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Modeling bending strength of oil-heat-treated wood by nearinfrared spectroscopy

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Abstract Refractory wood species like American beech (Fagus grandifolia Ehrh.) and pin oak (Quercus palustris) are difficult to be treated by impregnation with wood preservative formulations or chemical modification agents. They need to be subjected to a preservative process that uses a heat treatment in a deep fryer with vegetable oil at 220 °C for 120 min. Such heat treatment influences several parameters related to the chemical and mechanical wood properties, such as the bending strength of earlywood (Ew) and latewood (Lw) growth rings. In this study, models to estimate bending strengths of Ew and Lw growth rings from near-infrared spectroscopy (NIRS) data have been proposed for oil-heat-treated wood samples of American beech and pin oak. The models were developed using the partition to latent structures (PLS) regression technique. For both species, the bending strength was significantly lower for the heat-treated wood compared to the untreated wood. In the case of beech, the mean bending strength was 112.87 ± 8.55 MPa for the untreated wood samples and 80.78 ± 4.79 MPa for the heat-treated wood samples. In the case of pin oak, the mean bending strength was 99.57 ± 5.34 MPa for the untreated wood samples and

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² Faculty of Forestry and Environmental Management, University of New Brunswick, Fredericton, NB, Canada 83.69 ± 5.71 MPa for the heat-treated wood samples. All the PLS models were significant at p value < 0.0001. For the Ew tissues, the R^2 with the validation data set was 0.78 for beech and 0.95 for pin oak. The corresponding root mean square errors of prediction (RMSEp) were 44.57 MPa for beech and 34.10 MPa for pin oak. The corresponding ratios of prediction to deviation (RPD) were 2.2 and 4.8 for beech and pin oak, respectively. For the Lw tissues, R^2 was 0.86 and 0.92 for beech and pin oak, respectively. The corresponding RMSEp were 50.01 MPa for beech and 47.87 MPa for pin oak. RPD values were 5.8 and 3.8 for beech and pin oak, respectively. For both the Ew and Lw tissues and species, B coefficients of the response surface models (ranging from -108.8 to 195.8) were statistically significant indicating that the models can be used to detect which wavelengths of the spectra have the highest impact on the effect of heat treatment on the bending strength.

Keywords American beech (*Fagus grandifolia* Ehrh.) · Bending strength · Near-infrared spectroscopy · Oil-heattreated wood · Pin oak (*Quercus palustris*)

Introduction

For suitable indoor and outdoor wood residential applications (deck and fence, framing lumber and wood sheathing panels, flooring), heat treatments are well known to improve wood physical and chemical properties by reducing wood hygroscopicity, color brightness, improving dimensional stability and significantly enhancing several other properties of structural wood such as the resistance against abiotic (UV-light oxidation) and biotic (microorganisms) agents (Tjeerdsma et al. 1998; Gérardin et al.

2008: Poaty et al. 2010). Oil-heat treatment is one of the most environmental-friendly processes that commonly uses heat transfers uniformly throughout wood and provides an oxygen-free environment that reduces radical formation during treatments and minimizes strength loss. It induces controlling effects of initial equilibrium of wood moisture content better than other thermal preservative processes, such as the Thermo Wood or Premium wood process in Finland, the retification process in France and the Plato process in Netherlands because oil-heat process is usually heated wood to stable temperatures ranging from 180 to 220 °C in low-cost oxygen environment such as in-stream or nitrogen and it allows an improved impregnation of several chemical preservatives (Esteves and Pereira 2009; Militz 2002). Oil-heat treatments are particularly adequate for refractory wood species (Sailer et al. 2000; Lyon et al. 2007; Mohebby et al. 2014) because oils are good heat transfer media and are potential carriers of the substances contained in the preservative mixture (Rapp and Sailer 2001; Mounanga 2015; Mounguengui et al. 2015). The heat treatments, however, modify the mechanical properties of the heat-treated wood because of the changes in the chemical composition of both the earlywood (Ew) and latewood (Lw) growth rings (Mott et al. 2002; Via et al. 2003; Mburu et al. 2007). Near-infrared spectroscopy (NIRS) can be a good tool to detect these chemical changes as the wood chemical composition is related to specific absorption bands of the near-infrared (NIR) spectra (Watanabe et al. 2011; Stirling et al. 2015). NIRS together with chemometric techniques was used to predict the change in several mechanical properties (tensile modulus of elasticity (MOE) and strength, bending stiffness and strength) of heat-treated wood from various species including European larch, radiate pine (Pinus radiate D. Don), and Douglas fir (Pseudotsuga menziesii var. menziesii) (Hoffmeyer and Pedersen 1995; Mott et al. 1996; Carneiro et al. 2010). In other studies, NIR spectra were used to predict several wood properties, including moisture content, surface color, contact angles, and adhesive bond strengths of wood products for a variety of wood species that include trembling aspen (Populus tremuloides Michx.), Eastern black spruce (Picea mariana var. mariana), and loblolly pine (Pinus taeda) (Meder et al. 2002; Kelley et al. 2004; Koumbi-Mounanga et al. 2015a).

