



Wood properties of mature loblolly pine grown in Hawaii, North Carolina, and Mississippi: impacts of climate conditions on specific gravity and chemical composition

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Abstract

Higher productivities for loblolly pine (*Pinus taeda* L.) growing at exotic locations (e.g., Hawaii, Brazil) indicate that the full growth potential of this southern pine species has yet to be reached in plantations across the southeastern United States (US). The higher productivity of Hawaii-grown loblolly pine has been attributed to more favorable climate conditions. To date, physical, anatomical, and chemical property data for loblolly pine wood from exotic locations are scarce. Mid-infrared spectroscopy coupled with multivariate analysis was used to screen for chemical differences between wood samples from Hawaii (HI), North Carolina (NC), and Mississippi (MS). The principal component analysis scores plot showed that the MS samples formed a distinct cluster apart from overlapping clusters for the other two sites. Using standard wet chemistry procedures, we found that the mean lignin content for the MS site (30.1%) was significantly lower than that determined for the NC (34.5%) and HI (33.9%) sites; said lower lignin value was offset by significantly higher glucose and mannose contents. Results on the US mainland were seemingly consistent with the greater formation of latewood at sites with longer growing seasons, and the lower lignin content of latewood relative to earlywood shown for pines. Given the low percent latewood for the HI site (37.2%) relative to both mainland sites (MS, 54.8%; NC, 53.1%), yet similar lignin contents between the NC and HI sites, it appears that the relative lignin value for these exotically-grown loblolly pine trees cannot be solely attributed to the proportion of latewood.

1 Introduction

The productivity of loblolly pine (*Pinus taeda* L.) plantations has increased through the implementation of genetic improvements and intensive silvicultural practices (Borders and Bailey 2001; Fox et al. 2007; Jokela et al. 2010). Sites that had growth rates of $6.3 \text{ m}^3 \text{ ha}^{-1} \text{ y}^{-1}$ can now produce

more than $24.5 \text{ m}^3 \text{ ha}^{-1} \text{ y}^{-1}$ using modern management practices (Fox et al. 2007). Despite such gains in productivity, the full growth potential of loblolly pine has yet to be reached in plantations across the southeastern United States (US), evidenced by higher carrying capacities in exotic environments in Hawaii (HI), Brazil, and Argentina (Harms et al. 1994, 2000; Schultz 1999; Borders and Bailey 2001; Samuelson et al. 2010). As a specific example, the maximum basal area, as a measure of carrying capacity (Yang and Burkhart 2017), was $103 \text{ m}^2 \text{ ha}^{-1}$ at an age of 34 years (Harms et al. 2000), and $93 \text{ m}^2 \text{ ha}^{-1}$ at an age of 48 years (Samuelson et al. 2010), for an experimental loblolly pine site in HI; basal areas for seven closed canopy US mainland stands monitored from ages of 6 to 24 years reached only 45 to $48 \text{ m}^2 \text{ ha}^{-1}$ (Jokela et al. 2004). Undoubtedly, favorable climate/environmental conditions such as mild year-long temperatures, longer day lengths, a longer growing season,

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and nutrient-rich soils contribute to the higher productivity experienced for HI-grown loblolly pine (Harms et al. 1994, 2000; Samuelson et al. 2010, 2013).

An outcome of high loblolly pine productivity is the ability to obtain merchantable material, such as pulpwood, after just 10 years (Dahlen et al. 2018). For loblolly pine on the US mainland, juvenile wood comprises about the first 10 growth rings near the pith (Tasissa and Burkhart 1998; Clark and Saucier 1989; Clark et al. 2006; Jordan et al. 2008). The quality of juvenile wood is usually deemed as inferior to mature wood for most end uses, but not all (Zobel and Sprague 1998; Larson et al. 2001; Moore and Cown 2017). Plantation loblolly pine wood properties have been widely studied on the US mainland to gain an understanding of the impacts of forest management practices and physiographic region on the quality of wood being produced (Clark et al. 2006; Jordan et al. 2008; Dahlen et al. 2018, 2021; Eberhardt et al. 2022). To date, only a few studies have reported physical property data for loblolly pine wood from HI (Skolmen 1963; Samuelson et al. 2010, 2013), with one of these studies (Samuelson et al. 2013) providing the only direct comparison with concurrent analyses of loblolly pine samples from mainland sites in Mississippi (MS) and North Carolina (NC). Although loblolly pine is the most planted pine in Brazil (Dobner et al. 2018), a search of the literature yielded only a limited number of reports of wood physical properties for this species in this favorable exotic environment (Higa et al. 1973; Hart 2009; Schimleck et al. 2020; Topanotti et al. 2021; Pereira et al. 2023).

Loblolly pine is the primary source of pulpwood both in the southeastern US (Schultz 1999; McKeand et al. 2003; South and Harper 2016) and Brazil (Dobner et al. 2018; Topanotti et al. 2021). During chemical pulping processes, much of the lignin needs to be removed to allow the fibers to separate. A search of the literature yielded only a few reports of wood chemistry data for loblolly pine growing in exotic environments, and none of these provide direct comparisons to native-grown trees. As examples, Bonfatti Júnior et al. (2019) recently compared wood chemistry values for *Cryptomeria japonica* wood chips from an experimental forest to industrial wood chips of loblolly pine, both sourced in Brazil, and Modes et al. (2019) reported lignin data collected in a few studies on Brazilian-grown loblolly pine.

Non-destructive spectroscopic methods based on chemical features of the sample are increasingly used for wood chemistry determinations since they provide the capacity to efficiently analyze large numbers of samples (Kelley et al. 2004; Sykes et al. 2005; Jones et al. 2006; Aspinwall et al. 2010; Acquah et al. 2015; Fahey et al. 2017). For example, global near infrared models were developed by Hodge and Woodbridge (2010) to predict lignin and cellulose contents for 517 samples of wood collected from tropical and

temperate pines growing in Brazil, Chile, Columbia, and South Africa; loblolly pine was included in the models, but was an exception among the species studied, being sourced from its natural range in the southeastern US. Direct side-by-side comparisons of predicted wood chemistry data between geographic regions have been made for various pine species; however, samples are generally collected from sites within the same country (Sykes et al. 2005; Aspinwall et al. 2010; Fernandes et al. 2017).

