

Rapid on-site Assessment of Wood Chips Moisture and Lignin for Kraft Pulping Digesters Using Portable NIR Spectroscopy

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Rapid on-site assessment of wood quality can significantly enhance decision-making and operational efficiency in the pulp and paper industry. Spectroscopic techniques, including near-infrared (NIR) spectroscopy, can be used to characterise wood, but there is a need for development to use these methods in the field, especially for optimising the continuous kraft pulping process. This study focused on using a portable NIR spectrometer to measure lignin and moisture content in Nordic softwood chips, while also distinguishing between bound and free water, with particular focus on challenges related to field-based measurements and process-relevant information. Partial Least Square Regression (PLSR) effectively reduced data dimensionality and simultaneously performed the regression. The performance of portable NIR model was found to be comparable with models based on data from a traditional benchtop NIR. However, several portable NIR spectra need to be acquired and averaged to achieve predictive capability. The results confirm that robustness of benchtop systems for accurate moisture determination. Portable NIR may be useful at kraft pulp mills during disturbance situations, when rapid information about chip quality is necessary.

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INTRODUCTION

For chemical pulping operations, accurate and rapid determination of moisture and lignin in wood chips is beneficial, or even essential, as the wood chip composition directly affects digester performance, steam and liquor balances, and ultimately pulp quality. It has been suggested that the most important chip quality properties to monitor at modern kraft pulp mills are moisture content and dry bulk density (Pietilä *et al.* 2015; Liang *et al.* 2019). Changes in the moisture content of the feedstock in a fibre line can cause process disturbances and downstream runnability problems. Further, the amounts of shives and screening rejects in the resulting brown stock pulp also increase (Germgård *et al.* 2024). In addition, variations in lignin content influence the chemical demand during cooking is directly reflected in the kappa number of the resulting pulp (Gullichsen and Fogelholm 2000; Gellerstedt 2009). This underscores the importance of fast and reliable measurement techniques for monitoring key chip properties and supporting stable and efficient kraft pulping operations.

In kraft pulping, NIR spectroscopy has proven useful in assessing the quality of wood chips. Early applications of NIR to wood date back to the late 1980s, when the technique was first explored for estimating moisture content, density, and chemical composition of solid wood materials (Birkett and Gambino 1989; Hoffmeyer and Pedersen 1995). Since then, NIR spectroscopy has been extensively developed within wood science, with numerous laboratory studies demonstrating its ability to predict lignin, cellulose, and moisture content under controlled conditions (Schimleck *et al.* 2001; Schwanninger *et al.* 2011). In parallel, this technique has gradually been translated from laboratory analysis to industrial applications, including at-line and on-line monitoring systems in forest products and pulp and paper industries (Mora *et al.* 2011; Tsuchikawa and Kobori 2015).

For building a reliable NIR model, it is necessary to calibrate NIR-based results with composition data determined by other experimental methods. Several techniques have been applied to determine wood chip moisture content. Oven drying at 105 °C remains the standard reference method, but it is destructive and time-consuming, such that it is unsuitable for real-time monitoring (Rahman *et al.* 2024). Alternative methods include electrical resistance and capacitance sensors (Dietsch *et al.* 2015), as well as microwave transmission systems (Nyström and Dahlquist 2004). For lignin content, wet-chemical methods such as Klason lignin are widely used as reference technique (SS-ISO 21436 (2021)), but they are time-consuming and sensitive to variations in the analytical procedure. Alternative approaches include thermogravimetric analysis (TGA) and spectroscopic techniques such as FT-IR, which provide faster measurements but require calibration (Carrier *et al.* 2011). Each technique carries its own calibration requirements and sensitivity to material variability.

In previous research, benchtop NIR models have been used to predict lignin contents for Nordic species under industrial-like conditions when constrained by species (Klecina *et al.* 2025). Elsewhere, on-line VIS-NIR was used to monitor chip quality on-site for several months at a fixed position, providing data on feedstock moisture, chemical composition and brightness, alongside temperature measurements, although the work was described as exploratory (Hans and Allison 2021). Together, these studies have established NIR spectroscopy as a mature and versatile technique for wood characterization in the laboratory. Robust, field-based and process-integrated measurements remain under development. In addition to quantitative prediction of wood properties, NIR spectroscopy has shown potential for qualitative classification of wood species. Sohi *et al.* 2017 demonstrated 100% correct classification when separating sub-alpine fir from spruce-pine lumber using PLS discriminant analysis on NIR spectra acquired from freshly cut specimens. Espinoza *et al.* 2012 showed that NIR can distinguish between pure pine species and their hybrids, although Cooper *et al.* 2011 noted that confounding variables such as moisture content and surface characteristics may reduce classification robustness. More recently, portable NIR instruments combined with advanced chemometric, or machine learning methods have achieved classification accuracies above 95% for closely related softwood species (Chen *et al.* 2026).

Moreover, the limited flexibility of on-line systems in sampling position motivates investigation of portable NIR devices, which enable operator-driven measurements at multiple locations. To understand the capabilities demonstrated in these applications and the challenges associated with extending NIR measurements from laboratory to field conditions and flexible positioning, it is necessary to consider the physical basis of NIR spectroscopy.

NIR spectra are governed by overtone and combination bands of fundamental vibrations, with X-H (O-H, C-H, N-H) groups dominating the signal (Gedde *et al.* 2021). This leads to broad, overlapping features and limits chemical resolution (Bower and Maddams 1989). Characteristic NIR absorption regions for water, cellulose and lignin have been reported (Schwanninger *et al.* 2011), whereas lower-fraction constituents such as hemicelluloses and extractives are harder to distinguish due to their weaker contributions and spectral similarity to dominating wood polymers. Consequently, multivariate data analysis and chemometric modelling are essential for extracting chemically relevant information from NIR spectra.

Quantitative analysis typically uses partial least squares regression (PLSR) to extract covarying spectral patterns linked to target properties while down-weighting confounders (Workman and Weyer 2012; Amaral *et al.* 2020). Because the O-H dominates the NIR spectra of wood, much of spectral variance reflects moisture. As a consequence, model performance hinges on the state of water in the chip. However, moisture experiences different chemical environments in wood, which can affect NIR spectral response.

Moisture in wood is present both as free water in the cell lumina and bound water held in the cell wall by hydrogen bonding to hydroxyl groups in cellulose and hemicellulose (Rowell 2012). Water binds primarily at polymer hydroxyl sites—most in hemicellulose, then cellulose, then lignin—and many cellulose OH groups are internally bounded with microfibrils, reducing their accessibility for water binding (Christensen and Kelsey 1959; Simpson 1980; Berthold *et al.* 1996; O’Sullivan 1997). Because free and bound water reside in distinct chemical environments within wood, they give rise to different O-H vibrational responses and therefore contribute differently to the NIR spectra, meaning that regression models may benefit from treating these moisture fractions as separate constituents (Yang *et al.* 2014; Ma *et al.* 2020; Moll *et al.* 2022).