The purpose of this study is to develop a method that uses NIR spectra and partial least square regression models to estimate the bending strength of oil-heat-treated Ew and Lw tissues of American beech (*Fagus grandifolia* Ehrh.) and pin oak (*Quercus palustris*) species. Such a method can replace costly and time-consuming measurements of bending strengths. The two refractory wood species were heat-treated with a simplified oil-heat treatment (OHT) process, which is derived from the German OHT process (*Menz Holz*, Germany) that utilizes soy oil. Among all the types of oil (linseed, sunflower, palm, rapeseed, and tall oil), soy oil is stable at a temperature approaching 230 °C (Militz and Tjeerdsma 2001; Wang and Cooper 2005; Tsuchikawa et al. 2005). The German OHT process was already shown to be particularly suitable for treating several refractory wood species like white spruce (*Picea glauca*), European beech (*Fagus sylvatica* L.), European oak (*Quercus robur* L.), and European silver fir (*Abies alba*, Pinaceae) (Shin and Lee 2000; Bailleres et al. 2002; Wentzel et al. 2018).

Materials and methods

Wood sample preparation for oil-heat treatment

The wood materials used in this study were extracted from American beech (Fagus grandifolia Ehrh.) and pin oak (Quercus palustris) lumbers. For each species, two boards were acquired green at a local lumber supplier in Southern Ontario (Canada) from two different trees. Working with more than two different boards allows considering more variable conditions. Thirty-six wood samples were extracted from one board, and 44 wood samples were extracted from another board. The wood samples were cut throughout the radial/tangential/longitudinal orientations with a dimension of 25 mm (radial) \times 25 mm (tangential) \times 10 mm (longitudinal) following the standard-size test specimens described in ASTM (2012). The 80 samples were randomly divided into a calibration data set of about 60 samples and a validation data set of about 20 samples following the method described in Hans et al. (2014). After being air-dried for 24 to 48 h at room temperature $(23 \pm 2 \text{ °C})$, each wood sample was conditioned to a relative humidity of approximately 2% prior to heating. While 36 wood samples were left as untreated control samples, about 54 wood samples were submerged and heated in fresh vegetal commercial soy oil (Brunge, Canada) at 220 °C and then held at this temperature during 120 min using the hot oil bath Fisher HiTemp Bath Model 160 chamber (Pittsburgh, PA 15275, USA) that was equipped with an internal fluid laminar flow. There were no external pressures activated to the heating system during the experiment. It is noted that to avoid the development of wood drying defects such as checking, cupping, crooking, bowing, twisting and directional grain raises due to temperature changes, a post-treatment procedure of delay cooling in degrading temperatures was applied to all the samples prior to examination. The heated samples were immediately removed from the hot bath after the 120 min of target temperature (220 °C), while the oil was still hot and then placed overnight in an oven conditioned at 103 °C using a *Despatch* heater chamber (Model# LDB1-69[S], Minneapolis, MN55044, USA) to gradually cooled down. The heat-treated and untreated wood samples were then subjected to a slow degrading temperature cascade in the *Despatch* heater chamber that was equipped with a digital temperature controller. The conditioned temperatures started at around 103 °C for 24 h and then reduced to 26 °C for the duration of the experiment in order to have the samples being around the relative humidity equilibrium. This setting was kept until the wood weight remained constant as described in Stamm (1964) and Hakkou et al. (2005).

