名古屋大学

Construction of combinative nondestructive measurement system for wood properties

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Chapter I: General introduction

Veneer and lumber are the importance in wood product and increasing demand in wood industry. According to "Annual Report on Forest and Forestry for FY2016", total wood supply/demand increased 0.8 % in 2015 with plywood 11% and saw wood is 3% increasing than previous year. The report also described that sugi (Japanese cedar) and hinoki (Japanese cypress) were the first and third important ranks in planted area/tree species in 2015. Nowadays, the price of wood product is increasing as the forest resource is decreasing. Thus, to use wood to its best advantage and most effectively in engineering applications, specific characteristics or physical properties must be considered. Various approaches have been developing to evaluate the properties of wood with many advantages like nondestructive, rapid and without sample preparation. Moisture content (MC) and density are the important properties that affect to the product process and use of wood material. In this study, we investigate two non-destructive methods including capacitance sensor and NIR spectrometer for evaluating these traits for wood.

High frequency capacitance method for predict MC and density of wood

Electric moisture meters have been used for many years in process and quality control, for MC determination of lumber, grains, soils, and so on. These MC meters is determined by measuring electric resistance, capacitance, or admittance (impedance). In the recent time, capacitance sensor has been using in wood material more popularly with the significant improvement of device. Electrical properties of wood vary with MC, so they can be used to measure MC reasonably accurately and very quickly. There are several factors which can affect the dielectric parameters of wood such as volume porosity, pore size, pore distribution, MC and density were the subject of many reports (Bossou, et al. 2010, Kabir, et al. 1998, Olmi, et al. 2000).

Near Infrared (NIR) spectroscopy for evaluate MC and density of wood

NIR spectroscopy is becoming a modern approach for academic research and industry quality control in a wide of applications ranging from chemistry to agriculture and from life science to environment analysis ((Mercer, et al. 2018, Verstraeten, et al. 2018, Manley and Shi 2018). Many researchers have reported studying the chemical composition, physical and mechanical properties, and anatomical structure of wood (Tsuchikawa, et al. 2013, Tsuchikawa, et al. 2015, Watanabe, et al. 2011, Karttunen, et al. 2008, Xu, et al. 2011, Defo, et al. 2007). NIR region contains absorption bands with the overtones and combinations of fundamental vibrations (Siesler, et al. 2008) due to hydrogen group (CH, OH, NH, …) vibrations (Ciurczak and Burns 2001, Kawano 1995). Basic NIR device set up for analysis: NIR spectrophotometer contains light source, monochromator and detectors to get the spectrum (Dyrby, et al. 2002). Some pretreatment methods sometimes are applied to remove physical phenomena in the spectra in order to improve the subsequent multivariate regression, classification model or exploratory analysis such as: scatter-correction methodps and spectral derivatives. Due to overlapping of overtones, PLS is applied to convert the complex spectral data into analytical parameters to maximum the covariant between the matrix X and matrix Y through the linear combinations of their scores and loadings. The PLS is based on the calculation of the reduction in variables, where the X (spectral) and Y (analyte) matrices are decomposed simultaneously in Fig. I-1.

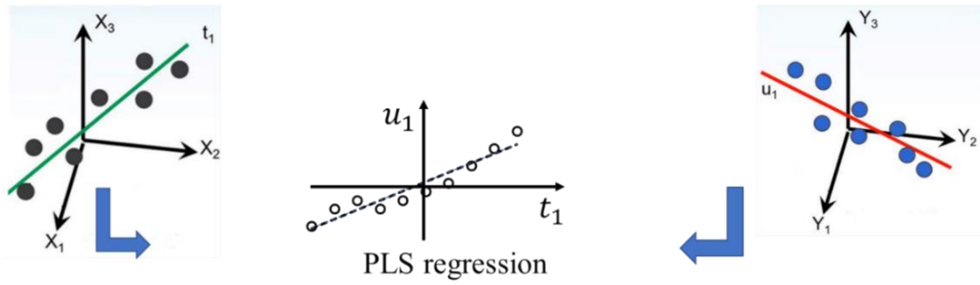

Fig. I-1 PLS aims to maximize the covarion between X matrix and Y matrix.

