

NEAR INFRARED SPECTROSCOPY (NIRS): A NON- INVASIVE TECHNIQUE FOR EVALUATING THE MECHANICS OF WOOD MATERIAL

Abstract

Wood material-based industries require simple, less time-consuming and cost-effective techniques to evaluate the properties of many wood samples. In the last couple of years, various researchers have recognized near-infrared spectroscopy (NIRS) as one of the most effective methods for assessing wood properties. The NIR spectrum wavelength range from 800 to 2500 nm (4000 to 12500 cm^{-1}) in which absorption bands correspond mainly to overtones and combinations of fundamental vibrations. The NIR spectra contain information on the functional group CH, OH, and N, which represent the backbone of all biological compounds that can be employed to determine the mechanical properties of wood material. The specific ability of NIRS to measure the modulus of elasticity (MOE) and modulus of rupture (MOR) is mainly attributed to the absorbance of light by lignin and cellulose at a specific wavelength of spectra. In order to assessment of wood mechanics, the degree of overlap and band complexity makes NIR spectra almost impossible to interpret without the help of a chemo-metric approach. The ability to handle the NIRS data from various wavelengths, the chemo-metric techniques, and multivariate statistical methods coupled with NIRS can be enabled spectral interpretation for properties of interest. Without chemo-metric and multivariate methods, NIRS applications would not be possible. This

Author

Dr. Aasheesh Raturi
Department of Physics
Dolphin PG Institute of Biomedical and
Natural Sciences
Dehradun, Uttarakhand, India.

chapter describes the application of NIR Spectroscopy for analysis of mechanical properties of wood. Other multivariate statistical parameters of model evaluation are explained. The main purpose of this chapter is to underline the need of combining chemo metric methods with NIRS technique to develop the most accurate prediction model for assessing wood properties with special reference to wood mechanics.

Key words: Wood Mechanics, NIRS, Chemo-metrics.

I. INTRODUCTION

Wood is a very important natural renewable resource; which is mostly derived from our age-less or everlasting forests. It is predominantly composed of cellulose, lignin, hemicelluloses, and minor amounts of extraneous materials (Izzekor and Fuwape 2010). The wood material can be heavy or light, stiff or flexible, and hard or soft depending on variations in the properties and volume of these components as well as variances in cellular structure. It's a common occurrence in daily life and is employed in the production of wood-frame homes, furniture, railroad ties, fence posts, utility poles, fuel wood, textile fabrics, paper, and organic chemicals.

Unlike other materials, it is an orthotropic and anisotropic material. It has different and independent mechanical properties in the direction of three mutually perpendicular axes because of the orientation of the wood fibers and the manner in which a tree increases in diameter as it grows (Amtzen and Charles 1994, Bergman et al. 2010). It can alternatively be described as a natural composite material made of hemicelluloses and lignin, with cellulose fibers serving as the reinforcing components. Mechanical properties of wood material vary along three mutually perpendicular axes: radial, tangential, longitudinal. The radial axis is perpendicular to the grain direction and normal to the growth rings, the tangential axis is perpendicular to the grain direction and tangent to the growth rings whereas the longitudinal axis is parallel to the direction of the fiber (or grain).

Although most wood properties vary along each of these three axes, variations along the radial and tangential axes are rather insignificant compared to variations along the radial or tangential axis and the longitudinal axis (Amtzen and Charles 1994). Additionally, it has a remarkable strength-to-weight ratio, and desirable building material with a sustainable utility.

The use of wood material for various end uses requires knowledge of the mechanical properties and the uniqueness that wood has in comparison with other materials used for structural purposes such as steel, iron concrete, bricks, and stone. It is important to inspect the Mechanical properties of wood material with the highest accuracy. Non-destructive measurement is strongly required for fast estimation of a large number of wood samples. Previous researchers have proposed and developed various Non-destructive techniques (NDT) techniques to predict the wood properties Mora and Schimleck 2010, Kothiyal and Raturi 2011, Raturi et.al. (2012, 2014 & 2017). As we know the rapid growth of technologies like artificial intelligence and its application in wood science has become a new chemometric method for NIR spectral analysis. The subject of machine learning is also gradually implementing in NIR spectroscopic techniques and has become the most preferred research methodology among the some groups of researchers (Lee et. al. 2020, Mishra et.al. 2022, and Wang et.al. 2022) in the field of wood science and technology.