Mechanical testing

Bending strengths were measured on the longitudinal faces of the wood samples using a *Zwick-Roell* load frame testing system (Zwick USA 1620 Cobb International Blvd, Kennesaw, GA 301552, USA) that was equipped with a 10 kN load cell. The *Zwick* testing system was associated with a control program of the screwdriver crosshead that was directly related to a three-point setup with the loading rate of about 4.05 mm/min following the standardized procedure (Jamsa and Viitaniemi 2001). It includes a support separation of 75 mm that is suitable for measuring spots in the Ew and Lw zones within the specimen as described in Biblis (1969).

Spectral measurements

Near-infrared (NIR) spectra were acquired on the two longitudinal faces of each wood sample having a moisture content of about 2% with an Ocean Optics NIR256-2.5-[HL-2000-FHSA-HP-LL-RS232] NIR spectrometer (Ocean Optics Inc., 830 Douglas Ave, Dunedin, FL 34698, USA). The Ocean Optics NIR spectrometer was equipped with an optical probe positioned at a 2 mm-diameter-beam on the top of the sample. It has a spectral resolution of 5 nm, and it was manually calibrated for white/dark after every three measurements. In order to build a robust model based on repeated measurements on samples at different periods, NIR spectral data were collected 2 to 4 times per day over the 6-month experiment duration with the Ocean Optics SpectraSuite program (Schimleck et al. 2005; Mara et al. 2012). Each wood sample was scanned 3 times on the Ew and Lw zones of both faces as described in Mounanga et al. (2012).

Data processing

Data processing was done with the Unscrambler 9.8 (CAMO software. Inc., 1 North Cir., Woodbridge, NJ 07095-2105, USA). The reflectance spectra that were

acquired over the 900-2450 nm wavelength range were smoothed by applying a second derivative 13-pt Savitzky-Golay transformation. They were then converted into absorption spectra, and a mean spectrum of 12 absorption spectra was computed. These mean NIR spectra were then related to the corresponding measured bending strengths using a partition to latent structures (PLS) regression modeling technique. Additionally, a response surface analysis was carried out to determine which wavelength is the most impacted by the variation in bending strength. Response surface analysis was performed to precisely describe the shape of the response surface using the Unscrambler program (CAMO software, USA) that includes ANOVA-based diagnostic tools. The PLS regression allows data reduction from a large number of interrelated variables in the NIR spectral region as described in Esbensen et al. (2002). In particular, it produces predictive models that use the NIR absorbance spectra to estimate bending strength in Ew and Lw growth rings. The PLS model was calibrated with measurements acquired over 21 and 39 untreated control and heat-treated samples, respectively. Then, the PLS was validated with measurements acquired succinctly over 5 untreated control and 15 heat-treated samples following the methods described in Martens 1985 and Kotz et al. 2005. In each case, the number of latent variables was 2 that corresponded to the minimum root mean square error (RMSE) (Martens et al. 2001). In parallel to the PLS model, response surface models were calibrated to determine the absorption bands where heat treatment effects are the most pronounced. Following Geladi and Kowalski (1986) and Faber et al. (2003), the PLS and response surface model accuracies were determined by the coefficient of determination (R^2) between measured and predicted values; the p value or significance level of R^2 , the root mean square error of prediction (RMSEp) and of calibration (RMSEc), the bias, which is the mean difference between measured and predicted values, the standard error of prediction (SEP) of the residuals values, and the ratio of prediction to deviation (RPD), which is a ratio of SEP and the standard deviation (STD) of the data.

