DAYANE TARGINO DE MEDEIROS

IMPACT OF MOISTURE AND NIR SENSORS ON CALIBRATION TRANSFER BETWEEN PREDICTIVE WOOD DENSITY MODELS OF *Eucalyptus grandis* W. Hill ex Maiden.

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To God, my parents and Felipe Gomes,
for their support, love and dedication!

Dedicated

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The caterpillars don't announce themselves, they make cocoons and, silently, the butterflies reappear.

Victor chaves

RESUMO GERAL

Soluções tecnológicas para rápida classificação da madeira com base na densidade e umidade são fundamentais para o controle de qualidade da matéria-prima em indústrias florestais. A espectroscopia no infravermelho próximo (NIR), associada ao aprendizado de máquinas, possibilita a avaliação da madeira de forma rápida, precisa e não destrutiva. Como limitação, esta técnica requer que modelos gerados por um equipamento sejam aplicados apenas em espectros registrados pelo mesmo sensor. Assim, a transferência de calibração entre equipamentos poder ser útil para avaliar materiais de diferentes origens com informações espectrais de diferentes sensores. Objetivou-se desenvolver modelos preditivos e realizar a transferência de calibração entre equipamentos NIR de bancada e portátil para estimar a densidade básica da madeira em diferentes condições de umidade. Para isso, corpos de prova foram produzidos a partir de discos de *Eucalyptus grandis*, os quais foram analisados da condição saturada (umidade > 30%) até a umidade de equilíbrio. Assim, assinaturas espectrais foram coletadas diretamente na superfície das madeiras por meio do espectrômetro NIR de bancada com esfera integradora e com o equipamento NIR portátil. Os espectros e a massa corrente dos corpos de prova foram medidos a cada 10% de dessorção de água na madeira em 10 etapas de secagem. Para obtenção da densidade básica da madeira empregou-se a razão entre massa anidra e volume saturado. Os espectros adquiridos nas diferentes etapas de secagem foram submetidos à análise de componentes principais, análise discriminante por mínimos quadrados parciais e regressão dos mínimos quadrados parciais. A regressão multivariada para estimativa da densidade da madeira foi ajustada a partir dos espectros no NIR coletados em diferentes condições de umidade nos dois equipamentos. Foram geradas regressões considerando o modelo NIR global, modelo NIR de bancada e modelo NIR portátil. Os modelos preditivos foram testados em lotes de amostras desconhecidas para verificar o seu desempenho em validação externa. Além disso, espectros obtidos pelo equipamento de bancada foram aplicados no modelo desenvolvido a partir dos espectros registrados pelo equipamento portátil e vice-versa. Por fim, validação cruzada completa (*leave-one-out*) e validação independente foram realizadas para avaliar a performance preditiva dos modelos e dos equipamentos em transferência de calibração. Os modelos desenvolvidos apresentaram coeficiente de predição variando de 0,78 a 0,91 e de 0,77 a 0,87 para os equipamentos de bancada e portátil, respectivamente. Na transferência de calibração, o coeficiente de determinação do modelo atingiu 0,93. Com isso, os modelos foram adequados para serem aplicados em informações espectrais obtidas por diferentes sensores NIR para estimativa da densidade básica da madeira em várias condições de umidade. Os resultados obtidos podem facilitar o monitoramento e padronização do controle de qualidade da matéria-prima e dos produtos de empresas de base florestal.

Palavras-chave: Relação água-madeira; monitoramento de qualidade; floresta 4.0; modelagem PLS; aprendizado de máquina.