While wood chemistry data taken from the literature can be compiled/compared to assess possible similarities (or differences) in the wood sourced from trees planted in exotic and native environments, direct comparisons (i.e., same method/lab/study) are more definitive. To accommodate the within-tree wood chemistry variability known to occur in pines (Via et al. 2007; Thumm et al. 2016), consistency in the sampling between the study trees is essential. Aspinwall et al. (2010) found that loblolly pine pith-to-bark wood strips from wood disks taken at breast height gave lignin and cellulose contents that were representative of whole tree values, estimated from wood disks taken at 2.4 m incremental heights; the coefficients of determination (R^2) of lignin and cellulose were 0.87 and 0.74, respectively. At this juncture, it should be noted that softwood chemical compositions do vary within a given growth ring, with the earlywood typically showing higher lignin (lower cellulose) contents relative to the latewood (see Gladstone et al. 1970; Saka and Thomas 1982; Gindl 2001; Fredriksson et al. 2018).

Our prior wood property comparisons of mature HI-grown loblolly pine to mature loblolly pine from the US mainland led us to attribute the lower ring specific gravity (SG) and percent latewood values to the more favorable climate/environmental conditions in HI (Samuelson et al. 2013). It remained to be determined if any wood chemistry differences coincided with these physical/anatomical property differences. We hypothesize that the observed lower ring SG and percent latewood, as a manifestation of lower latewood formation, will coincide with higher lignin (lower polysaccharides) values for the HI-grown loblolly pine. Using samples of loblolly pine wood remaining from our prior research (see Samuelson et al. 2013), Fourier transform infrared (FTIR) spectroscopy coupled with multivariate analysis was employed to screen for differences in wood chemistry between the mainland and HI sites. Results from this screening led us to justify wet chemical analyses to resolve the magnitudes and statistical significance of the wood chemistry differences, focusing on the contents of lignin and the polysaccharides. While other environmental factors cannot be excluded (e.g., soils, day length), the favorable climate conditions in HI are presumed to be a highly impactful factor for any differences in the physical, anatomical, and chemical property data.

2 Materials and methods

2.1 Source of increment cores and processing

Loblolly pine increment cores were collected from trees at 3 different locations within the US for a study involving assessments of wood properties determined by X-ray densitometry (Samuelson et al. 2013). The two US mainland sites (MS and NC), and the exotic site (HI), are briefly described in separate sections below, with some of the previously reported stand attributes shown in Table 1. Increment cores (12 mm diameter) were taken at breast height (1.4 m) from trees using an increment borer, processed for X-ray densitometry by gluing them into wood core holders, and then sawn into assemblies having a constant thickness (2.3 mm). Sawn assemblies from the HI and MS sites were extracted with acetone (Eberhardt and Samuelson 2015). An X-ACTO knife (#19 blade) was then used to remove both the adhering core holder wood and any increment core wood showing glue penetration. A hand lens facilitated sample inspection to ensure that all glue-containing pores were completely removed. The remaining glue-free material was then chopped into fragments using anvil pruning shears. Wood fragments representing each whole core were twice ground in a Retsch centrifugal mill (ZM 200) equipped with a 1 mm mesh screen. Matching duplicate cores available from the MS site were directly extracted/chopped/milled to provide samples for the spectroscopic and wet chemical analyses.

2.2 Study sites

2.2.1 Hawaii (HI)

The HI-grown loblolly trees were from a spacing trial (1.8, 2.4, 3.0, and 3.7 m² spacings with 4 blocks) established in 1961 on abandoned pastureland by the USDA Forest Service and the Hawaii State Division of Forestry and Wildlife (Whitesell 1970, 1974). The HI site (20.49°N, 156.17°W), at an elevation of 1140 m, was in the Olinda Forest Reserve on the island of Maui. Three randomly selected dominant trees from each block were sampled in January 2009 to give a total of 48 increment cores (Samuelson et al. 2013). For the current study, only 21 of the original 48 increment

cores remained available for spectroscopic and wet chemical analyses.

2.2.2 Mississippi (MS)

The first mainland site was in the USDA Forest Service's Harrison Experimental Forest (30.65°N, 89.04°W) within the DeSoto National Forest near Saucier, MS. The elevation at the site is 50 m. Trees sampled were from a randomized complete block design study with 5 cultivation-fertilization treatments and 4 blocks as described in prior reports (Schmidtling 1973; Johnsen et al. 2009; Samuelson et al. 2013). Trees sampled were from those cultivated with a single application of 448 kg ha⁻¹ of NPK fertilizer. Two increment cores were removed in July 2010 from 7 trees in each of 3 plots to yield 2 sets, each totaling 21 cores. The first set of increment cores was processed for X-ray densitometry. The second set of increment cores was dried in an oven (55 °C) and set aside, anticipating further analyses of these study trees; this second set of increment cores remained available for the current study, and fortuitously, negated the need for disassembly and glue removal.

2.2.3 North Carolina (NC)

The second mainland site was in the Duke Forest (36.00°N, 78.97°W) in Durham, NC. The initial stocking of the naturally regenerated stand, at an elevation of 90 m, is unknown. In 1944, the original stand was harvested, leaving only seed trees that were harvested in 1952, along with a liberation cut in the same year to release the remaining trees; the stand was subsequently thinned in 1980. The mean DBH of 100 trees from a 0.10 ha plot was determined and trees within 1 standard deviation were randomly selected for the collection of a single increment core from each tree sampled. Of the 30 increment cores collected in February 2011, 8 remained available for the current study; the average number of growth rings for these increment cores was 60.