Results and discussion

NIR spectra

Figure1 presents pictures of oil-heat-treated and untreated samples in the case of American beech and pin oak wood (Ew and Lw) tissues. The corresponding NIR reflectance spectra are presented in Fig. 2. They were acquired on the Ew and Lw portions of the longitudinal surfaces from both the heat-treated and untreated wood samples of both







Fig. 2 NIR spectra of untreated and oil-heat-treated earlywood (Ew) and latewood (Lw) tissues for American beech and pin oak wood samples. Each spectrum represents an average of 12 spectra

species. The overall NIR reflectance tends to gradually increase from about 55% to around 98% with the heat treatment action. The trend was similar for beech and pin oak wood tissues. This could be attributed to the variation of several wood properties (hygroscopicity, relative humidity, pH) due to the heat treatment in the Ew and Lw zones of both species. Figure 3 compares the second derivative Savitzky-Golay transformation of these NIR spectra. It reveals some spectral effects of the heat treatment induces spectral effects at around 1357–1459 nm, 1867–1935 nm, and 2071–2207 nm for the beech samples,



Fig. 3 NIR spectra after second derivative transformation $(d^2A/d\lambda^2)$ of untreated and oil-heat-treated earlywood (Ew) and latewood (Lw) tissues for American beech (*F. grandifolia* Ehrh.) and pin oak (*Q. palustris*) wood samples. Each spectrum represents an average of 12 spectra

and at around 1320–1460 nm, 1560–1620 nm, 1697–1799 nm, 1860–1940 nm, and 2071–2310 nm region for the pin oak samples. These wavelengths are related to the fundamental (first, second, and third overtones) stretching absorptions and the combination bands of vibrational transitions in near-infrared, mainly C-H (hydrocarbons), C=O (cellulose, holocellulose, hemicelluloses), O–H (water, alcohol, phenol), and N–H (lignin) functional groups (Lee and Luner 1972; Koumbi-

Mounanga et al. 2015b). The observed spectral changes are directly related to wood surface changes due to the oil absorption and heating and cooling effects within the specimen. Similar observations were reported by Mounanga et al. (2015) in a study about bending strength of Ew/Lw tissues from a variety of several refractory wood species such as eastern hemlock (Tsuga Canadensis L.), soft maple (Acer rubrum L.), and white spruce (Picea glauca). The observed spectral differences might also be due to the degradation of wood chemical elements such as holocellulose and hemicelluloses and/or lignin including carbohydrates and deacetylation reactions of polyoses. The degradation of individual wood chemical components under the effect of heating treatment actions was already reported in the case of Douglas fir (Pseudotsuga menziesii var. menziesii) and trembling aspen (Populus tremuloides Michx.) (Gindl et al. 2001; Koumbi-Mounanga et al. 2013, 2016). Indeed, wood samples reached 200 °C during the heat treatment, which is a temperature where wood chemical components like extractives, xylose, galactose, and mannose can be degraded (Sidorova 2008; Bachle et al. 2010; Hamada et al. 2017).

Bending strength

Figure4 shows the mean bending strength of 12 measurements performed on the longitudinal faces. They have a standard deviation (STD) of about 5.35. In our study, the separation between bending strengths measured on Ew and Lw was subjective due to the impossibility of isolating individual Ew and Lw tissues. The only option was to perform the bending strength measurements on variable portions of Ew and Lw of one bloc of the same sampling board for each species. The mean bending strengths at different Ew and Lw proportions within a board were then related to each tissue of interest. Indeed, the heat treatment



Fig. 4 Bending strength (in MPa) distribution in earlywood (Ew) and latewood (Lw) of untreated and oil-heat-treated American beech and pin oak wood wafer 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

changed both the chemical composition of Ew and Lw zones by affecting cell wall compounds and extractives (Via et al. 2009). For both species, the bending strength is significantly lower for the heat-treated wood compared to the untreated wood. In the case of beech, the mean bending strength was 112.87 ± 8.55 MPa for untreated wood samples and 80.78 ± 4.79 MPa for the heat-treated wood samples. In the case of pin oak, the mean bending strength was 99.57 ± 5.34 MPa for the untreated wood samples and 83.69 ± 5.71 MPa for the heat-treated wood samples. The decrease in bending strengths of heat-treated wood samples indicates a wood weakening due to the heat treatment action that was already reported for various wood species such as European aspen (Populous tremula L.), European oak (Quercus petraca Liebl.), spruce (Picea abies (L.) Karst), longleaf pine (Pinus palustris), and European larch (Awoyemi et al. 2009; Salim et al. 2010). The decrease in bending strengths for the heat-treated wood is larger for the beech than for the pin oak samples. This is due to different chemical compositions of the intrinsic cell wall chemistry between both the species (Gindl et al. 2004; Mecca et al. 2019). The observed mean bending strengths at the Ew and Lw zones were on the same order of magnitude for both opposite surfaces of the specimen. This is due first to the defect loading distribution and to the fiber deviation. Indeed, the strength is fundamentally affected by the internal wood loading distribution (tissues to tissues) within the board samples and the direction of the load in relation to the grain. Another reason is the low scanning capacity of the NIR instrument (especially its probe size) to detect with enough accuracy the Ew and Lw limits as well as other types of defects (i.e., hollows, cracks, wood zones deteriorated by microorganisms or experimental handling, knots, resin bags, and reaction woods).