ABSTRACT

Technological solutions for the rapid classification of wood based on density and moisture content are essential for quality control of the raw material in forestry industries. Near-infrared spectroscopy (NIR) combined with machine learning makes it possible to evaluate wood quickly, accurately and non-destructively. As a limitation, this technique requires that models generated by a piece of equipment only be applied to spectra recorded by the same sensor. Thus, transferring calibrations between equipment can be useful for evaluating materials from different sources with spectral information from different sensors. The aim was to develop predictive models and carry out calibration transfer between benchtop and portable NIR equipment to estimate the basic density of wood under different moisture conditions. To do this, specimens were produced from *Eucalyptus grandis* discs, which were analyzed from the saturated condition (moisture > 30%) to equilibrium moisture. Spectral signatures were collected directly from the surface of the wood using a benchtop NIR spectrometer with integrating sphere and portable NIR equipment. The spectra and the current mass of the specimens were measured every 10% of water desorption in the wood during 10 drying stages. The ratio of anhydrous mass to saturated volume was used to obtain the basic density of the wood. The spectra acquired in the different drying stages were subjected to principal component analysis, discriminant analysis by partial least squares and partial least squares regression. The multivariate regression for estimating wood density was adjusted based on the NIR spectra collected under different moisture conditions in the two pieces of equipment. Regressions were generated considering the global NIR model, the benchtop NIR model and the portable NIR model. The predictive models were tested on batches of unknown samples to check their performance in external validation. In addition, spectra obtained by the benchtop equipment were applied to the model developed from the spectra recorded by the portable equipment and vice versa. Finally, complete cross-validation (leave-one-out) and independent validation were carried out to assess the predictive performance of the models and the equipment in calibration transfer. The models developed showed a prediction coefficient ranging from 0.78 to 0.91 and from 0.77 to 0.87 for the benchtop and portable equipment, respectively. In the calibration transfer, the model's coefficient of determination reached 0.93. As a result, the models were suitable for application to spectral information obtained by different NIR sensors for estimating the basic density of wood in various moisture conditions. The results obtained can facilitate the monitoring and standardization of quality control of raw materials and products from forest-based companies.

Keywords: Water-wood relationship; quality monitoring; forest 4.0; PLS modeling; machine learning.

INDICADORES DE IMPACTO

O desenvolvimento de soluções tecnológicas para a rápida classificação da madeira com base na densidade e umidade tem impactos significativos no setor florestal, promovendo avanços sociais, econômicos, ambientais e tecnológicos. Socialmente, essa tecnologia contribui para a geração de empregos e capacitação profissional. No aspecto econômico, a automação dos processos de análise reduz custos operacionais, minimiza perdas de material e melhora a competitividade do setor florestal. Ambientalmente, o uso de métodos não destrutivos reduz o desperdício de recursos naturais e trata-se de técnica limpa, sem uso de reagentes, o que contribui para o desenvolvimento sustentável. No campo tecnológico, a combinação da espectroscopia no infravermelho próximo (NIR) com algoritmos de aprendizado de máquina representa um avanço na digitalização do setor, permitindo maior padronização e flexibilidade dos métodos de análise, tornando a tecnologia mais acessível e eficiente. Dessa forma, os resultados obtidos neste trabalho têm o potencial de transformar a indústria florestal, promovendo a produção mais eficiente, sustentável e inovadora.

IMPACT INDICATORS

The development of technological solutions for the rapid classification of wood based on density and moisture has a significant impact on the forestry sector, promoting social, economic, environmental and technological advances. Socially, this technology contributes to job creation and professional training. Economically, the automation of analysis processes reduces operating costs, minimizes material losses and improves the competitiveness of the forestry sector. Environmentally, the use of non-destructive methods reduces the waste of natural resources and is a clean technique, without the use of reagents, which contributes to sustainable development. In the technological field, the combination of near-infrared spectroscopy (NIR) with machine learning algorithms represents an advance in the digitization of the sector, allowing for greater standardization and flexibility of analysis methods, making the technology more accessible and efficient. In this way, the results obtained in this work have the potential to transform the forestry industry, promoting more efficient, sustainable and innovative production.