General concept for combining capacitance and NIR absorbance at 2 optimal wavelengths

Both techniques are nondestructive, rapid and without sample preparation; however, they have limitations. Capacitive sensors are not the fastest technology and can be slower than competing technologies. NIR spectroscopy has problems with a large data required calibration. Therefore, the purpose of this study was to investigate two devices and conducted analyses to achieve the best value regression model to develop a better procedure for experimentally assessing the density and MC of wood. There are some questions were justified:

1. What are the results attained from the capacitive device and micro NIR spectrometer for various wood species and sample thicknesses from the thin to thick thickness?
2. How about the accuracy from each device and from different range of moisture?
3. How will the data be analyzed to achieve a robust model as combing both two devices with the capacity and NIR absorbance data at two wavelengths?

Chapter II: Predicting the moisture content and density of veneer by combining of Capacitance sensor and NIR spectroscopy

Literature view

In this study, we investigated some non-destructive methods to evaluate MC and density of veneers. This research studied these two devices and conducted analyses to achieve the best models for assessing the density and MC of veneers. Two questions were answered: a) What are the results attained from them in various wood species and thicknesses? b) How will the data be analyzed to achieve a robust model? We also develop the robust models by combining the capacity and NIR absorbance at two wavelengths.

Materials and methods

The 14 species included softwood and hardwood to investigate the material from refresh to air-dried state. Three sample batches were prepared for the study with the dimensions of (2,6,12) x 50 x 150 mm (thickness x radial x tangential) for each species, namely batch 1 (2 mm thickness), batch 2 (6 mm thickness), and batch 3 (12 mm thickness). A high-frequency capacitance device with the frequency of 20 MHz (Electrical moisture meter HM-530, Kett Electric Laboratory, Tokyo, Japan) and A Portable NIR spectrometer (MicroNIR™ OnSite, © 2015 Viavi Solutions Inc, USA) were used to collect the data on the tangential section. MATLAB software was used for data analyses. Three calibration methods were used:

Method I: Multiple linear regression (MLR) using the capacity data for prediction

Method II: Partial least squares (PLS) regression using NIR spectral data

Method III: MLR for the capacity and NIR absorbance at two wavelengths was conducted.

Results and discussions

In Method I, the prediction accuracies were lower compared to Methods II and III for each thickness value for both density and MC. When a comparison of the results predicted the moisture content of samples with the different range of moisture, had not so difference in method II and III, but there was a significant decrease in method I. It was shown that the moisture of samples affected greatly the capacity of wood. In the case of prediction for density, there was not big changes. Sometimes, the coefficient of determination was greater, especially as employing the method II and method III. The dielectric properties of wood are anisotropic, following the structural anisotropy of wood (James 1975, James, et al. 1985). Therefore, the value of R^2_{val} of MC in 12 mm was decreased compared to other thicknesses.

In Method III, combining the capacity and NIR absorbance at two wavelengths indicated a very good performance for the density and MC. All the R^2_{val} values were above 0.8 and *RPD* was more than 2.00; these values were even higher for the 2- and 6-mm-thick samples. Compared to other methods, there were the improvement results with the better R^2_{val} and *RMSECV* values in all thickness's levels. In the case of the range of moisture under FSP, R^2_{val} values decreased slightly, but the considerate changes were seen in *RMSECV* with the maximum 5.46 % compare to 39,56 % as MC of samples from saturated point to air-dried state.

Conclusion

The moisture predictability of veneers from the green to air-dried state is higher than that from FSP to air-dried state. However, the result for estimating density is not significantly change between two range of MC sample. Generally, prediction for MC is higher than for density of veneers because capacitance sensor and NIR spectrometer based on the dielectric property of wood and chemical components directly related to the amount of water within samples. The results vary to thickness of veneers with the highest accuracy being 6 mm thickness, a little lower in 12mm and 2 mm thickness. When predicting for the whole samples of all thickness, the results decrease lightly due to the extension of thickness variable. A significant point in this study, as comparing to two techniques together, capacitance method is lower than NIR technique, however, the prominent results achieve when combining these methods together. The accuracy is improved in each kind of thickness, as well for the whole sample of all thickness. This work was to highlight the potential of the infrared spectroscopy combined with capacitance sensor as a tool capable of providing useful information for researcher and industrial company. This approach is rapid, high possibility for estimation of MC and density.