PLS regression models

In order to infer the heat treatment effect on the bending strength, the NIR spectra were first related to the measured bending strengths by a PLS regression model for each Ew/ Lw zone of beech and pin oak samples (Fig. 5). Each PLS model was built using an average of 6 absorption spectra. The related statistical metrics are summarized in Table 1. All predicted PLS models were statistically significant (*p* value < 0.0001). The R^2 values ranged from 0.78 to 0.92 for the beech (Ew and Lw) samples and from 0.86 to 0.95 for the pin oak (Ew and Lw) samples. The corresponding RMSEp were 44.57 MPa and 50.53 MPa for beech Lw and Ew tissues, respectively. RMSEp were 34.10 MPa and 47.87 MPa for pin oak Ew and Lw tissues, respectively. The R^2 and RMSEp values were on the same order as the ones obtained by Kohan et al. (2012), who used NIRS to



Fig. 5 Partial least squares regression model to predict the bending strength in the case of untreated and oil-heat-treated earlywood (Ew) and latewood (Lw) tissues of American beech and pin oak wood samples. Only validation datasets were represented for β .1.1) Beech

(Ew), β .1.2) Pin oak (Ew), β .2.1) Beech (Lw) and β .2.2) Pin oak (Lw). Each dot represents an average of 6 spectra. The related statistics are presented in Table 1

predict bending stiffness and tensile MOE of southern yellow pine (*Pinus* spp.) strand feedstock (R^2 between 0.43 and 0.76 and RMSEp between 0.77 and 16.01 MPa). The R^2 and RMSEp were not as good as the ones of Barré et al. (2018), who developed PLS models to predict with NIRS the degradation of bending strengths of silver fir (*Abies alba*) wooden strips during microbial decomposition (R^2 from 0.89 to 0.99 and RMSEp from 0.13 to 0.15 MPa) (Thumm and Meder 2011; Hein et al. 2011; Leblon et al. 2013).

For both species and tissues (Ew and Lw), the related ratio of prediction to deviation (RPD), i.e., the ratio of SEP corrected for bias to STD of the validation data sets, ranged from 1.9 to 8.2 (Table 1). They indicated models with moderate quality that can be used at least for rough screening. The RPD values were similar to those reported by the literature for Eucalyptus species (*Eucalyptus bentamii* and E. *pellita*) (RPD values ranging from 1.33 to 5.2 and 1.28 to 5.9, respectively) and for Japanese species (Japanese cedar and J. cypress) (RPD values ranging from 1.30 to 2.01 and 1.78 to 4.97, respectively) (Tham et al. 2019; Diniz et al. 2019). Our RPD values were slightly higher than those of Koumbi-Mounanga et al. (2015c) (between 1.2 and 6) who estimated moisture content of trembling aspen (*Populus tremuloides* Michx.) strands with NIRS data.