For kraft pulping, accurate and rapid determination of chip moisture is particularly valuable, as moisture directly affects digester performance, steam and liquor balances, and ultimately pulp quality (Pietilä *et al.* 2015; Germgård *et al.* 2024). Portable NIR devices could provide operators with rapid and flexible on-site sampling to optimize cooking conditions and prevent process disturbances. Recent developments demonstrate the use of portable/handheld NIR for on-site prediction of moisture, ash, and gross calorific value for bioenergy wood chip fuels, indicating potential for wider industrial use (Tsuchikawa and Kobori 2015; Toscano *et al.* 2022; Leoni *et al.* 2024). At the same time, the physical limitations of portable NIR spectroscopy, including restricted spectral range and a less controlled, manual scanning procedure mean that it remains an open question whether more detailed on-site analysis of wood composition beyond moisture content can be reliably achieved.

Given that benchtop NIR, and to some extent online NIR installations, have already proven effective for determining moisture content and lignin in pulping process, there is a need for comparative studies that systematically evaluate whether portable NIR instruments can deliver equivalent information with sufficient accuracy and robustness to support kraft pulp production (Hans and Allison 2021; Leblon *et al.* 2013; Amaral *et al.* 2020). In this study, the potential of portable NIR spectroscopy for kraft pulping applications was investigated by focusing on Norway spruce (*Picea abies*) and Scots pine (*Pinus sylvestris*). To reflect realistic mill conditions, both roundwood and sawmill residual chips were analysed across a range of moisture contents. NIR spectra were collected using both a portable NIR device and a conventional benchtop spectrometer, while laboratory analyses were performed to determine the chemical composition of the chips. Moreover,

Partial least squares regression (PLSR) models were developed and then compared to evaluate the predictive performance of the two instrument types, with the aim of assessing whether portable NIR can provide robust predictions of chip properties relevant for kraft pulp production.

EXPERIMENTAL

Materials

This study evaluated the potential of portable NIR spectroscopy for predicting moisture and lignin content in Nordic spruce (*Picea abies*) and pine (*Pinus sylvestris*), under conditions representative of industrial environments, using a benchtop NIR system as a reference.

Wood samples and their preparation

Six well-defined batches of spruce and pine wood chips were used in this study: spruce and pine chips from industry suppliers, and felled spruce and pine from Timrå region, Sweden (Table 1). Sawmill chips were obtained from the Kastet sawmill and Heby sawmill (Setra, Sweden), while roundwood chips were obtained from the Gävle Mill (Billerud, Sweden). Each batch of wood chips was homogenized through mixing.

The felled spruce and pine were approximately 30 years old, with breast high diameter of around 15 cm. They were harvested from a south-facing slope in a former pasture in Timrå municipality. Nothing atypical was observed in the annual rings. The harvesting took place during winter when the ground was frozen, and the logs were stored in piles until processing the following month of June, in accordance with local pulp industry standards. The logs were then chipped on a laboratory disc chipper (Hellström *et al.* 2011) with rotational speed 509 rpm, feed speed 14.2 m·min⁻¹, knife thickness 1 mm, and knife angle 30°. An overview of the sample preparation and measurement procedure is provided in Fig. 1. From the six batches, subsamples were prepared by drying at 105 °C for varying durations to achieve different target moisture, ranging from 10 to 50 wt%. In total, 54 subsamples were extracted for benchtop NIR analysis and 36 for portable NIR analysis. Subsamples were stored under vacuum seal between moisture determination and NIR spectrometry for four days. The effect of storage was evaluated for the spruce batch from Timrå with an initial moisture content of 36%, which showed a 0.85 percentage point loss in moisture during vacuum storage.

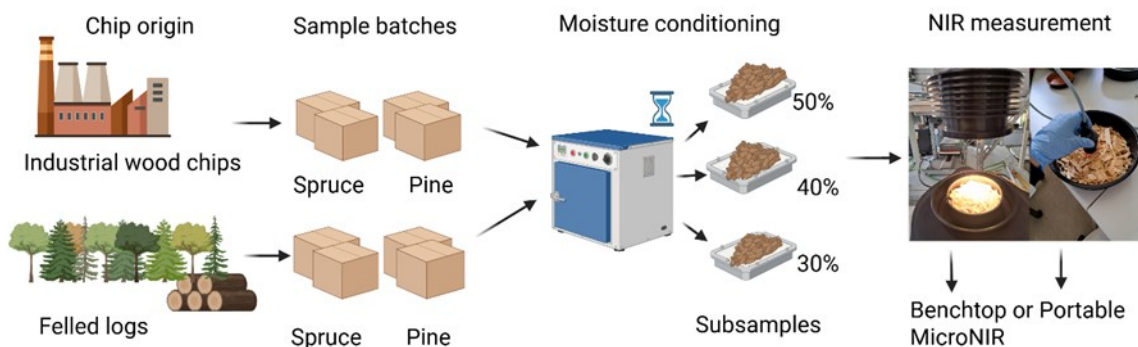


Fig. 1. Schematic explanation of sample preparation. Each box represents a batch of wood chips, while the trays represent subsamples

Klason lignin

Test portions were taken from each batch for Klason lignin analysis according to SS-ISO 21436 (2021). Each subsample was treated with 3 mL of 72 % sulfuric acid at 30 °C, diluted with 84 mL of deionized water, and autoclaved at 120 °C. The residue after hydrolysis was filtered off, dried, and weighed, and referred to as acid-insoluble lignin (AIL), or Klason lignin. A small fraction of lignin was dissolved during acid hydrolysis. The concentration of this acid-soluble lignin (ASL) was determined by measuring the absorbance of the filtrate at 205 nm. The total lignin content was determined as the sum of AIL and ASL (Table 1).

Table 1. Klason Lignin from the Six Different Batches

English name	Source	ASL (%)	AIL (%)	Total Lignin (%)
Spruce (Heby, Setra)	Sawmill	0.6	26.2	26.8
Spruce (Gävle Mill)	Roundwood	0.6	26.3	26.9
Felled Spruce (Timrå)	Roundwood	0.6	28.00	28.6
Pine (Kastet, Gävle)	Sawmill	0.6	26.2	26.8
Pine (Gävle Mill)	Roundwood	0.5	26.9	27.4
Felled Pine (Timrå)	Sawmill	0.5	28.8	29.3

Note: Repeated measurement on a control sample gave a standard deviation of 0.378%.

Spectral Data Acquisition

Near-infrared (NIR) spectra of wood-chip samples were acquired using both portable NIR and a benchtop NIR spectrometer, as described below.

Portable NIR

A handheld near-infrared spectrophotometer, Portable NIR 1700 ES (Viavi Solutions, Scottsdale, AZ, USA), was used for spectral data acquisition. This portable device spans a range of 950 to 1650 nm, with a spectral sampling interval of 6.2 nm and spectral bandwidth of less than 1.25% of the centre wavelength. Thus, its spectral range covers the first overtones and combination bands of X-H bond vibration.