2.3 Climate data

Mean monthly temperature and precipitation data for each site were obtained from the National Oceanic and Atmospheric Administration (<https://www1.ncdc.noaa.gov/pub/>

Table 1 Description of loblolly pine stands located in Hawaii (HI), Mississippi (MS), and North Carolina (NC) including mean plot-level diameter at breast height (DBH), height (H) and basal area (BA). The number of tree rings in the NC stand ranged from 49 to 62. See Samuelson et al. 2013 for further details

Site	Elevation (m)	Planted	Thinnings	DBH (cm)	HT (m)	BA (m ² ha ⁻¹)
HI	1120	1961	None	36.7	30.6	93
MS	50	1961	None	28.8	19.8	18
NC	90	Natural	1980	34.2	31.7	50

data/). These data were previously reported in Samuelson et al. (2013) and are presented herein to provide context to the analytical data from the current study; the mean monthly values are derived from the years used for climate correlation analyses. For the HI site, the precipitation data were measured at a weather station in Olinda, HI (1.8 km from study site; Olinda #1 332, Coop ID: 517041) while the temperature data were measured at a weather station in Kula, HI (3.6 miles from study site; Kula Branch Station 324.5, Coop ID: 515000). On the US mainland, the data for the MS site were measured at a nearby weather station (Saucier Experimental Forest, MS, Coop ID: 227840) while the data for the NC site were obtained from a weather station located in Durham (Coop ID: 312515).

2.4 X-ray densitometry

A subset of a previously collected X-ray densitometry dataset (Samuelson et al. 2013) was reprocessed for the increment cores specifically used for the current study. Briefly, increment core assemblies had been scanned on a Model QTRS-01X (Quintek Measurement Systems, Knoxville, TN, USA) densitometer using a 0.06 mm radial step resolution and a threshold SG value of 0.48 to differentiate latewood from earlywood. The densitometer was calibrated to a green volume and oven-dried mass basis. Samuelson et al. (2010; 2013) reported percent latewood, earlywood SG, latewood SG, and ring SG values weighted by the corresponding ring basal areas to obtain the mean basal-area-weighted wood property values for each tree sampled. Physical/anatomical property values in the current study were not weighted to allow direct comparison to the spectroscopic and wet chemical results that could not be weighted for the milled wood samples. Mean core lengths and standard deviations (in parentheses) for the increment cores used in the current study were as follows: HI, 16.7 (2.3) cm; NC, 14.4 (2.2) cm; MS, 12.2 (1.9) cm.

2.5 Screening by multivariate analysis of mid-IR spectra

Mid-IR spectra of the solvent-extracted and milled (1 mm) wood samples were collected using a Perkin Elmer Spectrum Two FTIR spectrometer equipped with an attenuated total reflectance (ATR) accessory (single reflection diamond, Spectrum Two Universal ATR). Aliquots (about 0.3 g) of each wood sample (each representative of a single wood core) were placed on the ATR diamond window. After the anvil was tightened to a constant pressure, spectra were collected over a spectral region of 4000–600 cm^{-1} , with a spectral resolution of 4 cm^{-1} and 8 scans. The analysis was repeated with a fresh aliquot; a total of 5 spectra

were collected per wood sample. Multivariate analysis of the spectral data was performed using Unscrambler X software (Version 10.4, CAMO, Woodbridge, NJ, USA). All the spectra were first mean-normalized and then averaged to a single spectrum per sample. Multiplicative scatter correction (MSC) was applied to reduce the effect of noise in the spectral data. Principal component analysis (PCA) was carried out with the spectral data to screen for differences and/or groupings between the three sites. Partial least squares (PLS) regression was used to model the wood properties. Models were generated with 2 or 3 principal components using cross-validation and assessed using measures of calibration performance including coefficient of determination (R^2), root mean square error of calibration (RMSEC), and root-mean-square error of cross-validation (RMSECV).

2.6 Wet chemistry analysis

Solvent-extracted wood samples were dried in an oven at 40 °C for at least 3 days to achieve a constant weight before being subjected to a two-step acid hydrolysis following National Renewable Energy Laboratory (NREL) protocols (Sluiter et al. 2012). Filtration of the hydrolysis products afforded the bulk of the lignin as an insoluble residue (Klason lignin) that was quantified gravimetrically. The typically small amount of lignin remaining in the filtrate was estimated spectrophotometrically using a wavelength of 240 nm; values for said acid-soluble lignin ranged from 2.9 to 3.2% on an extractives-free wood basis. The hydrolyzed polysaccharide sugars were determined by high-pressure liquid chromatography using a system (Perkin Elmer) equipped with a Aminex HPX-87P column (BIORAD, 300 mm x 7.8 mm, 9 μm particle size) and a micro-guard Carbo-P guard column (BIORAD). The oven temperature for the Aminex HPX-87P column was held at 85 °C; the mobile phase was DI water at a flow rate of 0.25 mL min^{-1} and the injection volume was 30 μL . The refractive index detector (Perkin Elmer) was set at 50 °C. Sugars quantified were glucose, arabinose, galactose, mannose, and xylose, with the mean values for cellulose being based on the values for glucose for each wood sample and the mean values for hemicelluloses being based on the other 4 sugars for each wood sample. Ash contents were determined by combusting about 1 g of sample in a muffle furnace at 575 °C for 24 h. Mean chemical composition values are all reported on an extractives-free oven-dry weight basis; all wood samples were analyzed in triplicate.