In this chapter, the outline of NIR spectroscopy as a noninvasive technique introduced to wood material science, which might be helpful to consider NIRS applicability to predict mechanical properties, more specifically, modulus of elasticity (MOE), and modulus of rupture (MOR) of wood samples. The subsequent sections of the chapter provide details of the NIRS research work done on the subject in the field of wood material science.

II. MECHANICAL PROPERTIES OF WOOD MATERIAL

The characteristics of a material in reaction to external forces are known as its mechanical properties. According to Amtzen and Charles (1994), an external force is any force that originates from outside of a specific piece of material and has a tendency to cause the material to physically deform. Wood's mechanical strength depends on the three polymers (cellulose, hemicellulose, and lignin) that make up the material (Kollmann and Cote 1968). The mechanical properties of the three polymers, which each significantly contributes to wood strength, are very distinct from one another.

When determining the mechanical properties of wood material, consideration is given to its stiffness or elasticity, tensile strength, compressive or crushing strength, shear strength, toughness, hardness, cleavability, and resilience. The mechanical properties of wood, such as its modulus of elasticity (MOE) and modulus of rupture (MOR), play a significant role in the performance of solid wood materials and have a significant impact on their end-use potential, which is mostly determined by variations in the structural properties of wood..

III. NON-INVASIVE ESTIMATION OF WOOD PROPERTIES

Evaluation of the mechanical properties of wood is obtained mainly through destructive methods of experimentation as per relevant standards (Pellerin and Ross 2002). In India, most of the testing is done as per Indian slandered which are mostly derived from British and ASTM standards. Different countries have different standards (Rajput et al. 1991). However researchers around the world are working on indirect methods to evaluate timber properties (Tanasoiu et al. 2002, Schimleck 2008, Mora and Schimleck 2009).

Non-invasive or nondestructive testing (NDT) is a method of testing that can assess mechanical properties and detect defects in a piece of wood specimen without affecting its end-use capabilities and then using this information to make decisions regarding appropriate applications of wood material (Pellerin and Ross 2002, Tanasoiu et al. 2002). A number of NDT techniques, such as ultrasonic stress wave, deflection method, electrical method, gamma radiation method or isotope method, penetration radar method, and X-ray method used in the forest products industry, can be used to monitor and assess the quality and properties of wood and wood structures (Pellerin and Ross 2002; Tanasoiu et al. 2002).

Near-infrared (NIR) technology has received considerable attention for its use in quality control and assessing wood quality and the technical review of the technology and its potential applications in wood products is summarized by So et al.(2004). The distinctive advantages of NIR over other NDT techniques are its ability to do simultaneous analysis of many parameters (Schimleck et al. 2006b, Schimleck et al. 2007, Schimleck 2008, Mora and Schimleck 2009). The other distinctive feature is its applicability to online monitoring in the industry. In fields, the portable NIR instrument and lab-based analysis make it an important tool. Easy calibration method transfer from one machine to another machine makes it a versatile tool (Meder at el. 2003, Brashaw at el. 2009).

IV. SPECTROSCOPIC BACKGROUND OF NEAR-INFRA RED (NIR) TECHNIQUE

Near Infrared Spectroscopy (NIR) has a long history that dates back to Herschel in the 1800 century. He found the light radiation beyond the visible spectrum (Barton 2002). The expansion of NIR spectroscopy has begun after 1950s from the agriculture realm into pharmaceutical, industrial processing, and imaging spectroscopy.

The NIR region with wavelength range from 800nm to 2500 nm (4000 to 12500 cm⁻¹) in which absorption bands correspond mainly to overtones and combinations of fundamental vibrations (Siesler 2006). NIR technology, which uses electromagnetic waves with transmitted or reflected spectra between 800 and 2500 nm (4000 and 12500 cm⁻¹), has primarily been utilised for nondestructive testing of organic materials including foods and agricultural products. However, it exhibits excellent potential in all areas of material assessment (for instance, polymer, textile, pharmacy, petrochemical, etc.).