Unlike the benchtop system, which records a continuous scan line, the portable device acquires point measurements. To represent each sample adequately, the probe was placed at 40 randomly selected locations, with chip remixing between scans. This produced 1440 spectra across 36 samples. For modelling, the scans from each sample were averaged to yield one spectrum per sample. Remixing was used to limit local heating and evaporation caused by the illumination. After each spectral acquisition, the wood chips were manually redistributed and repositioned to expose new surfaces to the probe, thereby reducing repeated illumination of the same area and minimizing localized drying effects. The spectra from the Portable NIR are plotted in Fig. 3c.

Benchtop NIR

Benchtop NIR spectra were obtained using an FT-NIR spectral analyser (MATRIX-F, Bruker Optics, Germany) operating with a contactless illumination detection head (Q410/A, Bruker Optics, Germany). The head was equipped with 2 tungsten sources which illuminated the sample during data acquisition. Diffuse reflectance was collected and relayed to the spectrometer *via* optical fibre. The spectrometer is equipped with a diode InGaAs detector. The scanning spot size was approximately ø10 mm. The head was fixed at a focal distance of 17 cm from the sample, which was placed on a turntable. The optical

axis was offset from the axis of rotation such that the illuminated spot moved at approximately $0.2 \text{ m}\cdot\text{s}^{-1}$. NIR absorbance spectra were collected over a range of 833 to 2500 nm with a wavenumber resolution of 8 cm^{-1} and interferometer mirror velocity of 10 kHz.

Benchtop measurements were done in triplicates per subsample. The benchtop instrument records an average across its scan line rather than separate spot readings as in the portable system, so additional scans would not significantly add information. Chips were remixed between scans, yielding a total of 162 spectra (Fig. 3a).

Signal-to-noise comparison

The stability of portable NIR measurements relative to a benchtop reference system was evaluated through signal-to-noise-ratio (SNR) testing. For each wavelength λ , the SNR was defined as follows,

$$\text{SNR} = \frac{\mu(\lambda)}{\sigma(\lambda)} \quad (1)$$

where $\mu(\lambda)$ is the average spectral intensity obtained from repeated measurements, and $\sigma(\lambda)$ is the standard deviation across those measurements.

Multivariate data analysis

The analysis of the spectra and composition followed a structured workflow designed to ensure consistent preprocessing, reproducible measurements, and compatibility across modelling steps (Fig. 2).

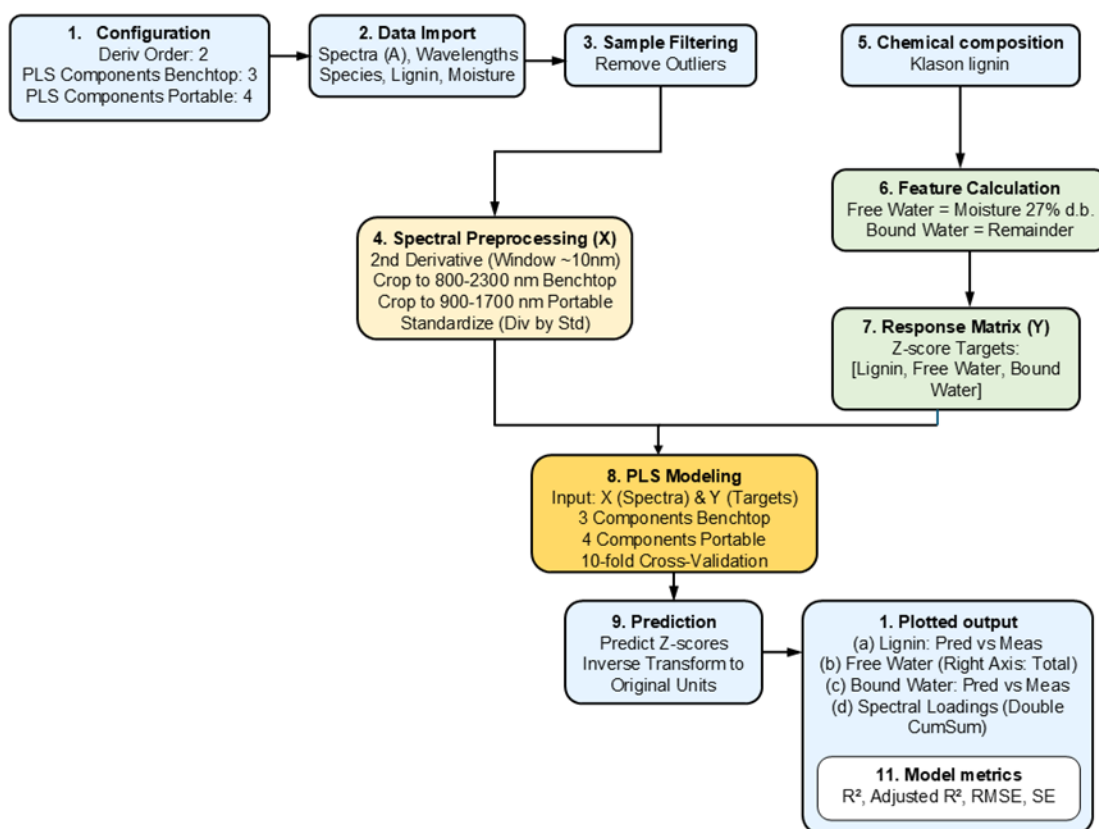


Fig. 2. Workflow for spectral data acquisition and preprocessing used for NIR modelling

The raw NIR spectra were first imported together with sample information such as species, lignin content, and moisture measurements. After filtering the dataset to remove outliers, the spectra were preprocessed using a second-derivative Savitzky–Golay filter with an effective window width of approximately 10 nm (Savitzky and Golay 1964). A second-derivative Savitzky–Golay filter was applied to reduce baseline effects and enhance subtle spectral features associated with overlapping absorptions, thereby improving the extraction of chemically relevant information for subsequent multivariate modelling (Zimmermann and Kohler 2013). The preprocessed spectra were standardized and used as predictors for the wood chip composition.

Spectral inspection and quality assessment

For each species and instrument, mean spectra and standard deviations were calculated, and difference spectra (Pine minus Spruce) were derived to highlight species-specific spectral features. The results were visualized in a four-panel figure (Fig. 3), showing mean spectra with variability bands (left) and corresponding difference spectra (right), using consistent colour coding and wavelength-band shading to enable clear comparison between instruments. The acquired NIR spectra are plotted in Fig. 3, with orange representing spruce and blue representing pine. The portable MicroNIR and benchtop NIR absorbance spectra show peak structures in the overlapping range (Fig. 3a and 3c), whereas difference spectra are qualitatively different (Fig. 3b and 3d).