SUMMARY

THESIS PRESENTATION

The thesis is divided into two parts. The first includes a general introduction, objectives and a literature review covering the industrial applications of *Eucalyptus* wood; wood properties and their correlation with industrial performance; near infrared spectroscopy and its applications; multivariate statistics associated with NIR, calibration methods; optimization: mathematical treatment, variable selection and *outlier* selection; validation methods; and NIR spectra calibration transfer. The second part of this thesis presents the articles generated during its execution, which are formatted according to the standards of the journal in which they were accepted for publication:

- ✓ **Article 1** deals with the development of statistical models to predict the basic density of *Eucalyptus grandis* wood, regardless of the moisture level of the material, using near-infrared spectroscopy (NIR) on benchtop and portable equipment.
  - ✓ Published in Industrial Crops & Products. 209 (2024) 117921. https://doi.org/10.1016/j.indcrop.2023.117921

- ✓ **Article 2** aimed to evaluate the potential of calibration transfer methods between benchtop and portable near-infrared (NIR) sensors to estimate the basic density of wood at different stages of drying, with a view to broadening the applicability of these models in different analysis conditions.
  - ✓ Published in Infrared Physics and Technology. 150 (2025) 105983. https://doi.org/10.1016/j.infrared.2025.105983

The third part corresponds to the general conclusion of the thesis, in which the main results obtained throughout the work are presented, as well as proposals for future studies.

FIRST PART

1 INTRODUCTION

Eucalyptus wood is a raw material widely used in various industries, meeting the demands of the pulp, panel, energy and sawn timber sectors (IBÁ, 2022). Among the most relevant physical properties for these segments, density and moisture content stand out, as they directly influence the behavior, processing and application of wood (AMARAL et al., 2020). Evaluating these characteristics is essential for monitoring the quality and appropriate sizing of wood products (COSTA et al., 2019).

Wood sampling and analysis procedures can be costly and time-consuming (ZAMPINI; OLIVEIRA, 2020). Technological advances have improved the processes for monitoring wood from planted forests, bringing more efficiency to industries and the marketing of forest products (RAMALHO et al., 2018; NISGOSKI et al., 2018). In this context, near-infrared spectroscopy (NIR) stands out as a promising tool for the qualitative and quantitative analysis of wood, using models based on multivariate statistics (HEIN et al., 2017).

The NIR technique is based on the electromagnetic radiation resulting from the interaction between biological material and the organic bonds present in its composition (DINIZ et al., 2019). Its main advantages include its non-destructive nature, the minimal need for sample preparation, the speed and accuracy of data collection, as well as the fact that it does not generate waste or require the use of chemical products. These characteristics make the technique environmentally sustainable and highly efficient (ESTOPA et al., 2017; UDDIN et al., 2020).

The statistical models developed for interpreting qualitative spectral information are mainly built using principal component analysis (PCA) and partial least squares discriminant analysis (PLS-DA). Partial least squares regression (PLS-R) is used to predict the properties of the samples analyzed (SANDAK et al., 2016; AMARAL et al., 2020).

Prediction models must cover broad sources of variation, as various factors can compromise their robustness. For example, the accuracy of the model can decrease over time due to different factors, such as changes in the chemical constitution of the material, variations between spectrometers and changes in sample conditioning (HONORATO et al., 2007). In view of this, calibration transfer has emerged as an effective strategy to

mitigate these problems, allowing the continued use of adjusted models in the face of instrumental and analytical variations. This avoids the need for frequent model reconstruction, a process that can be costly and time-consuming (ZHANG et al., 2022b).

Calibration transfer is mainly used to ensure that the calibrations developed on a particular piece of equipment (primary sensor) accurately match the spectra measured on another piece of equipment (secondary sensor) (FEUDALE et al., 2002). In this condition, the likelihood of changes occurring in the spectral data is clearly greater (VIA et al., 2005).