2.7 Statistical analysis

To compare the physical, anatomical, and chemical properties differences between the wood samples from the three

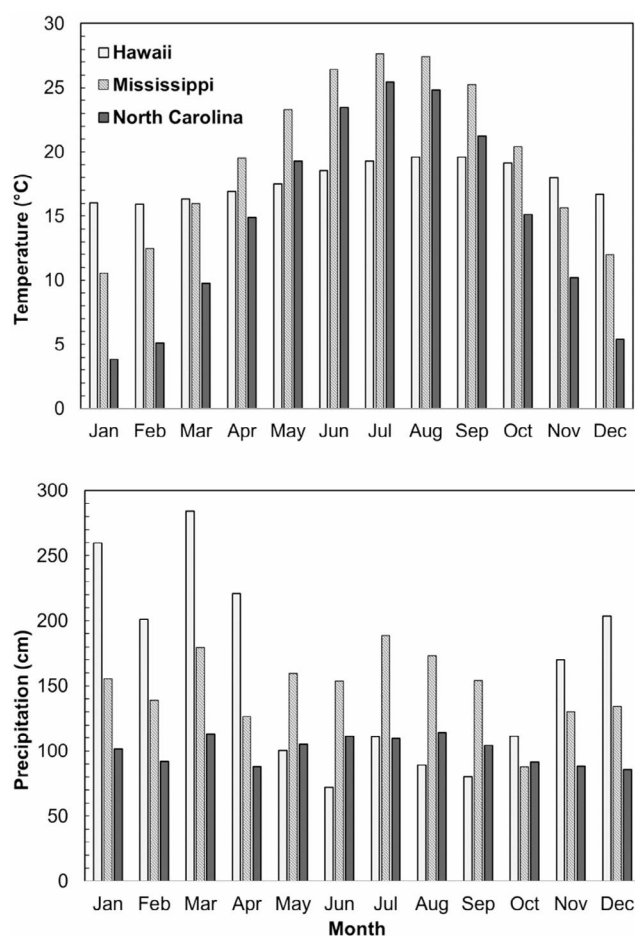


Fig. 1 Mean monthly temperature and precipitation for study sites in Hawaii, Mississippi, and North Carolina

sites, an analysis of variance (ANOVA) method was used in SAS 9.4 (SAS Institute, USA). Presented values are means of sample sets and Fisher's Least Significant Differences (LSD) was employed for mean separation at a significance level of $\alpha=0.05$.

3 Results and discussion

3.1 Climate conditions at study sites

Climate parameters of mean monthly temperature and precipitation are provided in Fig. 1 showing, in general terms,

how the climate conditions differ throughout the year at the HI and US mainland sites. Compared to the mainland sites, the HI site has a narrower mean monthly temperature range (15.9 to 19.6 °C) and less seasonal temperature variation. Mean monthly temperature ranges at the MS and NC sites were 10.5 to 27.6 °C and 3.8 to 25.5 °C, respectively. Taking the monthly temperature extremes for the mainland sites together, the difference between the winter-low temperature and summer-high temperature on the US mainland was more than 6-fold greater than the difference in the temperature extremes for the HI site. Mean monthly precipitation values for the mainland sites covered narrower ranges (NC, 86 to 114 cm; MS 88 to 189 cm) compared to the HI site (72 to 284 cm). Unlike the mainland sites, the HI site shows a very pronounced seasonal precipitation variation with the average precipitation calculated for HI site rainy season (220 cm, November through March) being twice that for the remaining months (110 cm, April through October). Taking the sum of the monthly precipitation values as an indicator of annual precipitation, the calculated annual precipitation for the HI site (190 cm) is about 7% higher than for the MS site (178 cm), and about 57% higher than for the NC site (121 cm). Samuelson et al. (2013) reported the ring width index for loblolly pine to show greater correlation to average monthly precipitation for the HI site and average monthly temperature at the NC site; at the MS site, there was only a correlation to temperature in November. Year-long favorable temperature conditions for the HI site led to nearly year-long radial expansion in loblolly pine, and year-to-year, a pattern of increased annual growth resulting from increased precipitation outside of the normal rainy season.

3.2 Physical properties of increment cores

Physical properties determined for the increment cores used in the current study are shown in Table 2. The observed differences between sites parallel our prior findings (Samuelson et al. 2013), with the current values derived from a subset of the original set of increment cores. The values in Table 2 are not weighted (e.g. corrected using ring basal areas) and therefore are directly representative of the wood substance ultimately subjected to the spectroscopic and the wet chemical analyses (i.e., not serving as a proxy for whole-disk values used to represent whole trees). Results

Table 2 Mean wood physical and anatomical properties determined for loblolly pine increment cores collected in Hawaii (HI), Mississippi (MS), and North Carolina (NC). Standard errors are shown in parentheses and means with different letters are significantly different ($\alpha=0.05$)

Site	Ring SG	Earlywood SG	Latewood SG	Percent latewood	Ring width (mm)
HI	0.447 (0.007) b	0.344 (0.004) b	0.622 (0.005) b	37.2 (1.7) b	3.93 (0.10) a
MS	0.558 (0.007) a	0.366 (0.004) a	0.719 (0.005) a	54.8 (1.7) a	2.98 (0.10) b
NC	0.551 (0.011) a	0.354 (0.006) ab	0.726 (0.009) a	53.1 (2.8) a	2.40 (0.16) c

show that the mean ring SG, latewood SG, and percent latewood values are all significantly lower for the HI site compared to the mainland sites. These results, along with the higher ring width for the HI loblolly pine can be attributed to the aforementioned favorable temperatures throughout the year (Fig. 1) resulting in minimal dormancy (Lanner 1966; Samuelson et al. 2013), thus near year-long radial expansion. Note that while cell formation ceases during winter dormancy on the US mainland, photosynthesis does not (Gough et al. 2004), nor does cell wall thickening for the last formed tracheids (Nix and Villiers 1985). Since the longer growing season at lower latitudes on the US mainland favors greater latewood formation (Jokela et al. 2004), seemingly higher values for percent latewood and ring SG could be rationalized for the MS site over the NC site (Samuelson et al. 2013). We account for the lack of a significant differences in the physical/anatomical wood properties between the MS and NC sites herein to both the use of unweighted values and the unavoidably reduced sample size for the NC site.