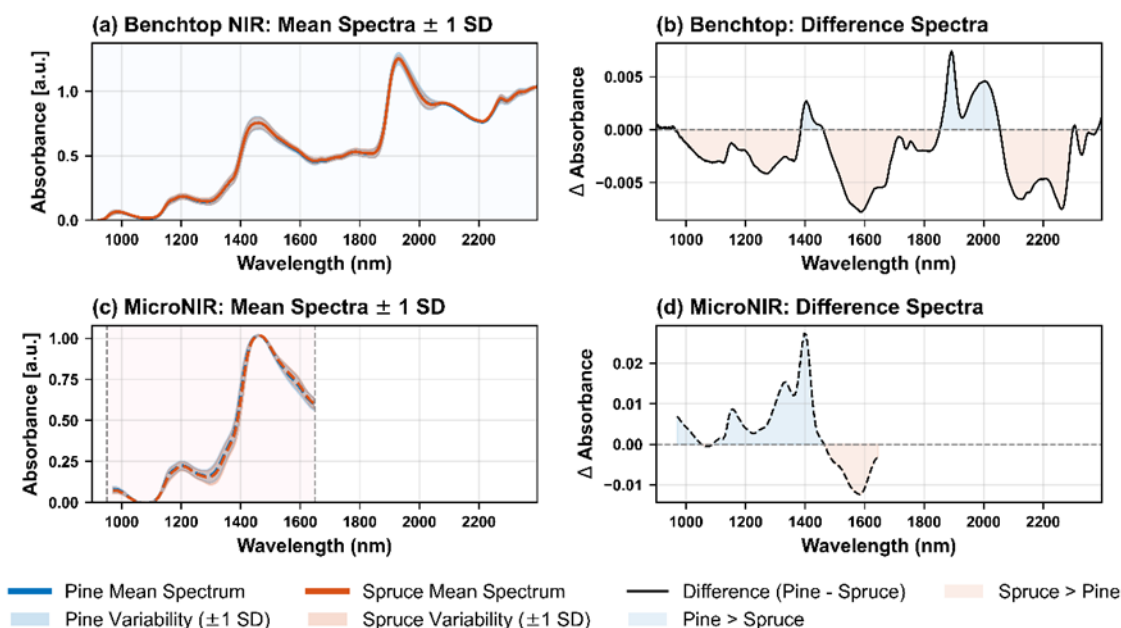


Fig. 3. Acquired spectra from Portable NIR and Benchtop NIR. (a) Mean Benchtop NIR absorbance spectra for Pine (blue) and Spruce (orange), with shaded areas representing ± 1 SD. (b) Difference spectrum (Pine minus Spruce) derived from Benchtop data. (c) Mean portable MicroNIR absorbance spectra (± 1 SD). (d) Difference spectrum derived from portable MicroNIR data.

Identification of deviating spectra

All raw NIR spectra, obtained from both the portable MicroNIR and benchtop instruments, were first visually compared to identify spectra that deviated strongly in intensity or shape from the main spectral clusters (Fig. 4). The following procedure was used to identify discrepant spectra. To correct baseline variations, a Savitzky–Golay (SG)

filter with a 200 nm window and a second-order polynomial was applied to each spectrum (Savitzky and Golay 1964). This preprocessing step effectively removed low-frequency drift while preserving the main spectral features. All portable MicroNIR spectra were retained, as none exhibited apparent anomalies relative to the overall dataset. However, a subset of benchtop NIR spectra showed significant deviations (Fig. 4); since these were acquired sequentially and could not be linked to sample composition or moisture level, they were considered non-representative and excluded from further analysis, although the underlying cause could not be determined.

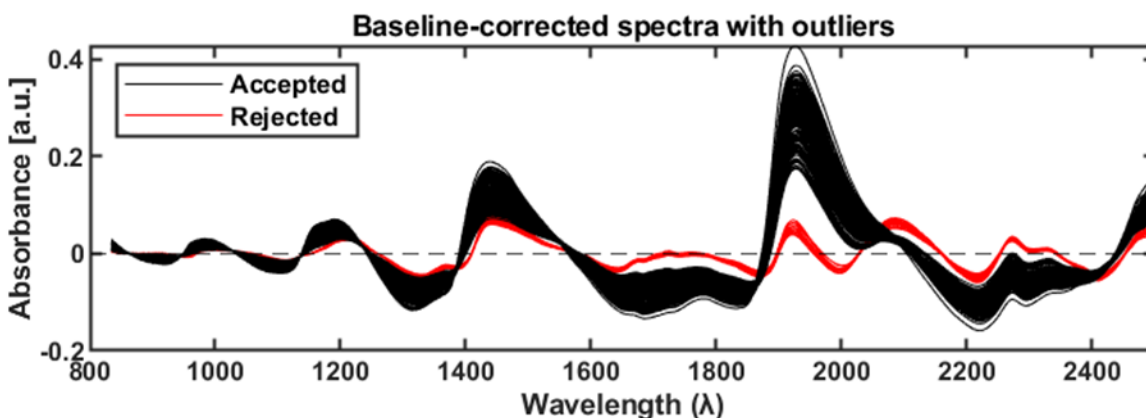


Fig. 4. Baseline-corrected benchtop NIR spectra showing retained measurements (black) and excluded outliers (red). The outliers could not be explained by moisture content or wood species and were therefore removed.

Regression Models

Partial Least Squares Regression (PLSR) is a multivariate technique that is widely used in chemometrics. It can relate a set of predictor variables (a NIR spectrum in this case) to one or more response variables (here, lignin and moisture fractions) by projecting the predictors onto a lower-dimensional space of latent variables. A linear relation is then modelled between these latent variables and the response.

To evaluate the predictive capabilities of portable NIR instrumentation, PLSR was applied to the second derivative spectra obtained from Portable NIR measurements. Spectral preprocessing involved Savitzky–Golay smoothing (Savitzky and Golay 1964) and differentiation with an optimized window width (10 nm) and polynomial order (3).

For each subsample spectrum, the preprocessed average spectrum served as the predictor, and the corresponding composition values were used as the response, as detailed below. The goal was to model the relative amounts of four key constituents in wood chips: cellulose, lignin, free moisture, and bound water. In this study, the discretised wavelength is a vector $\lambda = [\lambda_1, \lambda_2, \dots, \lambda_p]$, where p is the number of absorbance values in each spectrum. Each NIR spectrum i forms a predictor vector $\mathbf{x}_i = [A_{i1}, A_{i2}, \dots, A_{ip}]$, with $i=1,2,\dots,n$ where n is the number of subsamples and A_{ij} is the second derivative of the averaged absorbance for subsample i at the wavelength λ_j . These vectors are averaged into a predictor matrix $\mathbf{X} \in \mathbb{R}^{n \times p}$.

For each subsample, the response variables represent the mass fractions of the main components of the wood. Klason lignin characterization yields the total lignin fraction of a batch in the bone-dry state. Thus, for each subsample i , let $f_{L,i}$ denote the dry lignin mass fraction, while $f_{C,i} = 1 - f_{L,i}$ denotes the dry mass fraction of celluloses.

