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Estimation of Bending Stress in Earlywood and Latewood Growth Rings of Oil Thermally Treated Wood by Near Infrared Spectroscopy

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Keywords

Bending Stress, Eastern Hemlock (*Tsuga Canadensis* L.), near-infrared (NIR) spectroscopy, oil-treated Wood, Relative Percent Difference (RPD), Sample Specific Standard of Prediction, Soft Maple (*Acer rubrum* L.), White Spruce (*Picea glauca*)

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Abstract

This paper focuses on the estimation of bending strengths in earlywood and latewood growth rings of three refractory wood species such as white spruce (*Picea glauca*), eastern hemlock (*Tsuga canadensis* L.) and soft maple (*Acer rubrum*) wood samples by near infrared (NIR) spectroscopy. Wood samples were heat thermally treated in the deep fryer with vegetable oil at 220°C during 120 minutes. For earlywood zones, calibration R^2 achieved 0.44 and 0.63 in Spruce and Hemlock, respectively. The root mean square error (RMSE) ranging from 9.25 to 12.90MPa for all the species and relative percent difference (RPD) ranging from 1.0 to 1.6 in Spruce, Hemlock and Maple. For latewood zones, validation statistics R^2 achieved 0.27 and 0.32 in Spruce and Hemlock, respectively. RMSE ranging from 10.177 to 18.27MPa for all the three wood species, and RPD ranging from 1.2 to 1.5 for all the species. The NIR prediction results confirm that chemical reactions in wood sites resulting from the heat treatment account for the maximum amount of flexure stress that were related to main peaks of spectra data using 1100-2200nm region of the three wood species, while the oil absorbed by wood reduces the sensitivity of NIR reflectance.

1. Introduction

Earlywood (Ew) and latewood (Lw) tracheids are principal components of wood structure that involved mechanical properties of all wood fibers in the basic cell morphology (Mott et al. 2002; Via et al. 2003 and 2005; Carneiro et al. 2010). Mechanical properties of individual fibers are known to be reduced proportionally with the hygroscopicity after a thermal treatment of wood, however, many other properties of the wood enhances significantly as for example the resistance against non-biotic agents (UV, Oxidation…) and biotic agents of wood-degradation (biological attack…) (Tjeerdsma et al. 1998; Sailer et al. 2000; Lyon et al. 2007; Gérardin et al. 2008; Mounanga 2008; Salman et al. 2014). Among the existing thermal modification of wood, the oil-thermally treatment has one of the best heat transfer media as well as a good potential carriers for other substances (Jamsa and Viitaniemi, 2001; Militz and

Tjeerdsma, 2001; Poaty et al. 2010). In order to further enhance the benefits of this treatment, we focus on the thermal treatments using one type of hot oil in this project, with a variance of the original idea from oil-heat treatment in Germany (OHT-Process)(Shin and Lee, 2000; Rapp and Sailer, 2001; Militz 2002). Thermally treated wood is also used to defects in service that is especially attractive for refractory wood species like spruce (Picea) and fir (Abies) which are difficult to penetrate with wood preservatives or chemical modification agents (Hein et al. 2011; Wang and Copper, 2005; Mohebby et al. 2014; Mounanga 2015). A good understanding of relationship between mechanical properties and radiance absorption of wood-oil-thermally treated is comparatively new concept including while the influence of chemical reactions in the wood sites with regard to contribution of defects to variation in fiber properties, which are not well documented and less understood (Hoffmeyer and Pedersen, 1995; Mott et al. 1996; Kohan et al. 2012). The potential of near infrared spectroscopy (NIRS) as a method to rapid non-destructive analysis and to predict wood chemical composition based on the spectral absorption properties of chemical components on the wood surface is also of interest (Bailleres et al. 2002; Watanabe et al. 2011; Mounanga et al. 2008; Leblon et al. 2013). NIRS has been used to predict and estimate wood physical and mechanical properties, moisture content, surface colour, contact angle, adhesive bond strengths in loblolly pine (*Pinus taeda*) wood products, trembling aspen (*Populus tremuloides* Michx.) stands, Eastern black spruce (*Picea mariana* var. *mariana*) wood products, European larch and radiate pine (*Pinus radiate*) wood products (Meder et al. 2002; Gindl et al. 2004; Kelley et al. 2004; Tsuchikawa et al. 2005, Koumbi-Mounanga et al. 2015b,c).

This study provides informations on the prediction of bending strengths of oil thermal treatment of white spruce (*Picea glauca*), eastern hemlock (*Tsuga canadensis* L.) and soft maple (*Acer rubrum*) wood samples by near-infrared (NIR) spectroscopy. We related the NIR reflectance spectra date collect from earlywood and latewood growth rings of three wood species to multivariable analysis through partial least square (PLS) regression method and sample specific standard error of prediction, which may serve to prevent and improve the durability of wood thermal treatments for suitable indoor and outdoor applications.

