-dry density and that predicted by NIR spectra is shown in Fig. 3. The predictive ability of NIR spectra was good with an R^2 of 0.81, a SEP of 15.3 kg/m^3 and an RPD of 2.14, indicating that the model was robust and applicable to populations in a different region. Compared with the statistics for the validation set presented in Table 3, the prediction model gave slightly poorer R^2 and RPD, but a better SEP, because the oven-dry densities of the boards were within the range of the samples in the validation set.

Table 3 Results of density prediction model

Optimum number of factors	Calibration set		Validation set		
	R^2	SEC	R^2	SEP	RPD
13	0.85	17.4	0.85	17.6	2.54

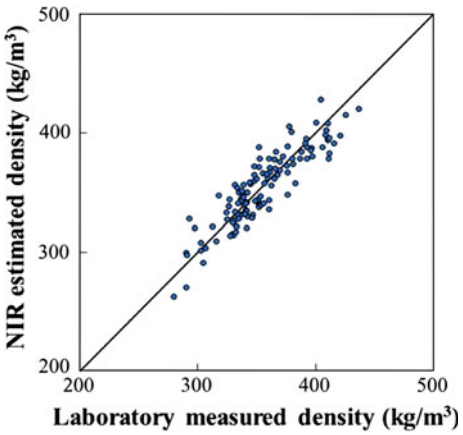

Fig. 3 Relationship between laboratory-measured oven-dry density of the board's end of 4 mm in thickness and NIR-predicted one. *Solid line* a one–one relationship between measured and estimated values

Effect of density calibration on performance of moisture meter

The oven-dry MC of the boards ranged from 12.1 to 28.9 % with a mean of 20.2 %. There was a MC gradient from the core to the surface within the boards. Table 4 summarizes the results of the MC measurement using the

Table 4 Results of MC measurement using a hand-held capacitance-type moisture meter and an in-line microwave moisture meter

Density calibration	Hand-held		In-line	
	R^2	SEP	R^2	SEP
Mean measured density	0.75	5.8	0.78	1.8
NIR-predicted density	0.82	5.0	0.83	1.6

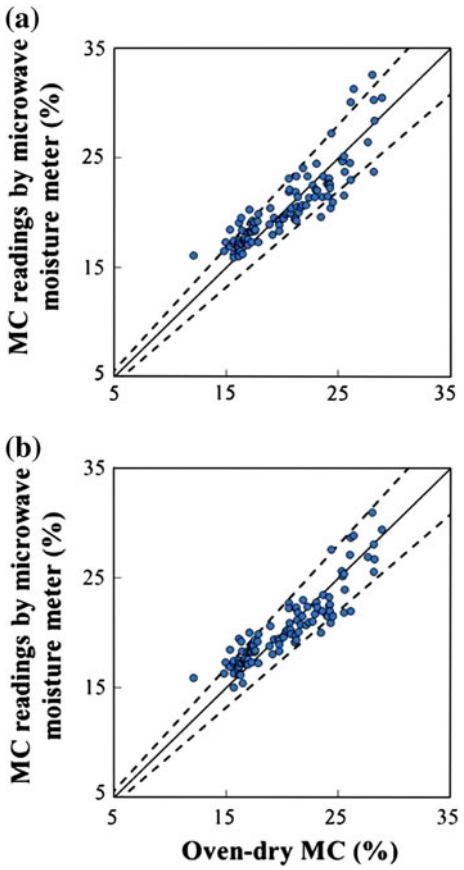

Fig. 4 Plots of oven-dry MC versus MC reading from the microwave moisture meter. The moisture meter was calibrated by the known mean density of the boards (**a**) and NIR-predicted density (**b**), respectively. *Dotted line* indicates ±12 % range of the oven-dry MC. *Solid line* a one–one relationship between oven-dry MC and MC reading