In moist wood, bound water and free water experience slightly different chemical environments, so that they effectively behave as two distinct compounds. For a moist wood sample, the mass fraction of lignin, cellulose, bound water and free water are denoted by $\phi_{L,i}$, $\phi_{C,i}$, $\phi_{w_b,i}$ and $\phi_{w_f,i}$ respectively, and

$$\phi_{L,i} + \phi_{C,i} + \phi_{w_b,i} + \phi_{w_f,i} = 1. \quad (2)$$

Only the total mass fraction of water $\phi_{w,i} = \phi_{w_b,i} + \phi_{w_f,i}$ is easily accessible in experiments and this makes it possible to identify the following:

$$\phi_{L,i} = (1 - \phi_{w,i})f_{L,i}, \quad (3)$$

$$\phi_{C,i} = (1 - \phi_{w,i})(1 - f_{L,i}). \quad (4)$$

Bound water originates from interactions with the solid constituents of the material and therefore scales with the dry fraction. Thus, a bound-water saturation constant $x_{w_b,lim}$ was defined as the maximum amount of bound water per unit dry mass. The fraction of free water is zero until this saturation point is reached, which implies that

$$\phi_{w_b,i} = \min [\phi_{w,i}, x_{w_b,lim} (1 - \phi_{w,i})], \quad (5)$$

$$\phi_{w_f,i} = \phi_{w,i} - \phi_{w_b,i}. \quad (6)$$

Due to the mass balances in dry and moist state, each subsample composition has three degrees of freedom. The response vector was chosen as follows,

$$\mathbf{y}_i = [f_{L,i}, \phi_{w_f,i}, \phi_{w_b,i}]^T, \quad (7)$$

understanding that $f_{C,i}$, $\phi_{C,i}$, and $\phi_{L,i}$ can be calculated from that triplet. Thus, the response matrix $\hat{\mathbf{Y}} \in \mathbb{R}^{n \times q}$, where $q = 3$ is the number of response variables, is assembled from these vectors. Note that $\hat{\mathbf{Y}} = \hat{\mathbf{Y}}(\phi_{w_b,lim})$ depends on the bound water upper limit, which needs to be determined through optimization.

Here, a linear model linking predictors and responses is built using PLSR. Let $\mathbf{X} \in \mathbb{R}^{n \times p}$ be the predictor matrix of second-derivative absorbances spectra (B. Zimmermann and A. Kohler, 2013), and $\mathbf{Y} \in \mathbb{R}^{n \times q}$ the standardized response matrix. PLS finds a small number l of latent variables that maximize covariance between $\mathbf{X}$ and $\mathbf{Y}$, giving the multiresponse linear relation

$$\hat{\mathbf{Y}} = \mathbf{1}\mathbf{a}^T + \mathbf{X}\mathbf{B}, \quad (8)$$

where $\mathbf{a} \in \mathbb{R}^q$ are intercepts and $\mathbf{B} \in \mathbb{R}^{p \times q}$ are regression coefficients computed from the l PLSR components. The number of latent variables l is selected by 10-fold cross validation, choosing the shoulder point in cross-validated mean square error (CV-MSE) to balance bias and predictive performance. The quality of the model fit is summarized using R^2 , adjusted R^2 , and Root Mean Square Error (RMSE) where

$$R_{adj}^2 = \left(1 - \frac{(1-R^2)(n-1)}{n-l-1}\right). \quad (9)$$

Separate models were created for the portable instruments and the benchtop reference.

RESULTS AND DISCUSSION

A comprehensive dataset was obtained alongside the NIR spectra by measuring moisture content in the wood samples together with Klason lignin. This data set enables the correlation of absorption at specific wavelengths with wood composition. To ensure that regression models are of sufficient quality, the measurement stability was assessed.

Measurement stability

The portable MicroNIR exhibited substantially higher effective SNR (4–6x across 900 to 1700 nm) compared with Bruker Matrix F in this specific sampling configuration (Fig. 5). The authors believe that this is not due to detector limitations, but to the different measurement geometries. The portable MicroNIR operated in close-contact mode, providing more stable sample-sensor interaction, whereas the non-contact measurement head of the Matrix F introduced greater variability due to surface heterogeneity and distance fluctuations. These results support the suitability of portable NIR instrumentation for wood chip analysis, particularly in moisture-sensitive regions such as the 1450 nm OH overtone band, where the portable MicroNIR showed notably lower noise.

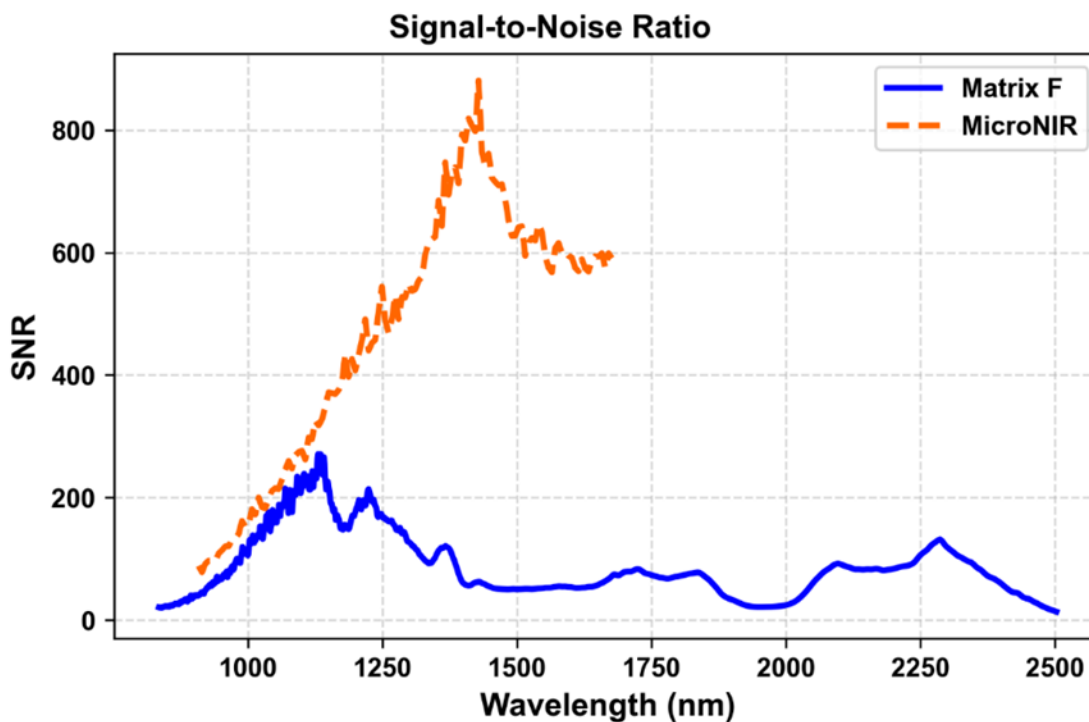


Fig. 5. Standard deviation of repeated spectral scans used to quantify instrument noise for benchtop and portable NIR devices

Portable NIR model

For the portable NIR dataset, the response matrix $\mathbf{Y}$ depends on the assumed upper limit for bound water, $x_{w_b,lim}$, which means that two parameters must be selected when constructing the PLS model: the number of latent variables and $x_{w_b,lim}$. This was addressed by computing the cross-validated mean squared error (CV-MSE) for models with 1 to 10 latent variables across several values of $x_{w_b,lim}$ from 0.0 to 0.50. By plotting CV-MSE

against the number of latent variables for each tested $x_{wb,lim}$, a common shoulder point was identified that defined the optimal number of latent variables ($l=4$), and at this number of latent variables $x_{wb,lim}$ the lowest error occurred at $x_{wb,lim} = 0.27$ (Fig. 6). The bound-free water split is conventionally referenced to the fibre saturation point (FSP), which for softwood is typically about 30% on a dry-mass basis (Glass and Zelinka 2021; Thybring *et al.* 2022; Sebera *et al.* 2025). The number of latent variables was selected by repeated 10-fold cross-validation using the 1-SE rule (Hastie *et al.* 2009).