2. Materials and Methods

2.1. Sample Preparation for Oil-Heat Treatment

Wood samples used in this investigation were white spruce (*Picea glauca*), eastern hemlock (*Tsuga canadensis* L.) and soft maple (*Acer rubrum*) wood samples, acquired green from a local lumbers supplier and then cut in true radial/tangential orientation with dimensions of 25mm (radial) x 25 mm (tangential) x 10mm (longitudinal), which were a relative humidity (RH) > 2% prior to heat treatment in commercial soy oil (from Brunge Canada) at 220°C for 2h, in an oil bath (Fisher HiTemp Bath Model 160). Some wood samples were left as untreated control samples and the entire sample sets (treated and untreated) were kept as conditioned in a chamber at 103°C for 24h, and then at 26°C for the duration of the experiment in order to remain around their equilibrium moisture content with constant weights as described in Stamm (1969).

2.2. Mechanical Testing

The bending tests were conducted on the wood samples using a Zwick-Roell load frame equipped with 10 kN load cell and computer-controlled screwdriver crosshead over three-point setup with a loading rate of 4.0mm/min, and support separation of 75 mm in EW/LW spots as described by Biblis (1969).

2.3. Spectral Measurements

NIR spectral data were manually acquired from each wood wafers (RH=0-2%) with an *Ocean optics Inc. Labspec® 256-HL-2000 NIR* spectrophotometer (*Ocean optics Inc., 830 Douglas Ave, Dunedin, FL34698 USA*) equipped with an optical probe positioned on the top of the sample in 2 mm diameter beam. The instrument has a spectral resolution of 2 nm and was calibrated manually for white/dark after every triplicate measurement. Each wood sample was scanned both faces three times at successively various EW/LW zones on a flat surface (bark-side up and bark-side down) as described in Mounanga et al (2012).

2.4. Data Processing

The data processing was done using the *Unscrambler® 9.8* (*CAMO software. Inc., 1 North Cir., Woodbrige, NJ 07095-2105, USA*). All the reflectance spectra acquired over the whole wavelength range (1100-2400nm) were smoothed by applying a second derivative 13pt Savitzky-Golay transformation and then were related to bending strengths by the partition to latent structures-PLS regression method and sample specific standard error of prediction (Geladi and Kowalski, 1986; Esbensen et al. 2002; Faber et al. 2003).

3. Results and Discussion

3.1. NIR Spectra

Figure 1 shows the visible-NIR reflectance spectral comparison in earlywood and latewood tissues of oil-thermally wood and control samples for white spruce (*Picea glauca*), eastern hemlock (*Tsuga canadensis* L.) and soft maple (*Acer rubrum*). The global trend could be interpreted as a decreasing of NIR reflectance of Spruce, Hemlock and Maple, whereas, earlywood/latewood untreated and treated sample zones were differently affected. The main areas of absorption were clearly identified the fundamental (first, second and third overtones) stretching absorptions and the combination bands of vibrational transitions in infrared region, mainly C-H (hydrocarbons…), C=O (cellulose, hemicelluloses…); O-H (water, alcohol, phenol…) and N-H (lignin…) functional groups (Oye and Okayama, 1989; Cozzolino and Murray, 2003; González-Martín et al. 2003; Schwanninger et al. 2011). These series of overtones were generated by the hot treatment, which reduced the reflectance gradually from 95 to 55%. The heights of the main peaks decreased with oil absorption resulted from heat treatment and cooling duration for all the species as was described for measuring (earlywood/latewood) contact angles of Douglas-fir (*Pseudotsuga menziesii* var. *menziesii*) and trembling aspen (*Populus tremuloides* Michx.) veneers by Koumbi-Mounanga et al (2013, 2015a). The effect on NIR reflectance spectra was more evident by applying a second derivative Savitzky-Golay transformation represented in Figure 2. The overlapping overtones of transformed NIR spectral were also generated by the hot treatment that revealed some singular areas of absorption, mainly at 1320-1350nm and 1720-1750nm wavelength regions for spruce; 1330-1360nm and 1620-1650nm region for hemlock, and 1520-1550nm and 1625-1655nm regions for maple; thus differenced earlywood/latewood untreated to treated as well as identified one specie to each other. The decreased absorption previously mentioned might be induced by the degradation of the wood chemical components such as lignin and hemicelluloses including carbohydrates and deacetylation reactions of polyoses. In fact, the wood had reached over 200°C during the hot treatment; above this temperature is conditioned to change the wood properties with regard to the degradation of its components (Beall and Eickner, 1970; Lee and Luner, 1972; Mitsui et al. 2008; Sidorova 2008; Bachle et al. 2010).