V. FT-NIR SPECTROPHOTOMETER

The most commonly used NIR spectrometer in the field of wood science is FT-NIR spectrophotometer of Bruker Optics, MPA with wave number between 4,000 and 12,820 cm⁻¹ (780 to 2,500 nm), working in the diffuse reflectance mode and a resolution of 8 cm⁻¹ using zero filling of factor 2. Two sampling accessories, i.e., fiber optic module and integrating sphere. Integrating sphere with RT-Pbs (external, NEP 5 9 10-10 WHZ-1/2) detector setting permits direct measurement of large sample areas (15 mm spot size) of wood samples, while the fiber optic module having TE-InGaAs (internal, NEP 2 9 10-13 WHZ-1/2) detector setting allows the measurement of a small sample area (10 mm spot size). Both the accessories work on the principal of diffuse reflectance with the integrating sphere having wider collection geometry than the later (optical probe). The optical probe has flexibility in terms of remote analysis and is suitable for standing trees and logs and sawn timber. The integrating sphere is useful for lab analysis although it covers more sample area.

VI. NIR SPECTROSCOPIC ASSESSMENT OF MECHANICAL PROPERTIES OF WOOD MATERIAL

Generally, the mechanical property is estimated through destructive experimentation with the help of mechanical testing machines in the laboratory. Hoffmeyer and Pedersen (1995) used near-infrared spectroscopy for the first time to offer a technology for non-destructive mechanical testing of wood that is completely distinct from standard methods (Tsuchikawa 2007). Several researchers have reported that the NIR spectroscopy can be used as nondestructive techniques for determining mechanical properties of solid wood (Kelley et al. 2004a, 2004b, Tsuchikawa et al. 2005, Fujimoto et al. 2007, 2008, Schimleck et al. 2007, Mora et al. 2008, Kothiyal and Raturi 2011).

The use of Near Infrared Spectroscopy (NIR) as non-invasive tool to evaluate specific mechanical properties, such as the modulus of elasticity (MOE), and modulus of rupture (MOR) in bending tests have been also demonstrated by a number of researchers (Mora et al. 2008, Schimleck, 2008, Schimleck et al. 2009, Hein et al. 2009, Mora and Schimleck 2010, Kothiyal and Raturi 2011).

Further, in the continuation of mechanical properties estimation by NIR spectroscopy, Kothiyal and Raturi (2011) tried to short out the problem, regarding the limitation of NIR spectroscopy application with variation in moisture content of wood in timber yards. They obtained NIR models for predicting MOR, and MOE. According to McClellan et al. (1991), lignin and cellulose absorb light at particular wavelengths, which allows NIR spectra to calculate the modulus of elasticity (MOE) and modulus of rupture (MOR) of materials.

VII. CHEMO-METRICS AND MULTIVARIATE MODEL DEVELOPMENT USING NIR TECHNIQUE

Chemo-metrics is a method of measurement and analysis based on the concept of indirect observation. The value of property of interest is deduced from measurements of a substance's chemical composition using a mathematical relationship (Lavine 1998). It aids NIR devices in resolving large, heavily overlapped peaks with high sensitivity to sample physical properties. In order to obtain the most pertinent information from the analytical data, Chemo-metrics must be used for NIR spectroscopic model development (Massart et al. 1988).

Through the use of various multivariate analysis techniques, it is possible to relate different analytical variables (such as those found in an NIR spectrum) to measure properties and extract the analytical information present in NIR spectra. With the help of the most appropriate multivariate techniques, samples with similar traits can be grouped together in order to create classifications and identify some attributes of unidentified samples for qualitative and quantitative analysis, respectively (Blanco and Villarroya 2002).

Without chemo-metric and multivariate methods, NIRS applications would not be possible. The chemo-metric technique covered the calibration by multiple linear regression (MLR), choosing a training set, spectral pre-treatments, tuning and validation, principal component analysis (PCA), quantitative calibration using partial least squares (PLS), principal component regression (PCR), qualitative calibration using discriminate analysis and regression methods for developing the calibration models as their use in the modeling process, those are included in all chemo-metric software packages.