Chapter III: A new approach based on a combination of capacitance and NIR spectroscopy for estimating the moisture content of timber

Introduction

It is important to correctly measure and predict MC of timber at the sawmill in order to control and treat the material effectively. The MC influences fungal wood decay (Ammer 1963a; Ammer 1963b; Meyer and Brischke 2015; Meyer, et al. 2015; Stienen, et al. 2014a; Stienen, et al. 2014b; Thybring 2013). In the research 1, we evaluated the MC of many wood species using a combination of capacitance sensor and NIR spectrometer for thin samples with the good results. However, we don't know the predictability for the thick samples like timber. And how to develop the novel predictive models by combining capacitance data and the NIR absorbance at two optimal wavelengths. In this study, we will investigate these issues for timbers from the moisture range from the green to FSP and the range from FSP to air dried state.

Materials and methods

Sample preparation

This study will investigate wood timbers with the cubical dimensions of 100-mm cutting from the green trees of the Japanese cedar and hinoki. The capacitance and NIR spectra were measured on the cross section and tangential section of the samples with three different positions on each section. Sample weight, capacitance, and NIR absorbance were measured from the green state to the air-dried state. The data was collected from 20 samples of each species with 46 measured times until the samples reached the air-dried state approximately 12 % MC. After that, we determine the oven dried weight for calculating the MC of samples. A high-frequency capacitance device (Electrical moisture meter HM-530, Kett Electric Laboratory, Tokyo, Japan) and A portable NIR spectrophotometer (MicroNIR™ OnSite, © 2015, VIAVI Solutions Inc., USA, were used to collect the data.

Analytical processing

Capacitance-MLR: The models employed only the capacitance of wood under MLR regression

$$MC = A * C_p + B \qquad \text{(III-1)}$$

where *MC* is the predicted moisture content (%), *A* is the regression coefficient, *B* is the intercept, and *Cp* is the capacitance of wood

NIR-PLS: The models used only NIR data under partial linear regression. The models of PLS were calculated using the functions below:

$$Y = C * \boldsymbol{X} + E \qquad \text{(III-2)}$$

$$C = (\boldsymbol{X}^T\boldsymbol{X})^{-1} * \boldsymbol{X}^T * Y \qquad \text{(III-3)}$$

where **X** is the spectra matrix, *Y* is the independent variate, *C* is the coefficient constant, *E* is the intercept, $\mathbf{X}^{T}$ is the inverted matrix of **X**, and T denotes transpose

Capacitance+NIR-MLR: The models operated the capacitance and NIR absorbance at two informative wavelengths under multiple linear regression. After selecting the two optimum wavelengths, we built the predicted models using linear and nonlinear regressions and compared to choose the most suitable one.

$$\text{Func1: } MC = A_1 * Abs(\lambda_1) + A_2 * Abs(\lambda_2) + A_3 * C_p + D \qquad \text{(III-4)}$$

$$\text{Func2: } MC = A_1 * \left(Abs(\lambda_1) - Abs(\lambda_2)\right)^{A_2} * C_p + D \qquad \text{(III-5)}$$

where *Cp* is the capacitance of wood; Abs(λ_1) and Abs(λ_2) are NIR absorbance at two wavelengths; A_1, A_2, and A_3 represent regression coefficients; *D* is the intercept; *MC* is the predicted moisture content; Func1, Func2 are function 1 using multiple linear regression and function 2 using the logarithm regression for the models in Capacitance+NIR-MLR calibration. Some statistically analysis was used to compare the accuracy and performance of two functions.

1. Confidence interval for accuracy: For large test sets (*N*>30), the accuracy *acc* has a normal distribution with the mean *p* and variance *p*(1-*p*)/*N*. Confidence level 95%, $Z_{\alpha/2}$ = 1.96.

Confidence interval for *p* is:

$$P\left(Z_{\frac{\alpha}{2}} < \frac{acc-p}{\sqrt{p(1-p)/N}} < Z_{\frac{\alpha}{2}}\right) = 1 - \alpha \qquad \text{(III-6)}$$

2. We conducted an analysis of variance using the alpha =0.05 level to determine if the model performance was different between models.