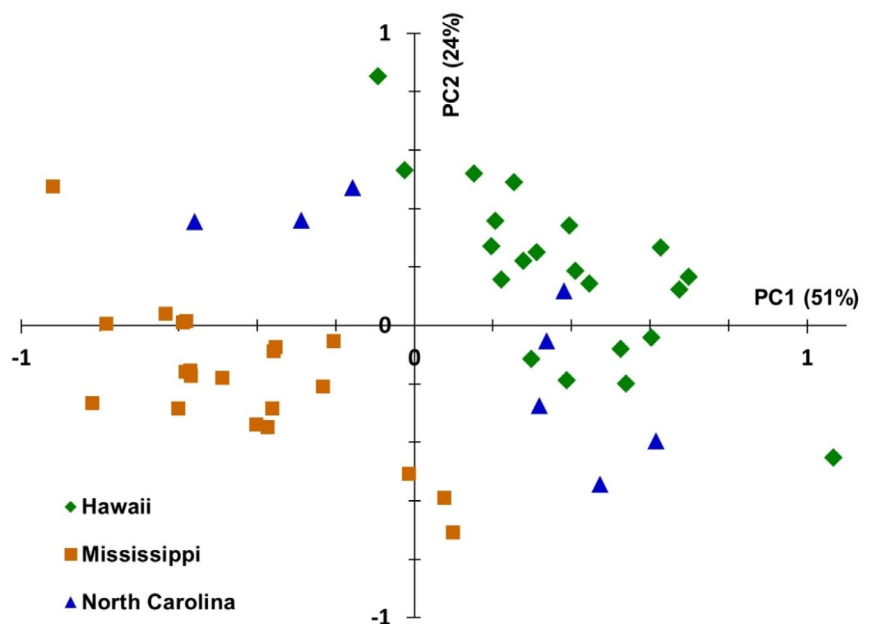
3.3 Qualitative chemical analyses

Principal component analysis (PCA) was performed on the mid-IR spectral data to screen for differences in wood chemistry for the loblolly pine samples from the HI and mainland sites. From the scores plot (Fig. 2), we observe that the MS samples differ from the HI and NC samples by principal component 1 (PC1) which accounts for 51% of the variation and by principal component 2 (PC2) which accounts for 24% of the variation. There is some overlap between the HI and NC sites; the data points for these two sites form a larger cluster apart from the cluster of data points for the MS site.

The PCA results are particularly interesting in that although the data points for the NC site do show some intermediate clustering between the HI and MS site, the data points for NC site do not overlap with those for the MS site.

The PCA loadings plots for PC1 and PC2 highlight the chemical features contributing to the separations along the principal components; wavenumbers with large loadings, both negative and positive, are those that contribute the most to the variation along PC1 and PC2. Figure 3 shows some common broad bands (O–H stretching at $3600–3200\text{ cm}^{-1}$; C–H stretching at $2950–2850\text{ cm}^{-1}$) typical for lignocellulosic materials within the mid-IR range. In the fingerprint region ($1800–650\text{ cm}^{-1}$), there are well-defined lignin signals such as the bands at 1603 cm^{-1} and 1511 cm^{-1} which can be assigned to aromatic skeletal vibrations of lignin. The band observed at 1659 cm^{-1} is usually assigned to conjugated C=O stretching vibration, 1307 cm^{-1} to CH_2 wagging, 1235 cm^{-1} to OH vibration in guaiacyl ring, C–C, C–O, and C=O stretches in lignin, 1107 cm^{-1} to C–O–C stretching in cellulose and hemicellulose, 1031 cm^{-1} to C–O stretching in primary alcohols in cellulose, 951 cm^{-1} to O–H out of plane deformation in carboxylic acid, and 877 cm^{-1} to glycosidic linkage. The positive signals at 1603, 1511 and 1235 cm^{-1} for PC1 and PC2 in loadings plots, along with the data point pattern shown in the PCA scores plot, are indicative of higher lignin contents for the HI site samples relative to those for the MS site. Given the interrelationships between wood chemistry and the other wood properties, samples exhibiting similar wood physical/anatomical properties might be presumed to have similar spectral features, and thereby group together in the PCA scores plot; however, this was not the case with the MS and NC samples that share similar physical/anatomical properties (e.g., ring

Fig. 2 Principal component analysis (PCA) scores plot from the multivariate analysis of mid-IR spectra for loblolly pine increment cores collected in Hawaii, Mississippi, and North Carolina



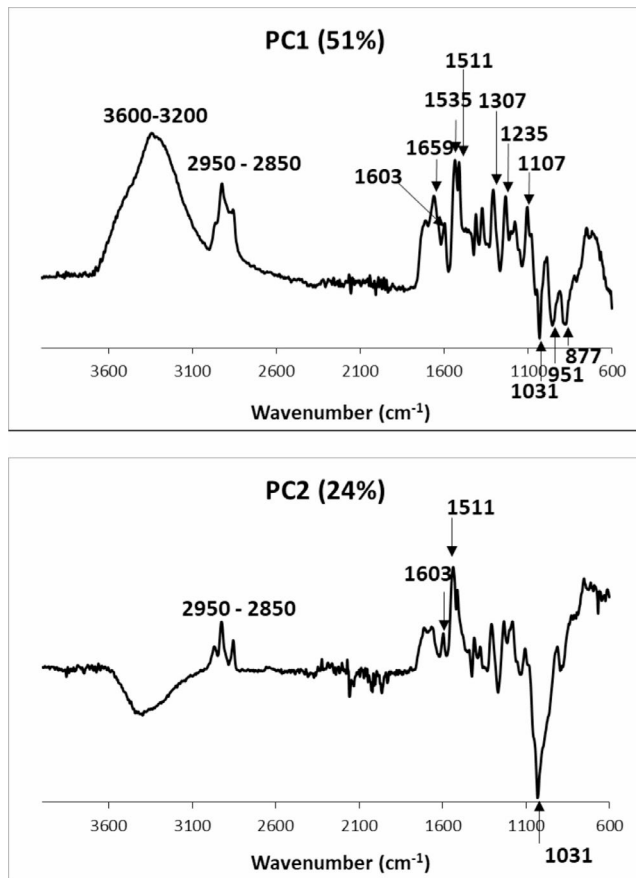


Fig. 3 PC1 and PC2 loadings plots associated with the PCA scores plot shown in Fig. 2

SG, percent latewood), but seemingly have different wood chemistries as evidenced by the PCA scores plot (Fig. 2). Altogether, these results demonstrated detectable differences in wood chemistry, thereby justifying the time-consuming wet chemical analyses to quantify the apparent wood chemistry differences.