Numerous multivariate analysis techniques can be categorised based on their objectives and computing approaches. The chosen approach depends on the analysis's objectives, the specific characteristics of the samples, and the degree of complexity of the system under evaluation (such as its nonlinearity). Once models have been developed, it is necessary to test their prediction ability using independent sets of samples that experienced the same spectral pretreatments and spectrum recording conditions as those used for calibration but were not included in the calibration regression model. The most frequently used multivariate-regression techniques in NIR spectroscopy, such as partial least squares (PLS), principal component regression (PCR), and multiple linear regression (MLR), are the best techniques for calibrating the relationship between the properties to be measured and the absorbance of NIR spectra (Agelet and Hurburgh, 2010).

Both principal component regression (PCR) and partial least-squares (PLS) regression can be applied to the entire spectrum or to specific spectral regions, and they both enable the calibration model to incorporate additional information. While PLS determines the directions of greatest variability by taking into account both spectral and target-property

information, the new axes are referred to as PLS components or PLS factors, and PCR uses the principal components obtained by PCA to perform regression on the specimen property to be predicted (Martens and Naes 1991). Multiple linear regression (MLR) combines a number of spectral regions, or X-variables, in linear combinations that have a relationship as closely related to a single response (material property), or Y-vector, as possible.

The most prominent models in the field of wood material research are PLS and PCR, and both are capable of resolving significant multi-co linearity issues (Via et al. 2003). Before developing the NIR calibration model, mathematical pre-treatment of the NIR spectra is essential to enhancing signal quality and reducing noise.

Further, The two categories of pre-processing methods that are widely used are spectral derivatives and scatter-correction approaches. Multiplicative Scatter Correction (MSC), Standard Normal Variate (SNV), and Normalisation are the three fundamental preprocessing ideas used in the scatter-corrective preprocessing method. The first derivative and the second derivative are two fundamental preprocessing ideas included in the spectral derivative. Broad calibration sets may be required for heterogeneous material (like wood) than for homogeneous material. In order to account for the widest possible diversity in the calibration set, several researchers have collected more than 100 wood samples (Fujimoto et al. 2008, Hein et al. 2009, 2010).

For each regression, calibration and validation statistics include coefficient of determination (R^2), root mean standard error of calibration (RMSEC), root mean standard error of cross-validation (RMSECV), and root mean standard error of prediction (RMSEP). The ratio of performance of deviation or relative predictive determinant (RPD), and range error ratio (RER), are the two final statistics to be discussed.

The coefficient of determination R^2 can be utilized in relation to statistical models whose primary objective is the forecasting of future results based on additional pertinent information. According to Steel and Torrie (1960), this is the proportion of a data set's variability that the statistical model can explain. It gives an indication of how precisely the mathematical model is likely to anticipate future outcomes.

The root mean squared difference between the forecasts and the reference data is used for calculating the RMSEP value. It is essential to stress that this approach only works when the reference value noise is insignificant compared to the actual forecast uncertainty. Prediction errors are defined with respect to true quantities rather than noisy reference values. An absence of noisy reference values is the ideal condition for the perfect model. Of course, this limit is impractical, however (Difoggio 1995, Coates 2002) demonstrated how adding noise to the reference values can always get close to the ideal model. It should be obvious that the forecasts are accurate, and the measurement error in the reference values is the only factor that might contribute to RMSEP. In this extreme scenario, RMSEP would merely estimate the standard deviation (square root of the variance) of the measurement error of the reference value. It wouldn't be significant to the real prediction uncertainty.

The description of RMSECV is the same as RMSEP. The difference between statistical values of these parameters is used as a method for figuring out the best number of independent variables to use in building a calibration equation and based on a repetitive

algorithm that selects samples from a sample set population to develop predictive model and then predicts on the remaining unselected samples (Difoggio 1995, Coates 2002).