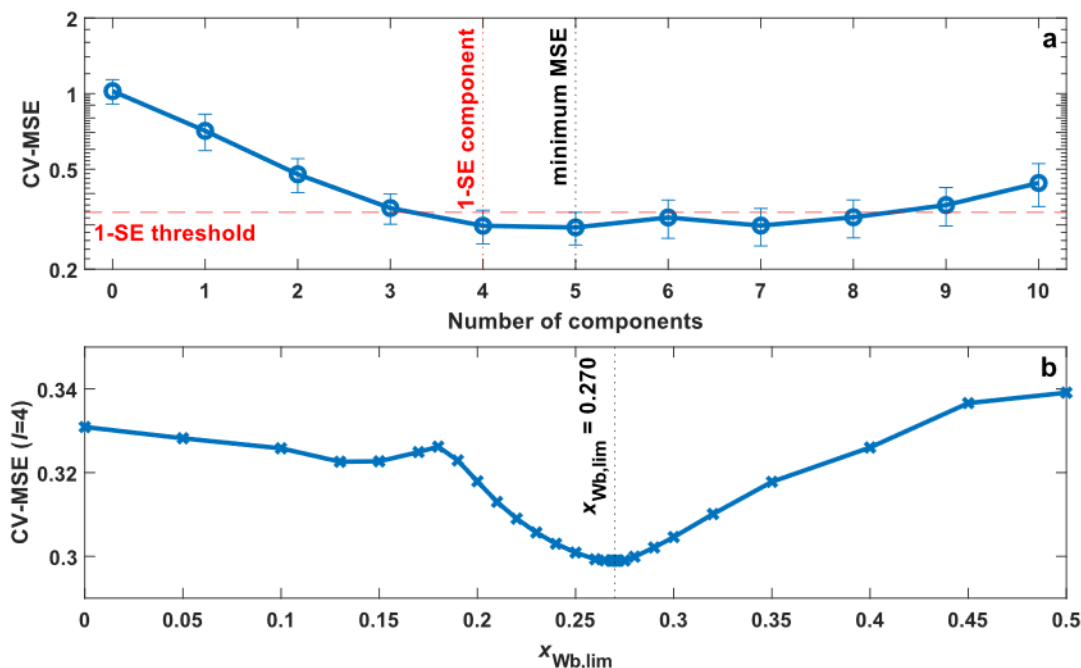


Fig. 6. (a) Cross-validated MSE for Portable NIR. The error indicates one standard error across folds. According to the 1-SE rule, the number of components, $l=4$, is taken as the minimum that produces a CV-MSE under the threshold. (b) Cross-validated MSE as a function of limiting fraction of bound water at a constant $l=4$

The portable NIR model gave a fair fit for lignin and moisture content of spruce and pine chips. For lignin (Fig. 7a), predictions showed a narrow range of measured values and moderate agreement with reference data, reflecting the limited chemical sensitivity of the portable device. The results showed that the model differentiated between fit of free (Fig. 7b) and bound (Fig. 7c) water fractions. Despite these limitations, the composition could be fit to portable NIR data for spruce and pine using a single model.

The adjusted coefficients of determination for the portable model were $R^2_{adj}=0.7294$ for lignin, 0.8430 for free water, and 0.6746 for bound water, corresponding to overall $R^2=0.7545$, 0.8576, and 0.7048, respectively (Table 2). These results indicate that the portable NIR performed best for predicting moisture-related variables, particularly free water (Fig. 7b), while predictions of lignin were less accurate (Fig. 7a).

The spectral loadings of the portable NIR PLS model (Fig. 7d) highlight the wavelength regions that contribute most strongly to separating lignin, free moisture, and bound moisture. The loading patterns remains consistent when the number of spectra used for averaging is reduced from 40 to 10, indicating that the model interpretation is robust to reduced spectral averaging. Pronounced features were observed in regions associated with O-H vibrations, consistent with the known dominance of water-related absorptions in the

NIR spectra of wood. Strong contributions occur near the second overtone of O-H stretching around 970 to 980 nm and in the first overtone region near 1410 to 1440 nm, both characteristic of water and consistent with literature-reported absorption regions compiled in Klecina *et al.* (2025). These regions contribute strongly to the discrimination of free moisture, reflecting the sensitivity of liquid-like water to changes in hydrogen bonding and absorption intensity. The bound-moisture loadings showed more distributed structure across these O-H regions, indicating that the model exploits subtler spectral differences associated with water molecules interacting with cell wall polymers rather than bulk liquid water.

In addition to water-related features, the lignin loadings exhibited notable contributions in the 1140 to 1200 nm region, which is associated with C-H combination bands in lignin, as summarized in Klecina *et al.* (2025), as well as weaker contributions overlapping the O-H overtone region near 1410 to 1440 nm. Overall, the spectral loadings demonstrated that model discrimination is driven primarily by variations in water-related absorptions, with lignin information extracted from broader, partially overlapping bands with the limited spectral range of portable instrument.