Tissue/species	Slope	Intercept	RMSEp (MPa)	SEP (MPa)	Bias (MPa)	\mathbb{R}^2	RPD	p value	n
α) Calibration									
a.1) Earlywood(Ew)									
Beech (Ew)	1.12	71.05	46.13	46.45	0.89	0.81	2.35	.0001	295
Pin oak (Ew)	1.26	47.68	34.92	35.23	0.94	0.97	5.84	.0001	255
a.2) Latewood(Lw)									
Beech (Lw)	1.22	61.42	53.56	54.12	0.69	0.98	8.28	.0001	360
Pin oak (Lw)	1.29	49.78	52.84	53.62	-0.98	0.95	4.56	.0001	294
β) Validation									
$\beta.1$) Earlywood(Ew)									
Beech (Ew)	0.90	57.35	44.58	44.88	0.85	0.78	2.17	.0001	96
Pin oak (Ew)	1.04	39.34	34.11	34.41	0.82	0.95	3.85	.0001	81
β .2) Latewood (Lw)									
Beech (Lw)	0.97	49.85	50.03	50.53	0.65	0.92	2.28	.0001	109
Pin oak (Lw)	0.93	41.55	47.87	48.58	-0.96	0.86	1.96	.0001	88

Table 1 Summary of the statistical metrics for the PLS model predicting bending strengths of earlywood/latewood of American beech and pin oak wood samples from NIRS data

(α) Calibration dataset and (β) validation dataset. *n* represents the number of objects which is an average of six scans measured for each model built in Ew and Lw tissues

Response surface models

The chemical changes due to the heat treatments are very subtle and thus difficult to be quantified by using simply the peak absorption bands of the spectra. By contrast, response surface analysis can be more sensitive to the subtle chemical changes due to the heat treatment (Via et al. 2005). Response surface models were established in the following wavelength domains, which limits were randomly selected for the analysis: 900-1630 nm (Model #1), 1630-1870 nm (Model #2), 1870-2200 nm (Model #3) and 2200-2450 nm (Model #4) following the method described in Kalil et al. (2000). The related model statistics are summarized in Table 2. The models have multiple correlation coefficients ranging from 0.718 to 0.887, R^2 values ranging from 0.515 to 0.786 and B coefficients, which express the link between variations in the predictor and in the response, ranging from -108.791 to 195.803. The variable heat treatment conditions induced different absorption response effects that can be roughly divided into two categories: (1) a negative effect that helps to check the amount of significant absorption response effects and (2) a positive effect that gives insight into the interpretation of the relationship between measured and predicted absorption response effects in specific spectral regions. The wavelengths of each response model that have the most significant absorption differences were assigned to particular chemical components using the method described in Beall and Eickner (1970) and Schwanninger et al. (2011) as follows:

- Model #1: Wavelengths around 1255.84 nm were assigned to the second overtone of C-H stretching vibration from CH and CH₂ groups (cellulose);
- Model #2: Wavelengths around 1698.46 nm were assigned to the first overtone of C-H stretching vibration from CH₃ groups (hemicelluloses);
- Model #3: Wavelengths around 1971.86 nm were assigned to the O–H stretching vibration from H₂O (water);
- Model #4: Wavelengths around 2281.31 nm were assigned to the O–H stretching vibration and C-H deformation vibration (semi-crystalline or crystalline regions in cellulose), and wavelengths around 2315.86 nm are assigned to the O–H stretching vibration and C–C stretching vibration and/or C-H stretching vibration and C-H stretching deformation vibration (cellulose).

The assigned bands were not specifically attributed to typical wood components (i.e., C=O functional groups in cellulose, holocellulose, hemicelluloses, or/and N–H functional group in lignin), as they corresponded to the second overtones of hydroxyls and third overtones of C–H stretching vibrations (Mounanga 2008; Green et al. 2010). However, the resulting overlaid spectra obtained by grouping the four models (Model#1 to #4) allowed defining the most statistically different absorption bands concerning heat treatment modifications of the Ew and Lw zones for both the beech and pin oak samples (Fig. 6). This figure shows that the heat treatment changed the spectrum shape between 1255 and 2315 nm, indicating that there is a

Table 2Summary of thestatistical metrics for theresponse surface analysismodels of Fig. 6