Fiber saturation point (FSP) is one the importance point which affects significantly to the produce and use of wood. We analyzed the results in two ranges of MC: 1-[Green to FSP] and 2-[FSP to air dried state]. In this study, the FSP was determined with Cedar being 24.04 % and FSP of Cypress being 22.17 %.

Results and Discussions

The results of three calibrations from the green to FSP were quite good and much higher than the results getting in the MC range from FSP to air dried state if we based on the R^2_{val}. Capacitance-MLR calibration from the green to FSP demonstrated a good predictive ability on each section and on the whole sample. The highest accurate prediction MC of timber was from cross sections (R^2_{val} = 0.94), intermediate results from whole samples (R^2_{val} = 0.92), and the lowest results from the tangential sections (R^2_{val} = 0.69) while the consequences as the MC range from FSP to air dried state were much lower with R^2_{val} of 0.43; 0.45 and 0.41, respectively. The reason may be due to the range of MC, both capacitance and NIR absorbance increase by the amount of MC. Higher MC more information for prediction accuracy. On the contrary, the performance in FSP to air dried state was better if we based on the SEP value (≤ 3 %) and in the green to air dried (≥ 6 %). In this study we built the predictive models with the best ones corresponding to R^2_{val} and *RPD.*

NIR-PLS calibration yielded high accuracy results on both of two ranges of MC: From the green to FSP with R^2_{val} values being 0.82, 0.91 and 0.92, and from FSP to air dried state with R^2_{val} values being 0.76; 0.76 and 0.79.

In case of Capacitance+NIR-MLR, the calibration tangential section, whole sample, and cross section, respectively in the MC range from the saturation point to FSP. The estimation R^2_{val} values was from 0.82 to 0.96 and the performance of R^2_{val} in the range from FSP to air dried, was lower from 0.64 to 0.72, however, *SEP* values also decreased under 2%. The same with Capacitance-MLR and NIR-PLS calibrations, the results from the cross sections were better than those from the tangential sections due to the anisotropic nature of the wood. This tendency was also seen in the range of MC under FSP. Comparing to others, Capacitance+NIR-MLR shows as a better approach with the accurate predictive capability from the green to FSP.

We continued by improving the models in Capacitance+NIR-MLR calibration. Models were built from two kinds of regressions: Multiple linear regression and Logarithm regression. The details of the models for estimating the MC of wood using two equations on the different sections according to the range from the green to FSP and from FSP to air-dried state. Generally, if we choose the higher R^2_{val} and RPD with the results in [Green to FSP], if we choose the small SEP, the results in [FSP to air dried state].

Fig. III-1a illustrates the original spectrum of the wood samples on cross section, tangential section and mean values for the whole sample, while Fig. III-1b describes the NIR absorbance for the whole sample at three levels of MC and some selected wavelengths using in the predicted models. The peaks in Fig. III-1a indicated high absorption of NIR electromagnetic energy. The height and shape of the spectra are dependent on scattering of light. This scatter is affected by difference in reflectance nature of the sample surface, moisture concentration and the structures of sections. Fig. III-1a shows that the different sections had different light absorbance, the highest being for the cross section. One of the reasons is its special anatomical structure being different to the tangential section, therefore, the amount of water and OH bonding groups normally contains higher when MC was drying from the green state. Fig. III-1b illustrates the

NIR spectral curves at three levels of moisture, with some of the informative wavelengths used in the predicted models. The higher MC had the higher NIR absorbance because of the high amount of OH bonding groups. An NIR spectrum comprises many bands owing to overtone and combination modes that are usually highly overlapping, and which often designate overly low absorption and more noise. Therefore, we studied to choose the two best wavelengths with the goal of reducing the noise and improve the prediction in Capacitance+NIR-MLR calibration. Chosen wavelengths corresponding to the lowest residual were selected for the models which indicated in Fig. III-1b. The wavelengths selected in the models were different in the measured sections due to the differences in structure between the cross and tangential sections, which have differing MC gradient and light penetration depth from the surface.