Another multivariate analysis technique, partial least squares (PLS) regression, has been widely applied to predict wood chemical composition using models based on how the spectral data vary with corresponding wet chemical data (Kelley et al. 2004; Sykes et al. 2005; Jones et al. 2006; Aspinwall et al. 2010; Acquah et al. 2015; Fahey et al. 2017). Extremely useful predictive models have also been obtained for wood physical, anatomical, and mechanical properties based on how the spectral data vary with corresponding property data (Kelley et al. 2004; Schimleck et al. 2005; Tsuchikawa and Kobori 2015; Dahlen et al. 2021). It is outside the scope of the study to develop robust PLS models for predictions of wood physical/chemical properties, let alone there is no pressing need for said models; however, it was of interest to see if variation in the spectral data among the samples from all sites would manifest in relationships to any of the wood physical properties. Specifically, given the higher lignin (lower cellulose) contents in the earlywood relative to the latewood (Gladstone et al. 1970; Saka and Thomas 1982; Gindl 2001; Fredriksson et al. 2018), we would expect stronger PLS models for those wood physical properties directly influenced by the relative proportions of earlywood and latewood. The strongest PLS model was for ring SG. Data points for the predicted vs. measured values for the ring SG model are shown in Fig. 4. Identifying each data point by site, we see an uneven scatter for each sampling site about the equivalence line; the strength in the PLS

Fig. 4 PLS predicted vs. measured plot for ring SG for calibration (empty shapes) and cross-validation (solid shapes) indicating study sites in Hawaii (HI), Mississippi (MS), and North Carolina (NC).

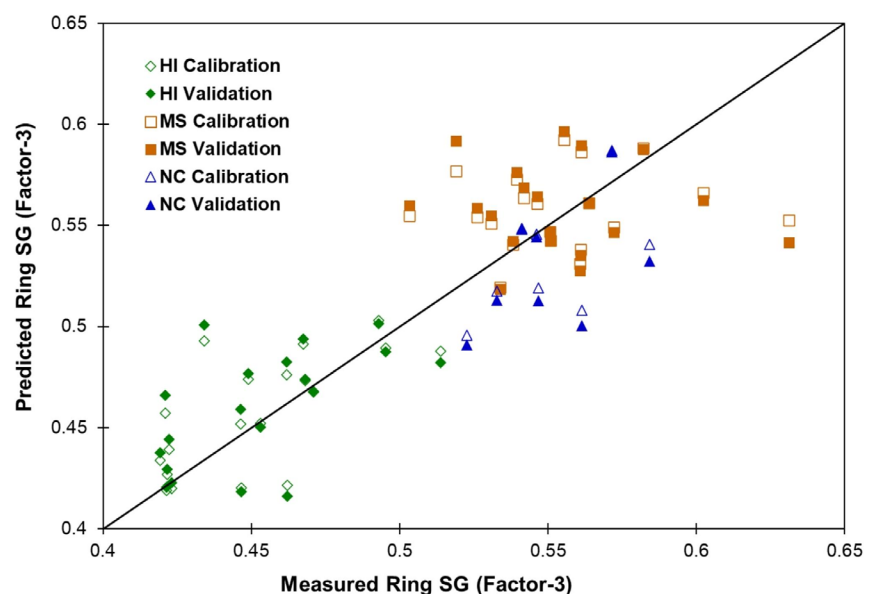


Table 3 PLS regression statistics for wood physical and anatomical properties determined for pooled data for all loblolly pine increment cores. Corresponding plots for ring SG and latewood SG are provided in Figs. 4 and 5

Wood property	Factors	Calibration		Cross-validation	
		RMSEC	R ²	RMSECV	R ²
Ring SG	3	0.031	0.76	0.036	0.68
Earlywood SG	2	0.015	0.40	0.017	0.28
Latewood SG	3	0.028	0.74	0.033	0.65
Percent latewood	3	6.9%	0.63	8.2%	0.50
Ring width	3	0.50 mm	0.53	0.63 mm	0.63

models comes from the significantly lower ring SG values for the exotic location.

The PLS regressions gave summary statistics (Table 3) with reasonable coefficients of determination (R²); the root-mean-square error of calibration (RMSEC) is determined from the residuals of the calibration while the root-mean-square error of cross-validation (RMSECV) is determined from the residuals during each cross-validation phase. The

next strongest model was for latewood SG (Fig. 5), which is not obviously influenced by the relative proportions of earlywood and latewood. This suggests that there is significant variation in the chemistry of the latewood itself between the HI and mainland sites to allow the prediction of latewood SG.

Most of the same FTIR bands (1535, 1303, 1260, 1100–1070, 951, 887, and 811 cm⁻¹) were responsible for the correlation for the ring SG and latewood SG, as per the regression coefficients given in Fig. 6, indicating that the same chemical features impact these two properties.

3.4 Quantitative chemical analyses

3.4.1 Lignin

Using established wet chemistry procedures (Sluiter et al. 2012), we provide here the quantitative data allowing a direct comparison of wood chemical composition for

Fig. 5 PLS predicted vs. measured plot for latewood SG for calibration (empty shapes) and cross-validation (solid shapes) indicating study sites in Hawaii (HI), Mississippi (MS), and North Carolina (NC).