Metrics (*)	SS	DF	MS	F ratio	p value	B coefficients	STD
Model #1	7.28E ⁺⁰⁴	22	$3.31E^{+03}$	6.84	0		
Error	1.98E ⁺⁰⁴	41	483.11				
Adjusted total	9.26E ⁺⁰⁴	63	$1.47E^{+03}$				
Intercept	516.61	1	516.61	1.06	$3.07E^{+03}$	99.63	96.34
949.35 nm	$1.35E^{+03}$	1	$1.35E^{+03}$	2.79	$1.02E^{+03}$	-103.74	62.08
1221.81 nm	$2.05E^{+03}$	1	$2.05E^{+03}$	4.23	$4.59E^{+03}$	375.21	182.28
1255.84 nm	$2.41E^{+03}$	1	$2.41E^{+03}$	4.99	$3.10E^{+02}$	-362.84	162.39
1289.86 nm	$1.48E^{+03}$	1	$1.48E^{+03}$	3.05	$8.81E^{+02}$	265.01	151.66
Model #2	$4.82E^{+04}$	7	6.89E ⁺⁰³	8.69	0		
Error	$4.44E^{+04}$	56	791.99				
Adjusted total	9.26E ⁺⁰⁴	63	$1.47E^{+03}$				
Intercept	$2.07E^{+03}$	1	$2.07E^{+03}$	2.61	1.11E ⁺⁰¹	99.63	61.67
1664.36 nm	$5.87E^{+03}$	1	$5.87E^{+03}$	7.41	$8.50E^{+03}$	53.26	19.57
1698.46 nm	$7.13E^{+03}$	1	$7.13E^{+03}$	9.01	$4.54E^{+03}$	-100.12	33.36
1766.70 nm	$3.03E^{+03}$	1	$3.03E^{+03}$	3.83	$5.53E^{+02}$	167.29	85.48
1869.18 nm	$4.52E^{+03}$	1	$4.52 E^{+03}$	5.71	$2.02E^{+02}$	-19.56	8.18
Model #3	6.57E ⁺⁰⁴	10	6.57E ⁺⁰⁴	12.99	0		
Error	$2.66E^{+04}$	53	506.07				
Adjusted total	9.26E ⁺⁰⁴	63	$1.47E^{+03}$				
Intercept	$1.06E^{+03}$	1	$1.10E^{+03}$	2.08	$1.54E^{+01}$	99.63	68.94
1937.61 nm	$1.88E^{+03}$	1	$1.88E^{+03}$	3.71	$5.94E^{+02}$	-54.03	28.05
1971.86 nm	$2.24E^{+03}$	1	$2.24E^{+03}$	4.43	$4.10E^{+02}$	92.07	43.72
2006.13 nm	$2.81E^{+03}$	1	$2.81E^{+03}$	5.55	$2.22E^{+02}$	-108.52	46.65
2040.42 nm	$2.59E^{+03}$	1	$2.59E^{+03}$	5.11	$2.75E^{+02}$	120.02	53.10
Model #4	$4.77E^{+04}$	6	7.94E ⁺⁰³	10.08	0		
Error	4.49E ⁺⁰⁴	57	787.81				
Adjusted total	9.26E ⁺⁰⁴	63	$1.47E^{+03}$				
Intercept	$2.46E^{+04}$	1	$2.46E^{+03}$	3.12	$8.25E^{+02}$	99.63	56.37
2246.80 nm	$4.14E^{+03}$	1	$4.14E^{+03}$	5.26	$2.55E^{+02}$	48.96	20.97
2281.31 nm	$8.59E^{+03}$	1	$8.59E^{+03}$	10.91	$1.70E^{+03}$	-157.94	47.82
2315.86 nm	$9.05E^{+03}$	1	$9.05E^{+03}$	11.49	$1.30E^{+03}$	190.10	56.07
2350.44 nm	$2.95E^{+03}$	1	$2.95 \ \mathrm{E}^{+03}$	3.74	$5.81E^{+02}$	-90.64	46.86

(*) SS = standard deviation of measured versus predicted values; DF = degrees of freedom; MS = mean square; F ratio = ratio between explained and residual variances; p value = significance level which measured the probability that an R^2 should be as large as it is with a real value zero; B coefficient or regression coefficient between variations in the predictors and variations in the response; STD = standard deviation of the reference data; intercept = offset or the point where a regression line crosses the ordinate (*Y*-axis)