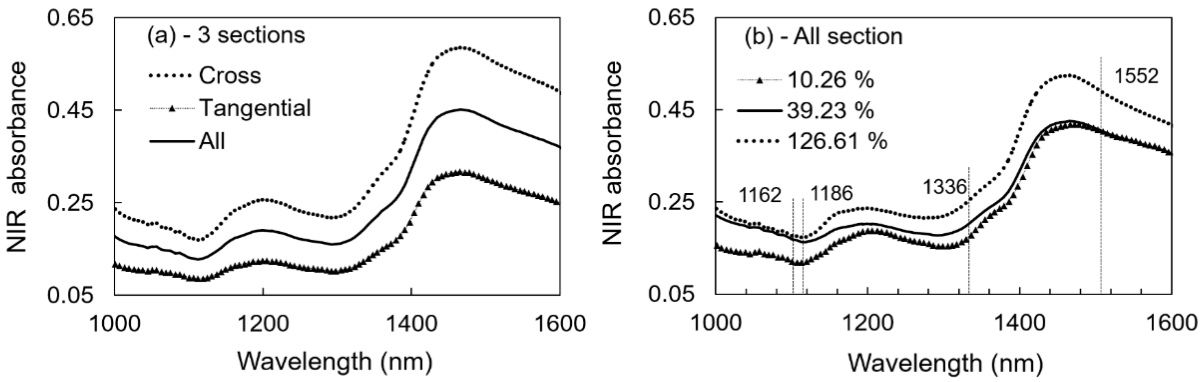

Fig. III-1 NIR spectra (a) on different sections: cross, tangential and average for the whole sample (All) and NIR absorbance on All section (b) at three levels of MC with showing some of the selected wavelengths for the predictive models for Func1 and Func2 in Capacitance+NIR-MLR calibration

Conclusion

This study demonstrates a new nondestructive approach for predicting the MC of timbers from the green to FSP and from FSP to air dried state by coordinating wood capacitance and NIR absorbance at two informative wavelengths. This study employed MLR and PLS to build predictive models. Three calibrations were implemented from the data obtained by a capacitance sensor and NIR spectrophotometer, that data being processed individually or in combination. All calibrations achieved good results in [Green to FSP], yielding a high accuracy for coefficient of determinations in cross-validation, but the results in [FSP to air dried state] had the smaller standard error of prediction. The predictions on cross-section were higher than that on the tangential section because of the anatomical characteristic of wood material. New method was studied as combining the data of capacitance and NIR absorbance at two informative wavelengths, and the predictive models were developed under two kinds of functions: Multiple linear regression and Logarithm regression. This new calibration improved the accuracy in [Green to FSP], and NIR-PLS calibration was better in [FSP to air dried state]. Depend on the MC ranges, two functions (linear and nonlinear regressions) had the different performance in [Green to FSP], and it may not be statistically analysis in [FSP to air dried state]. This research provides the basis for a new analytical method for estimating MC of timber, as well as to assess other properties of wood and wood-based materials.

Chapter IV: <u>Determining the relationship between the capacity of wood, MC and density of veneer and timber through the slope parameter between the capacitance and MC</u>

Introduction

Many methods have been made to precisely measure MC and density in wood. One of the methods is high frequency wave capacitance method which is non-destructive, rapid and high accuracy. There are many researches about the dielectric methods for the wood properties. Mobarak, et al. (1999) and Mounier, et al. (2000) studied the electrical properties of agricultural residue papers. Kabir, et al. (2007) established an equivalent circuit modeling of the dielectric properties of rubber wood at low frequency. The number of adsorbed water molecules was estimated at various relative humidity by a model of elemental cell wall (Yokoyama 2000). Zhao, et al. (1999) and Obataya, et al. (2001) analyzed the molecular mobility of absorbed water molecules in wood using dielectric analyses. The grain direction of the wood plays an important role ((Sahin and Ay 2004). In this chapter, we will focus to investigate the relationship between the capacity of wood with MC and density in many wood species from thin to thick thickness of samples. What the regression exists between them and its meanings.

Materials include veneer and timber samples in the chapter II and chapter III. The capacitance of veneers was measured on the tangential section. As each batch had a different sample thickness, the time for achieving the equilibrium air dried state differed between batches. The capacitance of timbers was measured on the cross section and tangential section of the samples. The data was collected every 4 hours from the green state to the air-dried state from 20 samples of each species with 46 measured times until the samples reached the air-dried state.