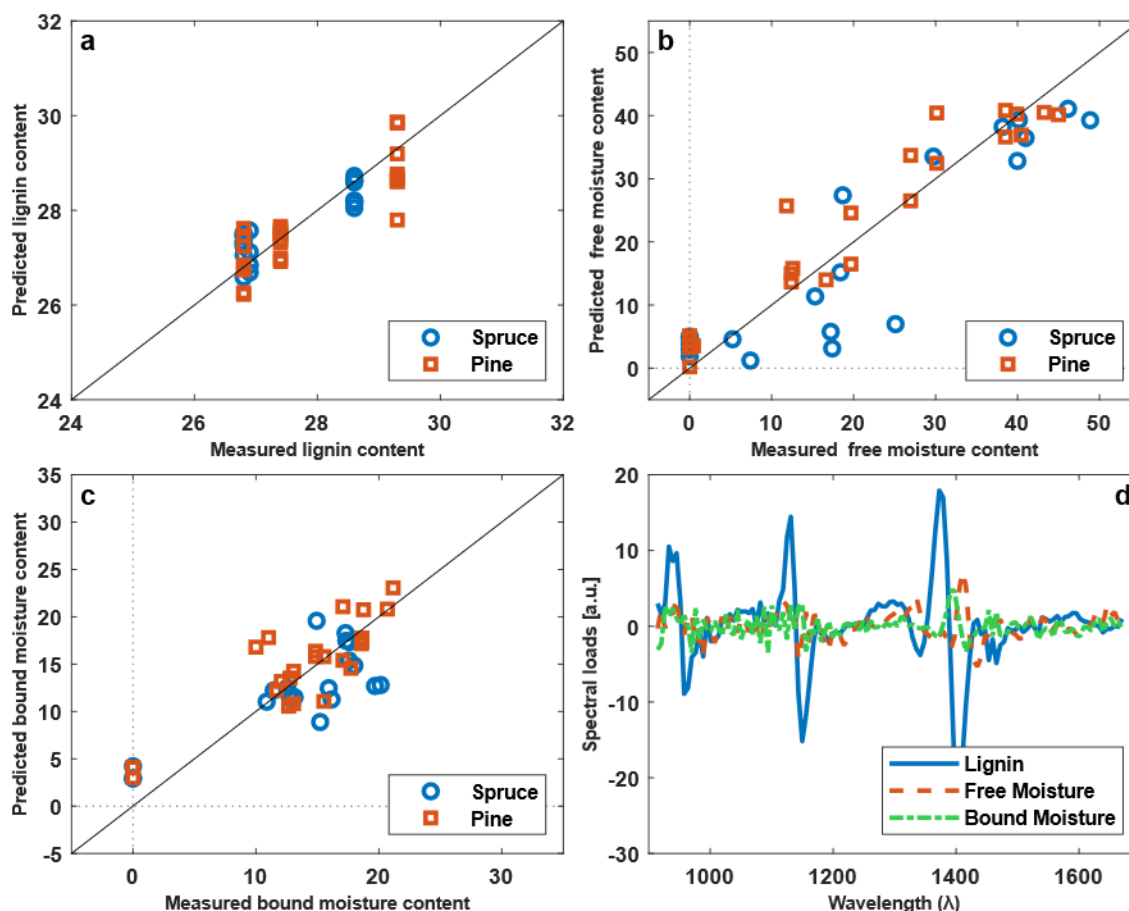


Fig. 7. Portable NIR PLS model with free and bound water a) Measured vs. predicted lignin content b) Measured free moisture content vs. predicted free moisture content c) Measured bound moisture content vs. predicted bound moisture content d) Spectral loadings as function of wavelength

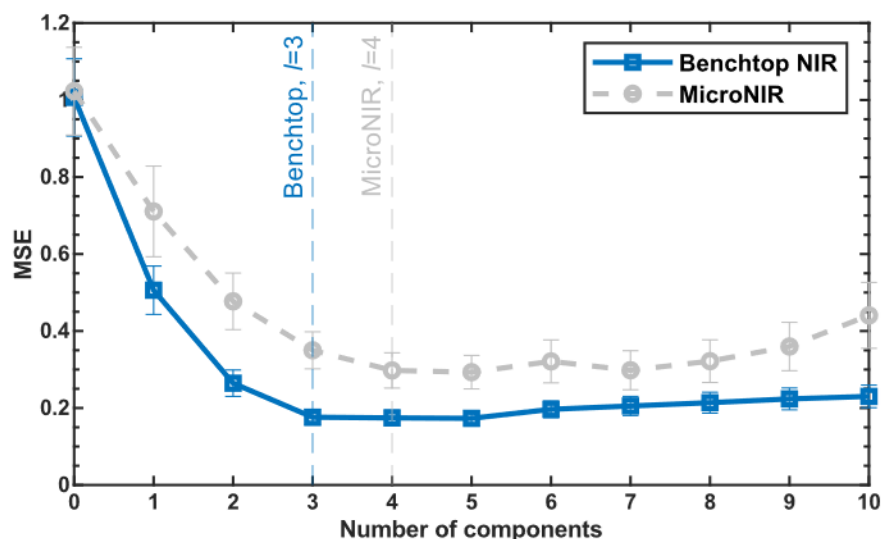
Table 2. Performance Matrices of Benchtop Model with 3 Latent Variables and Portable NIR Models with 4 Latent Variables

Instrument	Benchtop NIR	Portable NIR
Adj.R ² Lignin	0.8205	0.7294
Adj.R ² Free Water	0.8769	0.8430
Adj.R ² Bound Water	—*	0.6746
R ² Lignin	0.8247	0.7545
R ² Free Water	0.8798	0.8576
R ² Bound Water	—*	0.7048

*There is no measurement point for bound water in the Benchtop case

Benchtop NIR model

The benchtop NIR model was developed using the same procedure as for the portable NIR, giving only 3 latent variables according to the 1-SE rule. The subsamples used in these measurements were chosen to represent the moisture conditions typical for wood chips in the mill. Consequently, they did not have total moisture contents below the bound water limit $x_{wb,lim}$. A qualitatively similar relation between number of latent variables and the CV-MSE was observed for the two different instruments. However, the benchtop instrument yielded a model with lower CV-MSE and lower standard error (Fig. 8). This may be due to the broader spectral range of the benchtop NIR (833–2500 nm) and to its higher optical resolution, which resolve overlapping water and lignin absorption features more effectively.

**Fig. 8.** Comparison between benchtop and portable MicroNIR MSE. The benchtop model Pareto-dominates the MicroNIR MSE.

The benchtop model showed a clear separation of spectral features and fair predictive performance. In Fig. 9a, a strong linear relationship between measured and predicted values was observed for lignin, though some spread remains, particularly among spruce samples. The limited range of lignin content (~25 to 30%) together with the strong influence of water absorption in overlapping wavelength regions likely contributed to the moderate model precision in Table 2 ($R^2_{adj} = 0.8205$). Figure 9b illustrates the prediction of

free moisture, which achieved the highest accuracy among all responses ($R^2_{adj} = 0.8769$), thus confirming the instrument's ability to capture O-H overtones and combination bands associated with water. Figure 9c presents the spectral loadings of the benchtop NIR PLS model for lignin (blue) and free moisture (orange) across the 900 to 2300 nm. Several well-defined peaks and valleys coincided with established NIR absorption regions of water, including the second O-H overtone near 970 to 980 nm, the first overtone region around 1414 to 1438 nm, and strong combination bands in the 1916 to 1942 nm and ~1980 nm, as summarized in Klecina *et al.* (2025). These features dominate the free-moisture loading, reflecting the sensitivity of the benchtop system to variations in liquid-like water. In addition to water-related bands, distinct contributions were observed in regions associated with lignin, particularly in the 1140 to 1200 nm range attributed to C-H combination bands, as well as broader features above 2100 nm where overlapping C-H and aromatic vibration of lignin are expected (see Klecina *et al.* 2025). The clear separation and definition of these spectral features illustrate the superior ability of the benchtop instrument to resolve overlapping absorption bands compared with the portable system, enabling more physically interpretable loadings.