correlation between the reflectance at these wavelengths and the heat treatment action on Ew and Lw tissues. The related absorption peaks have correlations with bending strengths that are highly significant with p values less than 0.001 for both species (beech and pin oak) (Table 2). A statistical comparison of the mean square (MS) for the residual error and the standard deviation of measured versus predicted values (SS) for the pure error allows to determine a slight decrease in mechanical bending strength properties due to the heat treatment on the absorption spectra of Ew and Lw portions of both beech and pin oak woods (Rinnan et al. 2009; Liu et al. 2011; Koumbi-Mounanga et al. 2018). For the Ew and Lw tissues, the response surface models have low probabilities (p < 0.005), which means that these tissues were significantly influenced by the high temperatures occurring during the oil-heat treatment. For example in the wavelength regions between 1630 and 1870 nm (Model #2) as well as for the wavelength regions from 2200 to 2450 nm (Model #4), the heat-treated pin oak Ew tissues have better absorptions than pin oak Lw tissues, of about 11.8 \pm 6.89% for Model #2 and of 8.28 \pm 1.37% for Fig. 6 Response surface effects of bending strength distribution for earlywood (Ew) and latewood (Lw) untreated and oil-heat-treated wood wafers of American beech and pin oak. 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. The related statistics are presented in Table 2



Model #4 with the respective probabilities of 0.004 and 0.001. The beech heat-treated Ew tissues have as well moderate absorptions than beech heat-treated Lw tissues of about 5.86 \pm 4.04% and 1.91 \pm 2.99% for Models #2 and #4, respectively. The corresponding probabilities were significant at 0.005 and 0.001. These absorptions were related to specific absorption peaks as follows: at the wavelengths around 1698.46 nm and 2281.31 nm for pin oak Ew heat-treated samples and at the wavelengths around 1664.36 nm and 2315.86 nm for beech Ew heat-treated samples. These peaks corresponded to the mean bending strength of 83.69 \pm 5.71 MPa for pin oak Ew heat-treated samples and of 80.78 ± 4.79 MPa for beech heat-treated wood samples. They can be attributed to C-H (cellulose and hemicelluloses), O-H (celluloses) and C-C in cellulose or/and lignin functional groups (Mounanga et al. 2008; Xiong et al. 2016; Wang et al. 2018).

Conclusions

The present study proposed a reliable nondestructive testing method that related, through with a PLS regression model, NIR spectra to bending strengths of Ew and Lw tissues of samples subjected to oil-heat treatments in the case of two refractory wood species (American beech and pin oak). Both wood species are suitable for aesthetical indoor and outdoor residential applications, but their preservation process that required an oil-heat treatment has an impact on the bending strength and chemical composition of both the Ew and Lw tissues. For both species, the bending strengths were significantly lower for the heattreated wood (Ew and Lw) tissues compared to the untreated wood (Ew and Lw) tissues. In the case of beech, the mean bending strength was 112.87 ± 8.55 MPa for the (Ew and untreated wood Lw) samples and 80.78 ± 4.79 MPa for the heat-treated wood (Ew and Lw) tissues. In the case of pin oak, the mean bending strength was 99.57 ± 5.34 MPa for the untreated wood (Ew and Lw) tissues and 83.69 ± 5.71 MPa for the heat-treated wood (Ew and Lw) tissues. All the PLS models were significant at p value < 0.0001. For the Ew tissues, the R^2 with the validation data set was 0.78 for beech and 0.95 for pin oak. The corresponding ratios of prediction to deviation (RPD) were 2.2 and 4.8 for beech and pin oak, respectively. For the Lw tissues, the R^2 were 0.92 and 0.86 for pin oak and beech, respectively. RPD values were 5.8 and 3.8 for beech and pin oak, respectively. For both Ew and Lw tissues, the B coefficients of the response surface models ranged from -108.8 to 195.8 and they were statistically significant indicating that the models were relevant to detect the wavelengths that have the highest impact on the effect of heat treatment action on the bending strength.

While the proposed method gives encouraging results, the developed models are still empirical and there is the need to develop physics-based models to derive bending strengths from NIRS data. The heat treatment process and the use of oil soybean such as transfer media increased fatty acid and esters which have a possible influence in NIR absorption. It, however, makes difficult the identification of chemical functional groups by using NIR spectroscopy.

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Compliance with ethical standards

Conflict of interest No potential conflict of interest was reported by the authors.

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