Thus, the response function of the secondary sensor is to become close to the response of the primary sensor, on which the model was built. This standardization can be achieved through mathematical manipulation of the spectral data, such as using algorithms to convert the data and including the spectral variation between the sensors in the spectral matrix (HONORATO et al., 2007). In this way, obtaining calibration models using multivariate statistical techniques with stability and precision is one of the main issues to be analyzed if NIR technology is to be viable for application on an industrial scale (ZHANG et al., 2022b).

In this context, to date, there are no reports in the literature of studies that estimate and compare the basic density of wood regardless of the moisture content of the raw material using a portable and benchtop NIR spectrometer. In addition, only in the study by Zhang et al. (2022a) were calibration transfer methods analyzed in wood science and technology models. However, the research was carried out for lignin prediction and between portable equipment, making it necessary to carry out this thesis.

2 OBJECTIVES

2.1 General objective

To understand the effect of moisture and the type of sensor on the predictive capacity of models for estimating the basic density of *Eucalyptus grandis* wood using benchtop and portable NIR spectrometers, as well as making it possible to transfer calibrations between the equipment.

2.1 Specific objectives

- ✓ Evaluate the effect of moisture on the validation of predictive models for wood basic density.
- ✓ Analyze the effect of the wood surface on the predictive capacity of models based on NIR spectra in prismatic samples and wood chips.
- ✓ Understand how the sensitivity of benchtop and portable NIR devices affects the performance of models for predicting wood density.
- ✓ Develop solutions for transferring calibrations prepared from a benchtop device to spectra acquired with a portable sensor with different bandwidths and resolutions.

3 LITERATURE REVIEW

3.1 Industrial applications of *Eucalyptus* wood

The *Eucalyptus* genus, which originated in Australia, comprises more than 700 species and several hybrids used in cloning for forestry, serving industries in different sectors (SERENINI et al., 2020). This genus is widely cultivated in tropical and subtropical regions, including Brazil, where soil and climate conditions favor its growth (GHANI; LEE, 2021). Its high productivity and ability to adapt to different environments are essential characteristics that enable it to be used in various segments of the timber industry (KAZMIERCZAK et al., 2017).

In the forestry industries, *Eucalyptus* wood is the main source of short fiber for pulp and paper production, as well as being widely used in the manufacture of charcoal, reconstituted panels and furniture (PARVEE et al., 2021). According to IBÁ (2022), the cultivation of *Eucalyptus* contributes significantly to Brazil's position in the world ranking of pulp and paper producers. In addition, Schettini et al. (2022) point out that *Eucalyptus* is the main source of sustainable energy, being used in the form of charcoal to supply the blast furnaces of the Brazilian steel industry.

Several studies have reported information on the characteristics of the wood of this species for the aforementioned purposes. For example, Vieira et al. (2021) investigated the chemical composition of ten *Eucalyptus* spp. clones and found that the *E. urophylla* × *E. grandis* hybrid resulted in wood with high glucose contents, around 64.2% on average, which is indicated for the production of cellulose pulp. Protásio et al. (2021), analyzing the quality of *Eucalyptus* wood for charcoal production, concluded that the *E. camaldulensis* and *E. grandis* species are the most suitable for charcoal production, due to the values of gravimetric yield (~36%), retained carbon (~60%) and retained hydrogen (~20%) in the charcoal obtained after pyrolysis.

In the study by Alencar and Moura (2019), cross-laminated timber (CLT) panels made from *Eucalyptus grandis* and *Pinus taeda* were evaluated. These authors found that the panels containing *Eucalyptus* wood in the outer layers had greater stiffness than the panels with *Pinus* and the mechanical properties met the requirements of technical standards for CLT, making it feasible to use *Eucalyptus* in the production of engineered wood.

In addition, Gutiérrez et al. (2019) found shear strength values for wood from *E. urophylla* × *E. grandis* clones ranging from 5.15 to 13.36 MPa, these results being

suitable for the standard criteria of the furniture industry. Similarly, Fonte and Anjos (2021) highlighted the potential of this wood for furniture applications, although they emphasized that the surface finish requires greater care, due to growth tensions and cracks resulting from juvenile wood.