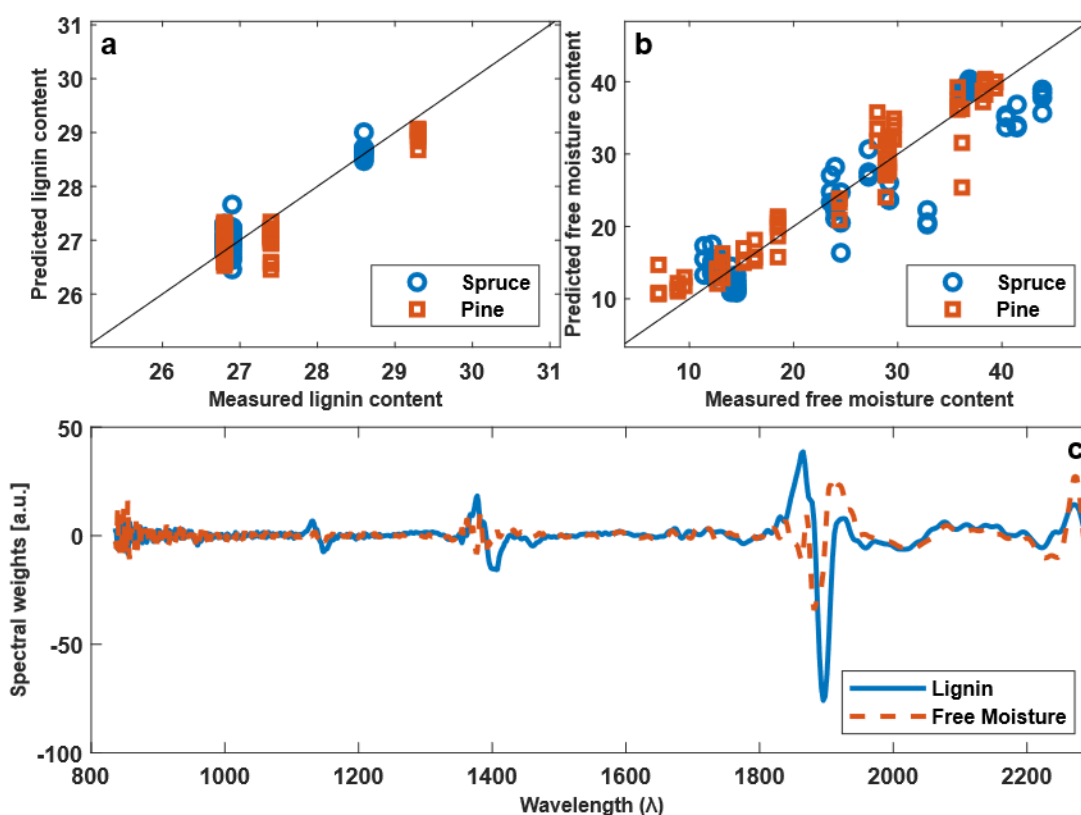


Fig. 9. Benchtop NIR PLS model with free water (the bound-water fraction is constant in the investigated regime) a) Measured vs. Predicted lignin content b) Measured free moisture content vs. predicted free moisture content c) Spectral loadings as function of wavelength

Assessment of Portable NIR

The results demonstrate that the portable MicroNIR provides meaningful calibration models for predicting lignin and free moisture in wood chips, although with lower predictive accuracy than the benchtop NIR reference (Table 2). The benchtop

instrument was able to serve as a benchmark, offering higher accuracy and reproducibility for both properties. Importantly, this performance difference was not explained by inferior signal quality, as SNR analysis showed comparable or higher effective signal-to-noise ratios for the portable MicroNIR across the 900 to 1700 nm range (Fig. 5). The difference was most likely due to its close-contact measurement geometry. The reduced predictive performance of the portable system may instead be attributed to intrinsic instrumental limitations, primarily to its narrower spectral range and lower spectral resolution, which limit separation of overlapping absorptions features, particularly for lignin. This interpretation is supported by spectral analysis, where the benchtop NIR exhibits more structured and physically interpretable regression profiles, while the portable MicroNIR coefficients are less distinct due to limited spectral information and derivative-based preprocessing. Nevertheless, both instruments captured the dominant trends for lignin and moisture. The portable MicroNIR system still produces usable, process-relevant information in applications where rapid and flexible measurements are prioritized over maximum analytical precision. For both instruments, sample heterogeneity and moisture distribution introduce variability, and the use of multiple scans or averaged measurements is recommended to improve robustness.

A relevant question is whether the portable NIR model performance reported here is sufficient for direct process control. The moderate correlations ($R_{adj}^2 = 0.73$ for lignin, 0.84 for free water) suggest that the portable NIR is most suitable as a rapid screening and decision-support tool rather than for closed-loop control of the digester, where higher accuracy is required. For digester control, it may be advantageous to combine portable NIR measurements with a complementary technology. Microwave transmission sensors, already installed at some kraft pulp mills, can determine bulk moisture content with accuracies around 1.0% and measure through the full chip bed depth (Nyström and Dahlquist 2004), but they do not provide information on chemical composition. A sensor fusion approach, combining microwave-derived moisture with NIR-derived compositional information (lignin, free and bound water fractions), could yield a more robust and complete characterization of chip quality than either technology in isolation. Such multi-sensor strategies have been advocated for industrial moisture measurement (Rahman *et al.* 2024) and represent a promising direction for future kraft pulping applications.

CONCLUSIONS

1. This study showed that a portable MicroNIR device, when averaging multiple measurements, can provide usable predictions of moisture and lignin in wood chips when calibrated against benchtop NIR reference data, although with lower accuracy than laboratory systems. The calibration models developed are specific to the investigated dataset and measurement conditions, and their application at other mill sites would require validation and potentially recalibration.
2. Despite its reduced spectral range and resolution, portable MicroNIR offers clear practical advantages through rapid, on-site measurements that are relevant for process monitoring and material screening in pulp mills. For applications requiring higher accuracy, combining portable NIR with complementary moisture measurement technologies may provide a more robust basis for process control.
3. Reliable use of portable MicroNIR requires well-defined measurement routines,

including controlled contact geometry, appropriate numbers of replicate scans, and robust software handling of spectral averaging, and a sufficient number of replicate scans; in the present data, averaging at least 10 scans preserved the relevant loading patterns.

4. These results indicate that separating free and bound water, rather than modelling total moisture alone, may improve MicroNIR model performance, but this approach requires further validation.
5. The portable MicroNIR was tested under laboratory conditions, not in the field; therefore, its performance under real operating conditions remains to be verified.

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Conflict of Interest

There is no conflict of interest.

Use of Generative AI

ChatGPT by OpenAI and Grammarly were used to refine the language and clarity of this paper. The authors assume full responsibility for all content, including analyses, conclusions, and interpretations.

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