Results and discussions

For the results of veneer, the performance was very different in these two MC ranges in some kinds of thickness. In the range from the saturation point to air-dried state, the results were quite good with R^2_{val} from 0.57 to 0.79 and *RPD* from 1.52 to 2.19 and higher than the performance in the MC range from FSP to air-dried state with R^2_{val} less than 0.42 and *RPD* less than 1.31. However, the *RMSECV* of this range also was lower than the first range. The results were a little higher in 2 mm and 6 mm, decreasing in 12 mm and all thickness. Because both capacitance sensor and NIR spectrophotometry can go through out in 2 and 6mm depth, but for 12 mm thickness not completely penetrated. Generally, the prediction for density of veneers was moderate about 0.5 to 0.6 in R^2_{val}. There is not so much distinguish between two MC ranges. For the results of timbers, the performance in the MC range from the green to FSP was better than in the range under FSP with quite high in R^2_{val} (>0.8) and *RPD* around 4 except the case in tangential section. When MC of samples under FSP, the predictability reduced equal or less 0. 5 in R^2_{val}, *RPD* < 2; however, the *SEP* was also smaller than that in the range from the green.

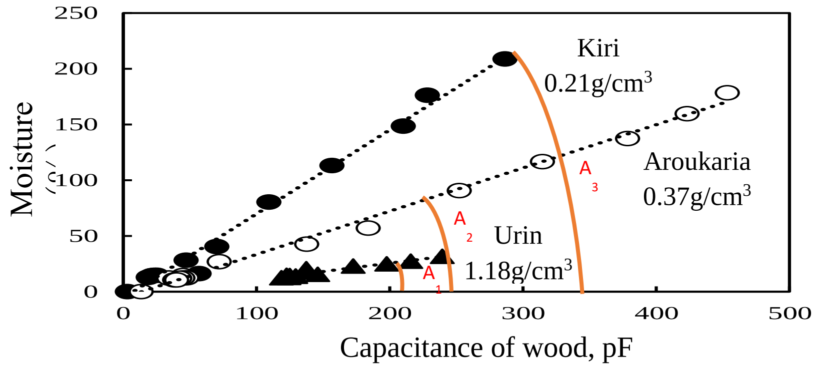

Fig. IV-1 Relationship between MC and capacity of three wood species

Fig. IV-1 represents that the relationship between the capacitance and MC of samples was the linear regression and the same tendencies in three different species. One species can calculate one slope value. However, the slope value was not the same for all wood species. Three wood species in Fig. IV-1 had the different densities corresponding to three different slopes. The slope in heavier species was smaller than that in lighter species or at the same level of MC, capacitance of heavier wood is higher than the lighter species.

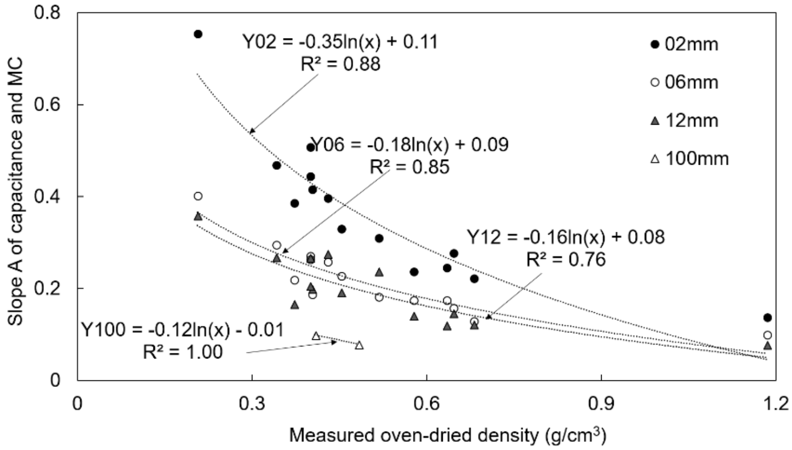

Fig. IV-2 Relationship between the slope A of capacitance and MC with oven-dried density for four kinds of thicknesses (Y2, Y6, Y12, and Y100: the equations for 2, 6, 12, and 100 mm thickness; y: the slope A of capacitance and MC; x: the measured oven-dried density;14 species for each kind of 2,6,12 mm thickness and 2 species for100 mm thickness samples.