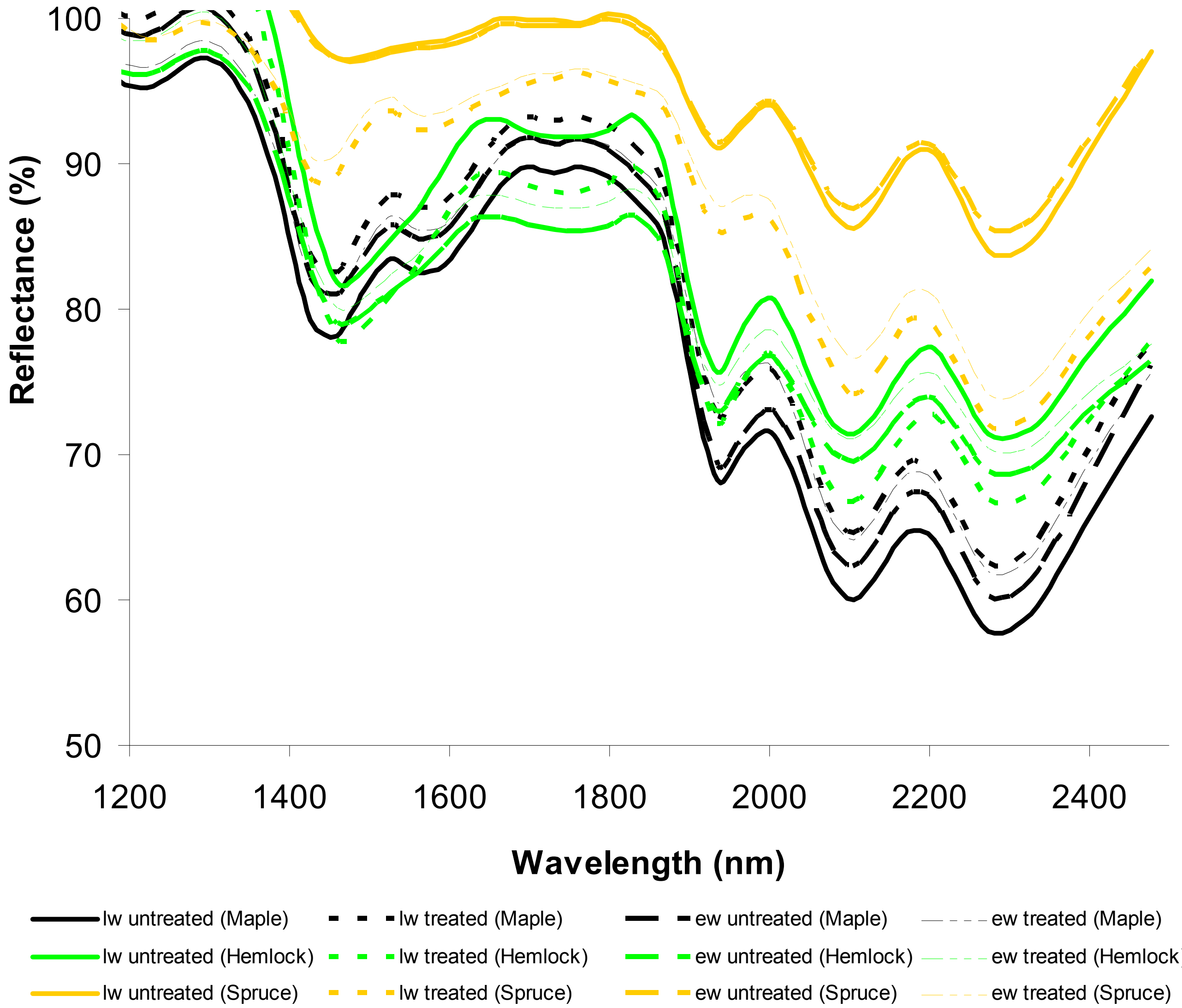


Figure 1. *visible-NIR spectra for white spruce (Picea glauca), eastern hemlock (Tsuga canadensis L.) and soft maple (Acer rubrum) wood samples in the earlywood (EW)/latewood (LW), which were oil thermally treated/untreated. Each spectrum represents an average of 6 spectra.*