hand-held capacitance-type moisture meter and the in-line microwave moisture meter, respectively. The MC readings of both moisture meters showed a good correlation with the oven-dry MC with R^2 of 0.75–0.82 (hand-held moisture meter) and 0.78–0.83 (in-line moisture meter), respectively. The much lower SEP values for the in-line moisture meter than those for the hand-held moisture meter indicate that the in-line moisture meter measured MC more accurately than the hand-held moisture meter. This may be due to two possible causes. The first one is the difference in measurement area, and the second one is the effect of wood properties on the moisture meters. In the in-line microwave moisture meter, microwave attenuation is measured when microwave passes through an entire board. The attenuation is mainly dependent on moisture, and represents average MC across the board thickness. In contrast, the hand-held moisture meter basically measures dielectric constant between two electrodes which come into contact with a board surface. Therefore, it is difficult to measure average MC across the board thickness. Furthermore, MC is converted from the dielectric constant that is strongly dependent on the grain orientation of wood as well as the moisture and density [42]. Thus, the relatively smaller measurement area and the effect of wood properties resulted in the lower precision in the hand-held capacitance-type moisture meter.

For both moisture meters, the density calibration with the NIR-predicted density gave a higher R^2 and a lower SEP than with the density calibration with the known mean density (Table 4). Thus, density calibration using NIR spectroscopy could improve the performance of the moisture meters. Figure 4 shows the plots of oven-dry MC versus MC reading from the in-line moisture meter calibrated with the known density and with NIR-predicted density. For both density calibrations, MC readings had good correlations with the oven-dry MC, and most of the readings were within the ±12 % range of the oven-dry MC. In the plots using the mean density calibration, 16 out of 108 boards fell outside the ±12 % range, while in the plot with NIR-predicted calibration, only 12 fell outside the range. Therefore, we consider that in terms of the accuracy of measuring MC, the in-line moisture meter calibrated by NIR-predicted density reasonably fulfilled the performance requirement of an in-line moisture meter for softwood lumber [41], although the size and MC range of the studied boards did not strictly satisfy the specification of the requirement.

The MC of the boards was not measured using the moisture meters, setting the density values that were obtained gravimetrically for each board from the end section of 4 mm in thickness. So the performance of the moisture meters calibrated by NIR-predicted density could not compared with the one calibrated by the individual measured density. Nonetheless, this study demonstrates that NIR spectroscopy could swiftly predict oven-dry density of the end surface of the Sugi boards. This finding shows that NIR spectra acquired from an end surface of lumber may be useful for density calibration of moisture meter. In this study, the board length was 250 mm, and NIR-predicted density could be considered to represent the density of whole-board. Thus, density calibration using NIR spectroscopy was successful. It should be pointed out, however, that an accuracy of density calibration with NIR spectra acquired from an end surface may decrease with

increasing lumber length, due to density variation along the longitudinal direction. For improving density calibration, it may be necessary to collect NIR spectra from both ends of lumber and average them. There are also a few limitations that should be considered with regard to potential future applications. In a real situation, knots often appear on an end surface of lumber, and NIR spectrum acquired from such a surface probably leads to an error in density calibration. In addressing this issue, hyperspectral NIR imaging [43] or scanning NIR probe [44] can be utilized to map the spatial distribution of NIR spectra on surfaces. Besides, NIR spectroscopy has been shown to be able to identify the presence of knots [45]. By combining these techniques, it may be possible to distinguish a knot region from a clear region in the end surface of lumber and to predict the density of lumber with NIR spectra obtained solely from the clear region. If the above issues are to be resolved, the density calibration system based on NIR technology will be applicable to commercial lumbers.

Conclusions

This study investigated the applicability of NIR spectroscopy to predict the oven-dry density of air-dried Sugi boards and to calibrate a hand-held and an in-line moisture meters for measuring the MC of the boards. Our results showed that the NIR prediction model for the oven-dry density was robust and applicable to populations in different regions. Performance of moisture meters could be improved by calibration with NIR-predicted density. Furthermore, the in-line moisture meter calibrated by NIR-predicted density reasonably fulfilled the performance requirement of in-line moisture meter for softwood lumber. However, it is uncertain that the density calibration using NIR spectroscopy is applicable to commercial-size lumbers that often contain knots. Nonetheless, the newly developed method to non-destructively calibrate commercial meters will allow us to measure the MC of air-dried Sugi lumber with varying densities more accurately than the conventional density calibration with constant values representing each species.

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