For the data of one species of each kind of thickness, we built the relationship between capacitance of wood and MC by using the linear regression. The slopes in this linear regression is very significant to show the relation between density, thickness, and MC of the samples. We used the slope to describe the relationship between capacitance and MC for wood species; the bigger the slope A, the stronger the relation between the capacitance and MC. Each species with its density value will correspond to one slope's value. Therefore, the slope can be represented for the influence of sample's density in prediction of MC. In this study, we investigate total 14 wood species at four thickness kinds of veneers and timbers. We can establish the relationship between the slopes and oven dried density for both veneers and timbers and the best fit showed under the logarithm regression. It is clear the difference in four logarithm lines corresponding of four kinds of thickness from thin to thick. The trend of the fitted curves, the angle of curves was smaller in thicker samples. It means the thinner samples will affected to the slopes much more than the thicker samples.

Conclusion

The slopes were calculated from the relationship between capacitance and MC under the linear regression, each species with its density value will correspond to one slope's value. Therefore, the slope can be represented for the influence of sample's density in prediction of MC. Different species commensurate to different slopes and had different coefficient of determination (R^2) in predicting MC. In this study, the relationship of slopes and oven-density from the thin to thick thickness of 14 wood species indicated that the best-chosen regression was logarithm function. Thinner samples of a specific species will be affected to the slopes much more than the thicker samples. Compare to density, higher density had lower slopes. On the other hand, the higher slope showed the stronger relationship between the capacitance and MC of wood. So, the slopes can be considered as one of significant parameter to show the relationship between the capacitance, moisture and density of wood.

Chapter V: General Conclusion

Capacitance sensor and NIR spectroscopy are the nondestructive methods using popularly for prediction of MC and density of veneer and timber with a good result. The main objective of this thesis is to develop the new methodologies based on the combination between both devices to achieve some advantages as well limit some drawbacks. The performance on the cross section is higher than that on the tangential in all kinds of calibrations due to the anisotropic nature of the wood. When coordinating capacitance and NIR absorbance at two informative wavelengths achieved a better predictability compare to using devices, separately. New method with supporting of chemometric and mathematics analysis, we choose the best two informative wavelengths, so that the scattering and overlapping phenomena would be reduced and the spectral interpretation would be more efficient. The wavelengths chosen in the models were different in the measured sections due to the differences in structure between the cross and tangential sections, which have differing MC gradient and light penetration depth from the surface. Capacity of wood and MC of veneers and timbers have the strong linear regression. MC of samples is higher, capacity of wood is higher. Capacitance, MC and density of wood can be evaluated through the 'slope' parameter. Higher slope stronger relation of capacitance and MC, thinner sample higher slope, heavier species smaller slope.

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List of Publications

1. Tham VTH, Inagaki T and Tsuchikawa S (2018) A novel combined application of capacitive method and near-infrared spectroscopy for predicting the density and moisture content of solid wood. Wood Sci Technol 52(1):115-129 doi.org/10.1007/s00226-017-0974-x

2. Tham VTH, Inagaki T and Tsuchikawa S (2018) A new approach based on a combination of capacitance and near-infrared spectroscopy for estimating the moisture content of timber. *under review (Wood Sci Technol)

Oral conference

3. The 68th Annual Meeting of the JWRS (Japan Wood Research Society). March 2018. Kyoto, Japan. Coordinating capacitance sensor and portable near-infrared (NIR) spectrophotometer to evaluate moisture content of Japanese cedar and Japanese cypress timbers.

4. 2018 SWST/JWRS International convention, November 2018, Nagoya, Japan. An Improved Nondestructive Approach Based a Combination of Capacitance and Near-infrared Spectroscopy for Estimating Moisture Content of Timber.

Poster conference

1. The 67th Annual Meeting of the JWRS (Japan Wood Research Society). March 2017.Fukuoka. The prediction of density and moisture content for solid wood by combining capacitive method and near infrared (NIR) spectroscopy.

2. The 33rd NIR Forum of the JCNIRS (Japan Council for Near Infrared Spectroscopy). November 2017. Tsukuba. Coordinating capacitance sensor and a portable near infrared (NIR) spectroscopy to evaluate moisture content of wood lumber from *Cryptomeria japonica* and *Chamaecyparis obtusa* species.