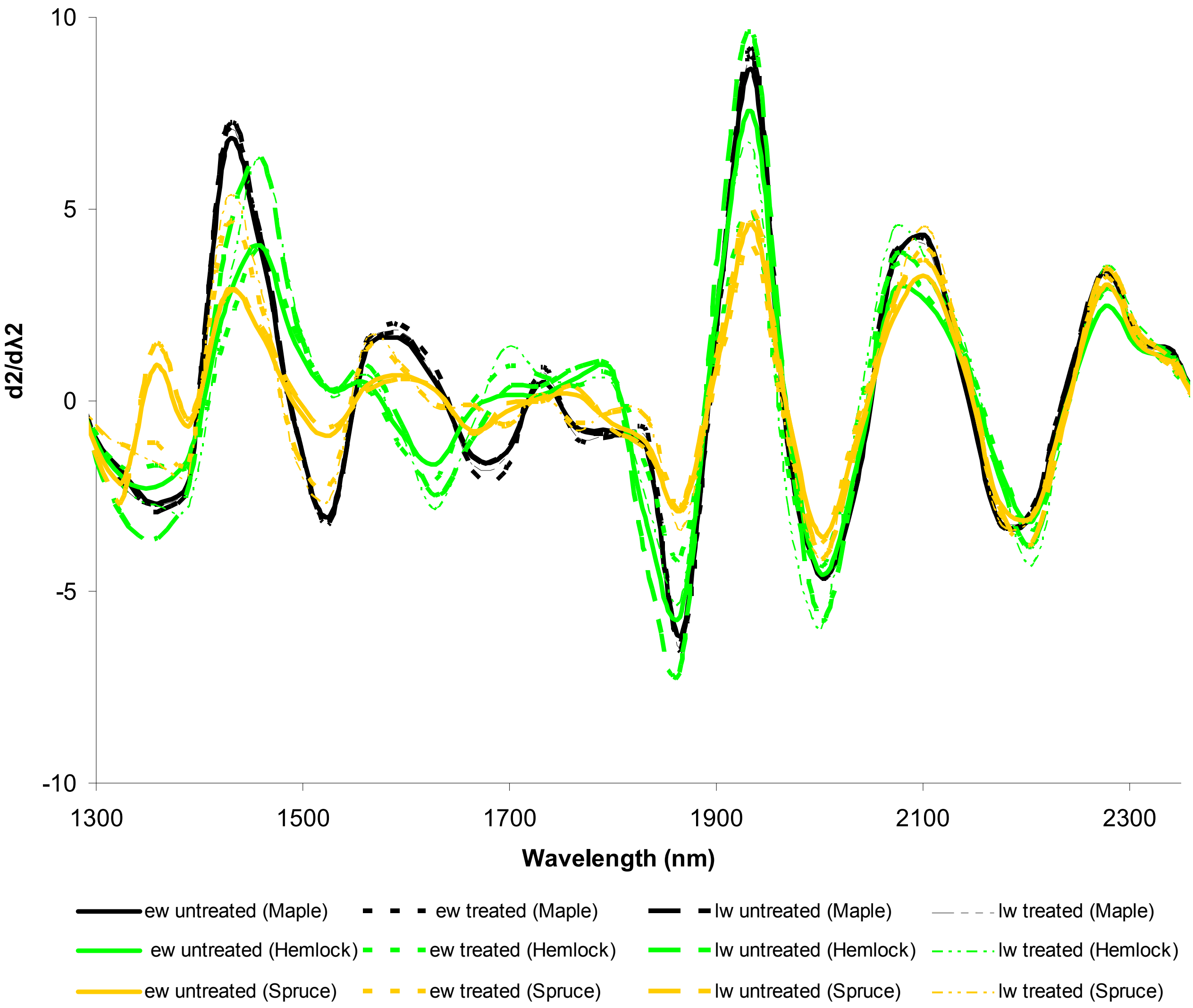


Figure 2. *NIR spectra after 2nd derivative transformation ($d^2A/d\lambda^2$) of white spruce (Picea glauca), eastern hemlock (Tsuga canadensis L.) and soft maple (Acer rubrum) wood samples in the earlywood (EW)/latewood (LW), which were oil thermally treated/untreated. Each spectrum represents an average of 6 spectra.*

3.2. PLS Regressions and Bending Strengths

Figure 3 shows the bending stress estimation to earlywood/latewood zones of Spruce, Hemlock and Maple. Regardless of the impossibility to isolate and test separately in bending strengths (BS) of earlywood (EW) and latewood (LW) from those individual species, especially for Spruce latewood, the scans conducted at different LW/EW zones were related to average BS values from the wood wafers at the EW/LW zones as represented in some data which might suggest that hypothesis. This should also explain the fact that the latewood BS values were not much higher than for earlywood as expected.

The thermal treatment affect equitably the wood tissues by reducing all bending strengths measured in earlywood/latewood when comparing the treated and untreated wood samples of all the three species.

Although oil-treated wood samples are not advised for mechanical properties usage, it was possible to make some general observations with the combined (treated/untreated) and earlywood/latewood tissues of three wood species (Gindl et al. 2001; Via et al. 2009).

Figure 4 presents the PLS regression models for earlywood (EW) zones of Spruce, Hemlock and Maple, that were built using the averaged (6 spectra) and normalized (1 to 100%) reflectance data acquired in the 1100-2400nm region. The statistics are shown in Table 1. In the same context, Figure 5 shows the PLS models for latewood (LW) zones of the three wood species. The related statistics are shown in Table 2. The regression models built in EW were quite different than those conducted in LW. All the PLS models in EW were able to provide good correlation (r) ranging from 0.42 to 0.79 between the measured and predicted bending stress for validation and calibration. Except the correlation on Maple were not significant r=-0.009 (p-value>0.9977) for validation (Figure 5). The relative percent difference ranging from 1.0 to 1.6 for all the wood species. Only the prediction performed in calibration (EW/LW treated/untreated) for eastern hemlock (*Tsuga canadensis* L.) wood samples were very statistically significant (p-value> 0.0022 and 0.0083 in EW/LW, respectively). Kohan et al (2012) found significant results on the prediction of bending strengths of wood strands with R^2 ranging from 0.35 to 0.76, and SE ranging from 0.62 to 13.9MPa. They related that fact to the flexure properties from differences in anisotropic and non-homogenous of wood tracheids within juvenile aspect. This was defined closely the weakest zones of the wood thermally treated in reducing mechanical properties of the entire wood material (Salim et al. 2010; Thumm and Meder, 2011; Hein and Brancheriau, 2011).