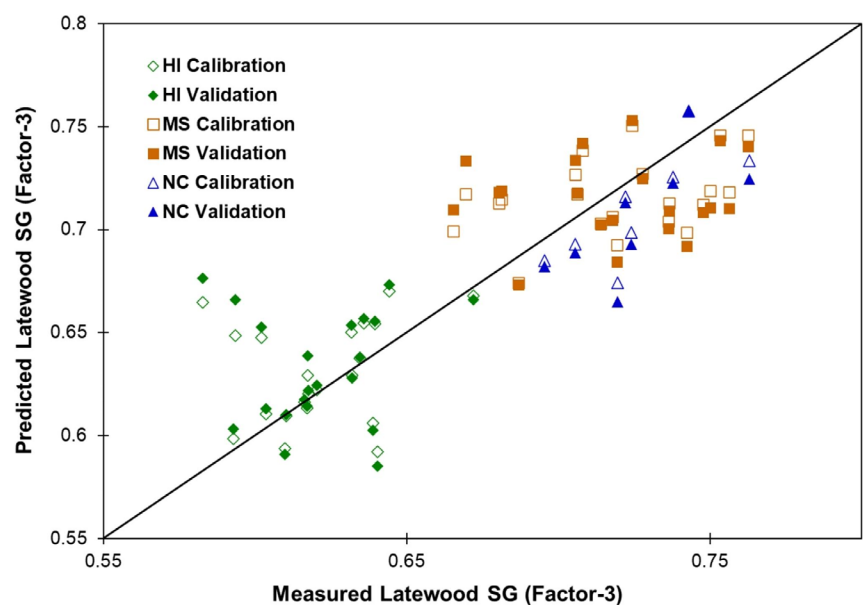


Fig. 6 PLS regression coefficients plots of ring SG (A) and latewood SG (B) prediction models

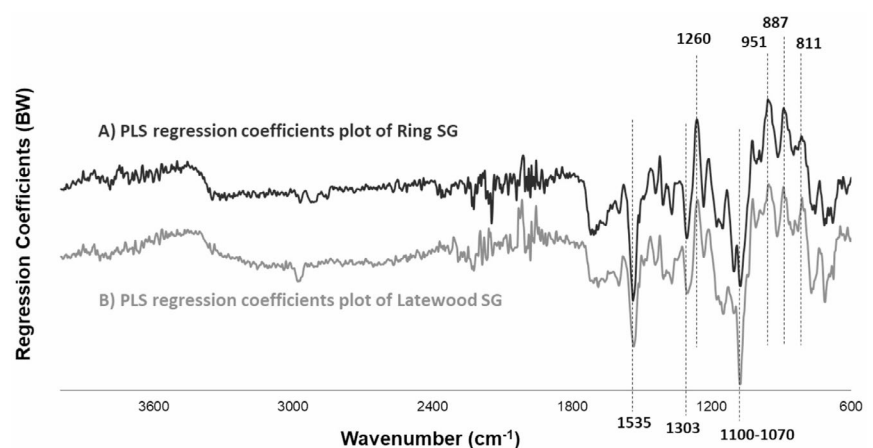


Table 4 Wood chemical compositions determined for loblolly pine increment cores collected in Hawaii (HI), Mississippi (MS), and North Carolina (NC). Data are mean percentages of extractive-free dry wood; standard errors are shown in parentheses and means with different letters are significantly different ($\alpha=0.05$)

Site	Lignin (%)	Cellulose (%)	Hemicelluloses (%)	Ash (%)
HI	33.9 (0.2) a	37.2 (0.3) b	20.2 (0.2) b	0.36 (0.02) a
MS	30.1 (0.2) b	40.6 (0.3) a	21.7 (0.2) a	0.39 (0.02) a
NC	34.5 (0.3) a	37.5 (0.4) b	20.4 (0.3) b	0.22 (0.02) b

Table 5 Polysaccharide sugar compositions determined for loblolly pine increment cores collected in Hawaii (HI), Mississippi (MS), and North Carolina (NC). Data are mean percentages of extractive-free dry wood; standard errors are shown in parentheses and means with different letters are significantly different ($\alpha=0.05$)

Site	Arabinose (%)	Galactose (%)	Glucose (%)	Mannose (%)	Xylose (%)	All sugars (%)
HI	1.3 (0.0) a	4.7 (0.1) a	37.2 (0.3) b	8.6 (0.1) c	5.5 (0.1) b	57.3 (0.3) b
MS	0.9 (0.0) b	4.3 (0.1) a	40.6 (0.3) a	10.7 (0.1) a	5.8 (0.1) a	62.3 (0.3) a
NC	1.0 (0.0) b	3.8 (0.2) b	37.5 (0.4) b	9.6 (0.2) b	6.1 (0.2) a	58.0 (0.5) b

loblolly pine grown at an exotic location (HI) to those on the US mainland (MS and NC). Consistent with the groupings in the PCA scores plot and the signals in the PCA loadings plots, the mean acid insoluble (Klason) lignin content for the MS site was measurably lower, at least 4% points less than the lignin content for the HI and NC sites (Table 4). Making some between-study comparisons with caution, we can state that the acid-insoluble lignin content (33.9%, extractives-free basis) for our exotic location (HI) is in line with the lignin content reported for loblolly pine wood (dry matter or extractives-free basis) from trees taken at various sites in Brazil: 26.18% (Vivian et al. 2015), 26.29 to 29.37% (Rigatto et al. 2004), 25.21% (Bonfatti Júnior et al. 2019), and finally, 27.09 to 34.70% (Pereira and Tomaselli 2004). As noted above for the mainland sites, those further south in the natural range have higher latewood formation than their more northern counterparts (Jokela et al. 2004; Samuelson et al. 2013). The lower lignin content for pine latewood, relative to the corresponding earlywood (see Gladstone et al. 1970; Saka and Thomas 1982; Gindl 2001; Fredriksson et al. 2018), likely manifests in the lower mean lignin content for the MS samples compared to the NC samples.