The ratio of performance to deviation (RPD: ratio of the SD of the reference results to RMSEP) is also an appropriate statistical measurement of the ability of a NIR spectroscopy model to predict wood properties. If RMSEP is not presented compared to the SD of the original reference data, it may be deceptive. The calibration is essentially predicting the population mean if $RMSEP = SD$ (Bailleres et al. 2002). Prior to the introduction of RPD, RER was used in NIR. The range of the reference values in the validation set is used for statistics, rather than standard deviation, making it comparable to RPD. Additionally, it lacks dimensions and is not dependent on any particular application (Fearn, 2002).

These are related to the capability of the model to predict future data with respect to the initial variability of the calibration data. Davies and Fearn (2006) reported the definition and application of all these terms. In the absence of a sufficient number of samples where a separate set of samples for prediction are not available other criteria are generally used (Derkyi et al. 2011) to assess the quality of a model including the root mean squares error of estimation (RMSEE), root mean square error of cross-validation (RMSECV) and the correlation coefficient of determination (R^2). An effective model should have a modest difference between RMSEC and RMSECV, low RMSEE, low RMSECV, and high R^2 between the predicted and observed values. For quantitative prediction, the correlation coefficient of determination (R^2), which is the most popular, should be higher than 0.8. The RMSEE to SD ratio should be less than 0.2 for superior models, where SD is the standard deviation of the reference values. The SD to RMSECV ratio should be > 2 , RMSECV to RMSEE < 1.2 , and the SD to RMSECV ratio should be > 2.5 . RMSECV can be utilized to determine how effectively an equation predicts samples that weren't used to develop the calibration equation when there aren't enough external validation samples.

Numerous researchers employed various pre-processing and statistical techniques to create superior calibration and validation models for mechanical properties of wood materials. Thumm and Meder (2001) demonstrated the use of spectra pretreatment, using first and second derivatives, and achieved a prediction coefficient of determination (R^2) of 0.55 to 0.72 for stiffness in the bending test. Schimleck et al. (2001a) also used the first and second derivatives to help develop PLS models that provided prediction R^2 over 0.80 for both MOR and MOE. Modulus of elasticity (MOE), modulus of rupture (MOR) wood species have been accurately predicted by the number of researchers (Hoffmeyer and Pederson 1995, Gindl et al. 2001, Schimleck 2001a, Thumm and Meder 2001, Kelley et al. 2004a, 2004b, Tsuchikawa et al. 2005, Fujimoto et al. 2008, Viana et al. 2009, Kothiyal and Raturi 2011). They used NIR and various statistical techniques (PLS, MLR, and PCR) and also found that the calibration model of green wood samples can be improved by using spectral pre-processing techniques. Figure 1. illustrates the flow diagram of NIR model development and model evaluation for the mechanical properties of wood. Despite recent research demonstrating NIR's potential as a non-destructive measure of wood strength, the wood based industry has not yet adopted it. The high cost of advanced NIR spectrophotometers may be the cause of this issue. However, NIR spectrophotometers with reduced spectral range are more affordable, have also been able to precisely predict MOE and MOR. Acceptable findings were obtained with reduced ranges between 400nm and 1100 nm (Kelley et al. 2004a). Further research involving NIR spectroscopy and chemo-metric

model development has produced best results and the strategy is promising for adaptation of spectral techniques to the production environment of wood based industries (Thumm and Meder 2001). The introduction of NIRS in the field of wood material science is a welcome step as more and more materials will be required for evaluation at low cost, high speed, and with improved accuracy in a non-invasive manner.

VIII. CONCLUSION

The introduction of NIRS in the field of wood material science is a welcome step as more and more materials will be required for evaluation at low cost, high speed, and with improved accuracy in a non-invasive manner. In light of various studies, it has been concluded that the chemical constituents of wood material are well-associated with the NIR wavelength. The chemical constituents that contribute to the prediction of chemical and physical properties of wood are also responsible for MOE and MOR. Further, the link between chemical components and mechanical properties cannot be fully comprehended. However, the NIR absorption bands related to lignin and cellulose are certainly the main contributors to developing robust models for the analysis of wood mechanics. The implementation of real-time monitoring of wood properties in the wood industry is the next step. It should be a valuable and effective analytical spectroscopic method for assessing wood properties. However, the precision of the spectrum acquisition and reference values (MOE and MOR) measurements determines the robustness of the prediction models.