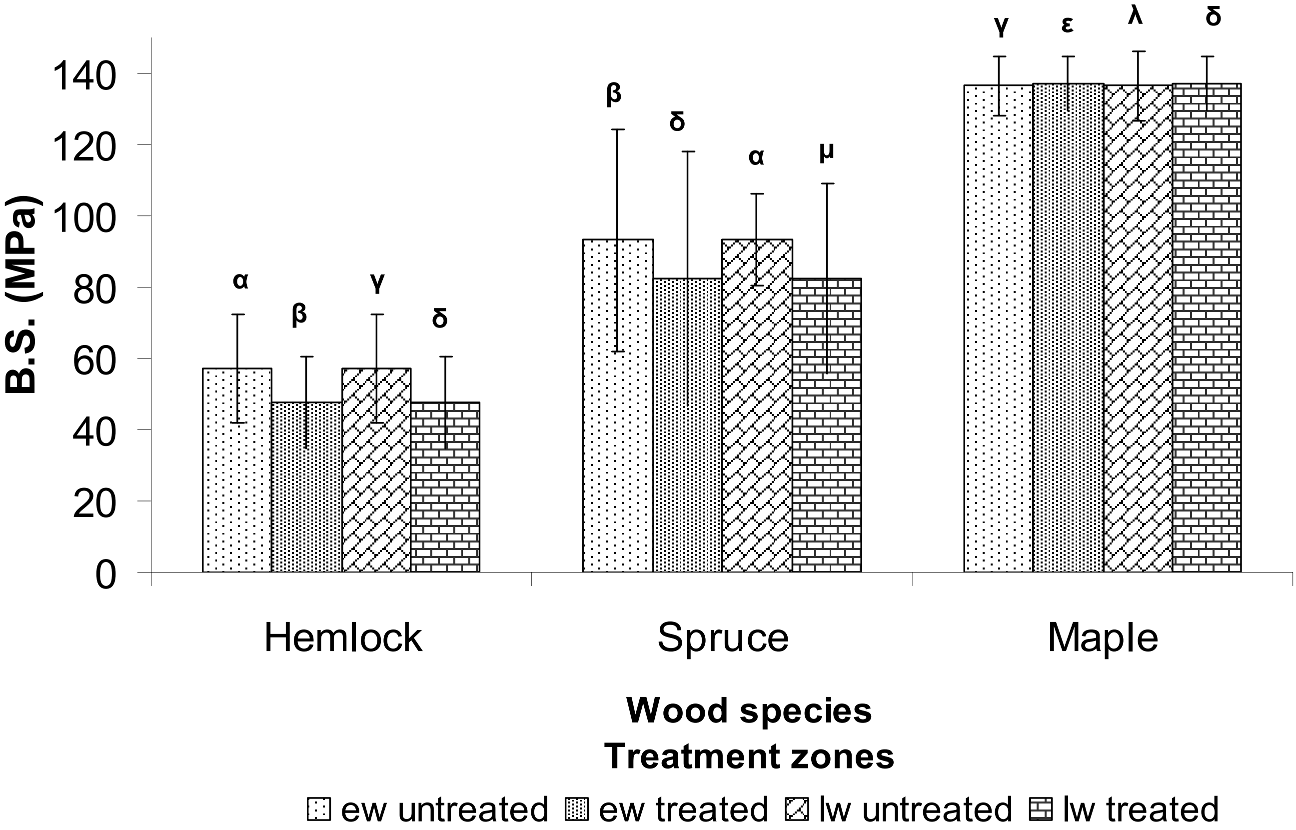
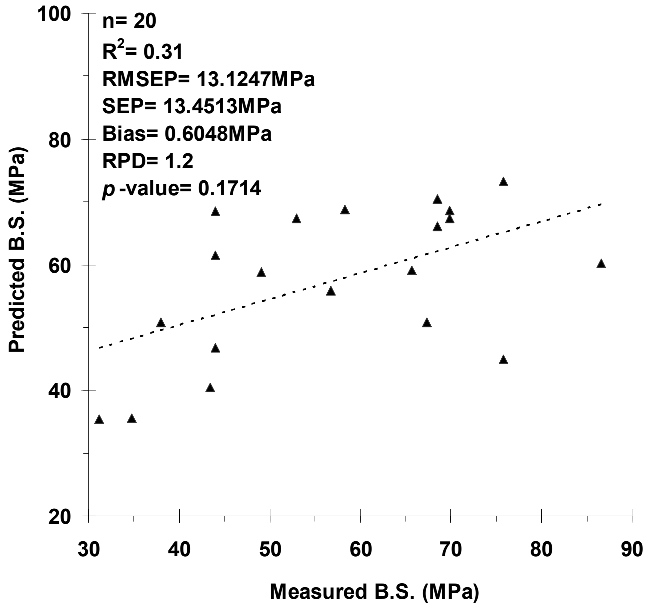
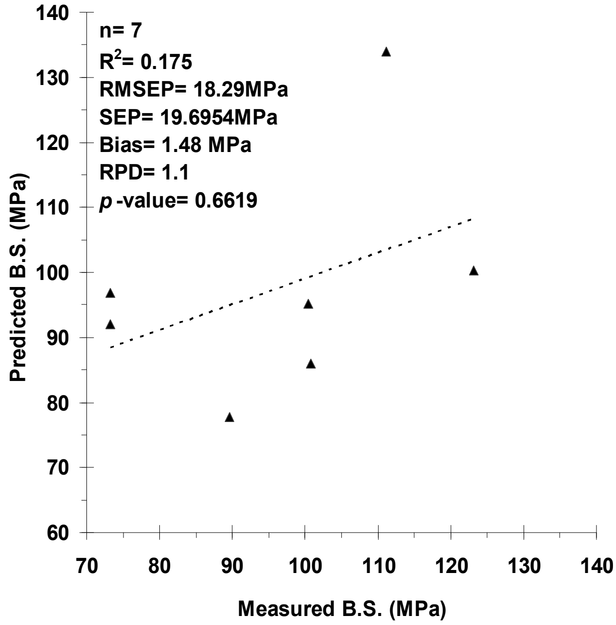