Given that the percent latewood for the HI site is 16% points lower than that for the NC site (Table 2), one might expect that HI samples to have a significantly higher lignin content than that for the NC samples; however, this is not the case with our data (Table 4). The lack of a significant difference in lignin content between the HI and NC sites seemingly suggests that the lignin content for loblolly pine growing at an exotic location may be the product of other factors (e.g., metabolism, anatomy) and not be solely related to the proportion of latewood formed. The PLS result above

for latewood SG may be indicative of chemical differences in the latewood itself between the HI and the mainland sites.

In a prior study (Eberhardt and Samuelson 2022), it was shown that successive years of drought following successive years of above-normal precipitation impacted wood chemistry to the extent that there were differences in the lignin contents for slash pine (*Pinus elliotii*) and longleaf (*Pinus palustris*) pine growth rings; however, for loblolly pine, identical lignin contents were obtained. This finding for loblolly pine was rationalized in part to the highly adaptable nature of the species to a wide range of environments (Schultz 1999). Therefore, we believe our wet chemistry findings herein to be more influenced by the typical/sustained climate conditions (Fig. 1) at our 3 sampling sites rather than any sporadic climate perturbations (e.g. periodic droughts) that may have occurred at one or more of the sampling sites.

3.4.2 Polysaccharide sugars

The mean value for all sugars recovered for the MS site samples is more than 4% points higher than for either the HI and NC sites; the mean value for glucose is about 3% points higher (Table 5). Using the glucose values directly as a proxy for cellulose, our results suggest that the conditions at the MS site favor the production of wood with an approximately 3% higher cellulose content (Table 4). We can attribute this result to the higher cellulose content of the latewood relative to the earlywood (Gladstone et al. 1970; Saka and Thomas 1982; Gindl 2001; Fredriksson et al. 2018). At this juncture, it should be noted that the predominant hemicelluloses in most softwoods are the galactoglucmannans, comprised of a backbone of glucose and mannose

with branches of galactose (Jones and Painter 1957, 1959; Rowell et al. 2013). While it does not impact our comparisons, we acknowledge some research groups may use corrections to account for the glucose estimated to be derived from the acid hydrolysis of the galactoglucomannans (see Marziales et al. 2008); herein, the total hemicellulose content (Table 4) is taken as the sum of the percentages for arabinose, galactose, mannose, and xylose.

The other main group of softwood hemicelluloses, the arabinoglucuronoxylans, are accounted for by the relatively low percentages for arabinose and xylose. It is interesting to note that values for these two sugars are significantly different between the HI site and both mainland sites. Whereas it would appear we have patterns for the lignin, cellulose, and total hemicelluloses that set apart the MS site apart from the HI and NC sites, there may be differences in the hemicelluloses themselves that set the HI site apart from the mainland sites. The results herein are intriguing in themselves and make the case for further work to better understand the wood chemistry differences for loblolly pine grown on exotic sites vs. those within the native range on the US mainland.

3.4.3 Ash content

Inorganic compounds represent a minor group of constituents in wood that are commonly quantified by the determination of the ash content, a value that is highly relevant to studies on the thermochemical conversion of woody biomass for its fuel value, or other biofuel applications (e.g., gasification, pyrolysis). For example, simulating higher ash contents by the impregnation of loblolly pine wood with potassium carbonate resulted in lower bio-oil yields by pyrolysis (Kim et al. 2021); the yields of bio-oil at 0.5% and 1.5% ash content (dry wt. basis) were 45.7% and 29.9%, respectively. The ash contents in the current study ranged from 0.22 to 0.39% which is consistent with values reported in the literature for loblolly pine (Owen et al. 2015; Acquah et al. 2017; McMillin 1969; Zicherman and Thomas 1972). The ash content for the NC location is significantly lower than the other two locations (Table 4). McMillin (1969) provides the most detailed analysis of the variability in ash content in loblolly pine, showing the ash content of the juvenile wood (0.42%) to be significantly higher than the mature (outer) wood (0.38%) and the ash content of the latewood (0.39%) to be significantly lower than the earlywood (0.43%).

Comparing the ash contents between the 3 sites in the current study did not always show consistent patterns with our wood physical and anatomical property data. The HI site had a significantly lower percent latewood value than the NC site, and the ash content for the HI site was indeed significantly higher than for the NC site; this result can

be rationalized when taking into consideration the results reported by McMillin (1969) for earlywood and latewood ash contents. Undoubtedly, the volcanic soils of Hawaii would be expected have different mineral availabilities than mainland soils and it is reasonable to assume that this would impact the inorganic constituents in the wood. As for the two mainland sites (MS and NC), they had similar wood physical and anatomical properties, but significantly different ash contents, which cannot be rationalized by taking into account the higher ash content of the earlywood shown by McMillin (1969).

4 Conclusion

Our results demonstrate that the lignin content for loblolly pine growing at an exotic site cannot be assumed to be similar to loblolly pine from a US mainland site (MS) also sharing favorable temperatures over a longer growing season, or for that matter, solely attributable to the degree of latewood formation that manifests in wood physical properties such as ring SG. The significantly lower lignin content for loblolly pine wood from the MS site was offset by significantly higher glucose and mannose contents, and thereby reflecting alterations to the relative contents of both cellulose and the hemicelluloses. It is acknowledged that our efforts to assess how climate conditions may impact wood chemistry in loblolly pine may be confounded by other influencing factors (e.g., genetics, soils, management history); however, herein we show that aside from differences in wood physical/anatomical properties shown for HI-grown loblolly pine, differences in wood chemistry could be demonstrated.

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Declarations

Conflict of interest There is no conflict of interest.

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