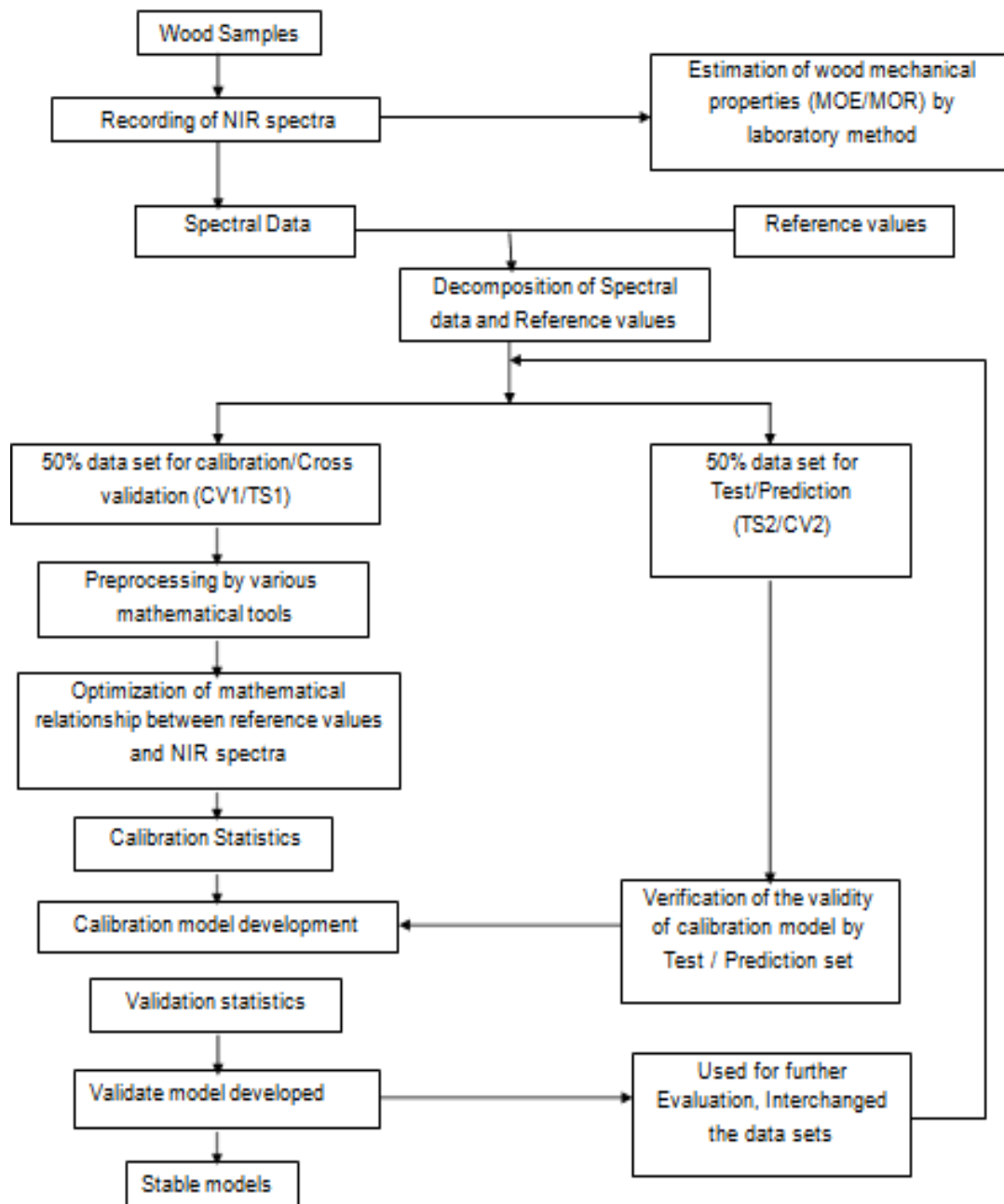


Figure 1: Flow diagram of NIR development and models Development

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