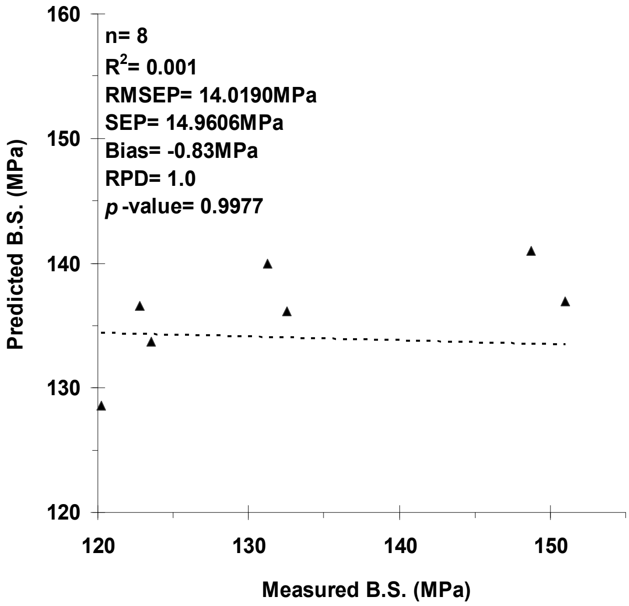
Figure 3. *Distribution of the bending strengths (B.S. in MPa) of earlywood (EW) and latewood (LW) for white spruce (Picea glauca), eastern hemlock (Tsuga canadensis L.) and soft maple (Acer rubrum) wood samples. For each probe, values sharing the same letter (α, β, γ, δ, ε, λ, μ) are not significantly different at the 5% level of confidence.*



(a) Earlywood Hemlock

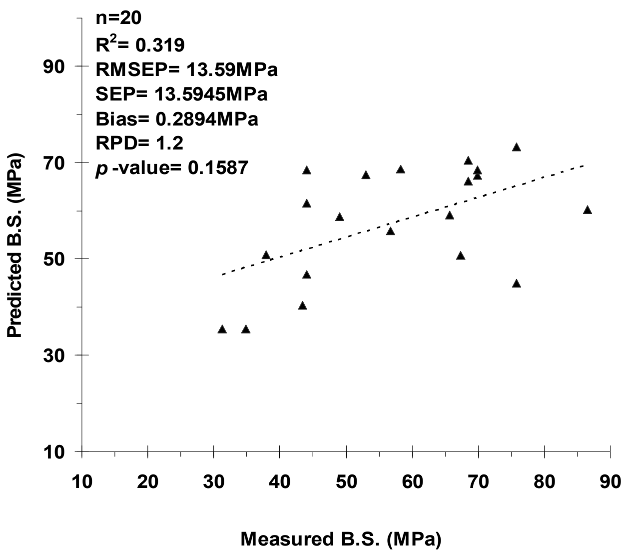


(b) Earlywood Spruce

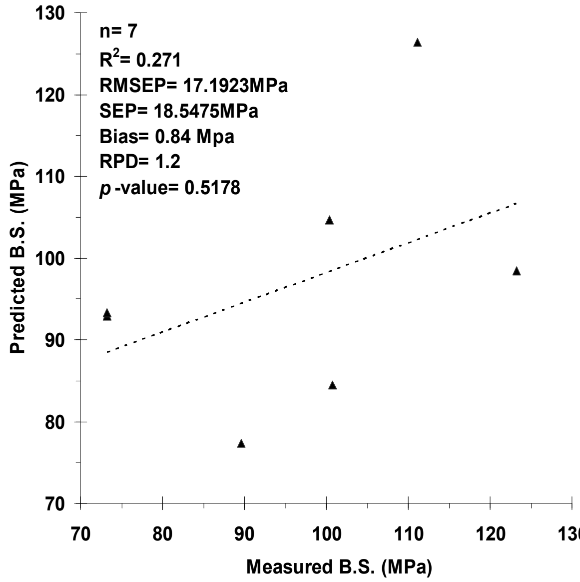


(c) Earlywood Maple

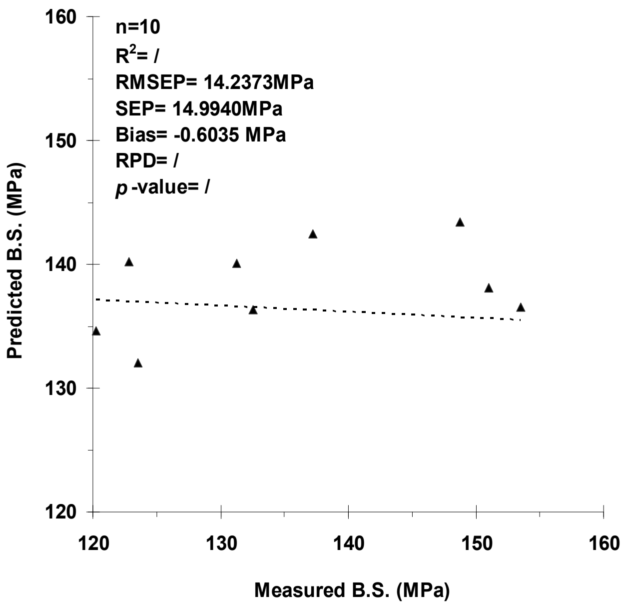
Figure 4. *Partial least square (PLS) regression model of earlywood (EW) for white spruce (Picea glauca), eastern hemlock (Tsuga canadensis L.) and soft maple (Acer rubrum) wood samples. Each dot represents an average of 6 spectra for calibration and validation set. The related statistics are presented in Table 1.*



(a) Latewood Hemlock



(b) Latewood Spruce



(c) Latewood Maple

Figure 5. *Partial least square (PLS) regression model of latewood (LW) for white spruce (Picea glauca), eastern hemlock (Tsuga canadensis L.) and soft maple (Acer rubrum) wood samples. Each dot represents an average of 6 spectra for calibration and validation set. The related statistics are presented in Table 2.*

Table 1. *Statistic parameters of the linear relationship of Figure 4, over PLS regression (calibration & validation) model for earlywood of white spruce (Picea glauca), eastern hemlock (Tsuga canadensis L.) and soft maple (Acer rubrum) wood samples: (a) Spruce, (b) Hemlock and (c) Maple. n represents the number of pairs that correspond to the average of 6 spectral scans.*

Species	Slope	Intercept	RMSE (%)	SE (%)	Bias (%)	R^2	*RPD*	*p-value*	n
Calibration									
a)	0.44	53.58	12.90	13.94	-1.09^{e-06}	0.44	1.3	0.2753	7
b)	0.63	21.07	9.26	9.50	6.10^{e-06}	0.63	1.6	0.0022	20
c)	0.24	104.18	10.51	11.07	7.63^{e-07}	0.24	1.1	0.4772	10
Validation									
a)	0.40	59.37	18.09	19.70	1.49	0.18	1.1	0.6697	7
b)	0.46	31.20	13.12	13.45	0.60	0.31	1.2	0.1714	20
c)	0.034	131.84	13.37	14.08	-0.39	0.001	1.0	0.9977	10

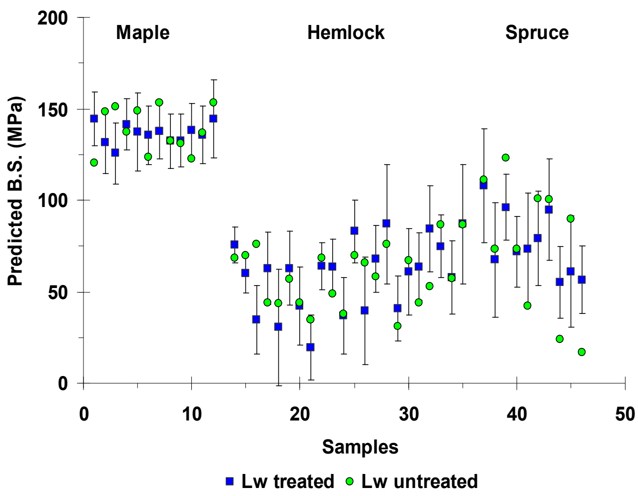
Table 2. *Statistic parameters of the linear relationship of Figure 5, over PLS regression (calibration & validation) model for latewood of white spruce (Picea glauca), eastern hemlock (Tsuga canadensis L.) and soft maple (Acer rubrum) wood samples: (a) Spruce, (b) eastern hemlock and (c) soft maple. n represents the number of pairs that correspond to the average of 6 spectral scans.*

Species	Slope	Intercept	RMSE (%)	SE (%)	Bias (%)	R^2	*RPD*	*p-value*	n
Calibration									
a)	0.45	63.01	12.83	13.86	-1.09^{e-06}	0.45	1.3	0.2632	7
b)	0.56	25.45	10.18	10.44	-5.72^{e-06}	0.56	1.5	0.0083	20
c)	0.18	112.58	10.92	11.51	7.63^{e-07}	0.18	1.1	0.5964	10
Validation									
a)	0.36	61.76	17.19	18.55	0.84	0.27	1.2	0.5178	7
b)	0.41	33.83	13.25	13.59	0.29	0.32	1.2	0.1573	20
c)	0.05	143.09	14.24	14.99	-0.60	-	-	-	10

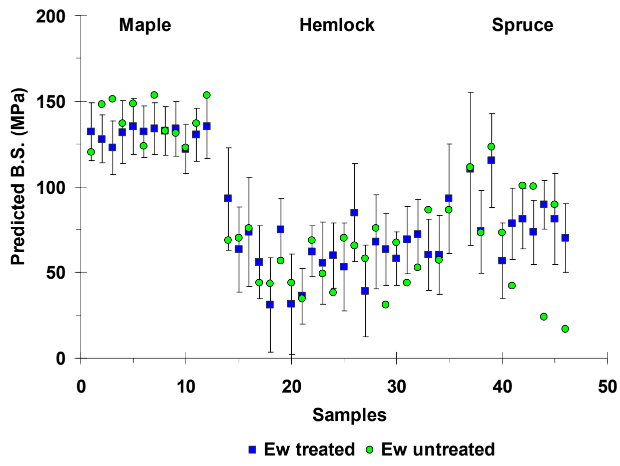
3.3. Sample-Specific Standard Errors of Prediction

Sample-specific standard errors of bending strengths prediction were shown in Figure 6. Average standard deviation values were in a similar range for all the three wood species; they were: 23.62 and 24.26 in Spruce (EW/LW treated/untreated), respectively; 23.64 and 19.86 in Hemlock (EW/LW treated/untreated), respectively; and 15.59 and 15.83 in Maple (EW/LW treated/untreated), respectively. Whereas, the related maximum bending strengths prediction values were: 115.28MPa (EW treated) and 123.21MPa (EW untreated), 107.9MPa (LW treated) and 123.21MPa (LW untreated) for Spruce; 93.01MPa (EW treated) and 86.55MPa (EW untreated), 86.93MPa (LW treated) and 86.55MPa (LW untreated) for Hemlock; 135.19MPa (EW treated) and 153.52MPa (EW untreated), 144.4MPa (LW treated) and 153.52MPa (LW untreated) for Maple.

The sample-standard errors of prediction of bending stress in all the three samples trended to underestimate the prediction in EW/LW similarly for treated and untreated wood samples. In EW/LW for Hemlock were again over-estimated. These prediction results were performed lower than the observations found by Faber and Bro (2001) and Koumbi-Mounanga et al (2015b), which were assessed a standard deviation of Gaussian peaks ranging from 3 to 5.



(a) Latewood



(b) Earlywood

Figure 6. *Sample-specific standard error of prediction for white spruce (Picea glauca), eastern hemlock (Tsuga canadensis L.) and soft maple (Acer rubrum) wood samples. Each dot represents an average of 6 spectra and the error bars are calculated by incorporating the standard deviation of measurement error into the predicted data values.*

4. Conclusions

Near-infrared (NIR) spectroscopy has the potential to estimate bending stress from wood components [in earlywood (EW)/latewood (LW)] after a hot oil treatment using the wavelength of 1100 to 2200nm region. The oil absorbed by the wood samples benefits the performance of NIR measurement in reduced the reflectance from 95 to 55%. Validation R^2 ranged from 0.18 to 0.31 for all the scans conducted in the EW zones that were underestimated than those in LW zones, whereas, validation R^2 ranged from 0.27 to 0.32 for Spruce and Hemlock. The Maple encountered a non linear regression that has been used as an active control in the present study. The investigation suggests that further work has to be developed as physics-based models to explain model influences with thermal treatment of wood material to the spectra. Also, we only tested the method to one commercially vegetal oil and further investigations on other varieties of oils as well as wood transformation samples are needed.

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