Despite the advantages of using *Eucalyptus* wood in various sectors of the forest-based industry, there are bottlenecks when it comes to handling and processing the raw material, given the variation in wood quality and moisture, which can occur due to differences in storage time and the diameter and height of the piles. There are difficulties in controlling these variables, especially using destructive methods.

3.2 Wood properties and correlation with industrial performance

Density and moisture content are the main physical properties that indicate the quality of wood, as they have a direct correlation with its physical-mechanical characteristics (ALTEYRAC et al., 2006). The basic density of wood is defined by the ratio between the anhydrous mass and the saturated volume, and can vary within the tree according to factors such as soil and climate conditions, type of wood, growth rate, silvicultural management, site characteristics, pith-bark and base-top position, as well as the species and age of the tree (MASCARENHAS et al., 2021; SOUSA et al., 2021; SORO et al., 2022; WANG et al., 2022).

Moisture in wood can be present in two forms: adsorbed in the cell wall, through hydrogen bonds with the amorphous regions of the cellulose microfibrils and/or with hemicelluloses, or as free water, located in the intercellular spaces and cell lumens (SIAU, 1984). Moisture variation in wood is influenced by various factors, including environmental conditions, dimensions of the piece, density, chemical composition, impregnation by extractives, porosity, as well as the arrangement and types of anatomical elements (REZENDE et al., 2010).

Wood density and moisture must be monitored from harvest to the final product (DONATO et al., 2014). This is necessary because the amount of water and the proportion of fibers can cause variations in wood properties. In the pulp industry, the calculation of the wood liquor ratio depends on the dry mass of the chips and the amount of reagent used to cook the wood, making it necessary to know the percentage of moisture and solids content of the raw material (AMARAL et al., 2020). In the energy sector, wood moisture contributes to a reduction in calorific value, causing a reduction in the yield of the raw

material as moisture increases, requiring a greater amount of material to generate heat (NASCIMENTO; BIAGGIONI, 2010).

Likewise, transportation costs are also influenced, as the purchase of raw materials is made by mass, so the wetter the material, the smaller the amount of solid purchased. The same is true for light timber, a lot of volume and little mass in a load (BIERMANN, 1996). On the other hand, when wood density and moisture analyses are carried out correctly, several advantages can be highlighted, such as improved workability, reduced dimensional instability and attack by xylophagous agents and greater attenuation of defects (SILVEIRA et al., 2013).

The most widespread method for determining wood density is immersion in water and kiln drying, as recommended in ASTM D2395-17 (ASTM, 2017). However, sampling is a limiting factor due to the need to destroy trees or pieces of wood in commercial dimensions in order to obtain samples. In addition, this procedure requires time (days or weeks) to obtain results (KOLLMANN; CÔTÉ, 1984). Regarding wood moisture, it can be determined by the gravimetric method and distillation, or estimated by dielectric meters (KOLLMANN; CÔTÉ, 1984).

The gravimetric or oven-drying method is the most widely used, as it is highly accurate and easy to carry out; however, it is time-consuming and destructive (CALONEGO et al., 2006). Dielectric meters estimate moisture immediately, but are less accurate, especially above the fiber saturation point (FSP) (FRIDH et al., 2018). In distillation, the proportion of water is obtained by measuring the volume using chemical reagents that do not mix with water, such as xylene or toluene. This method is reliable and suitable for woods with high volatile content, but requires the use of toxic and expensive products (KOLLMANN; CÔTÉ, 1984).

It can be seen that the classic methods for determining density and moisture are costly, time-consuming or may not be accurate at moistures above the PSF, especially in the industrial context and in charcoal production units, where the movement and conditions of the raw material cause great variability. It is therefore important to implement new techniques that are fast, efficient and cost-effective so that wood quality monitoring can be optimized by forest-based industries (DONATO et al., 2014; MEDEIROS et al., 2023).

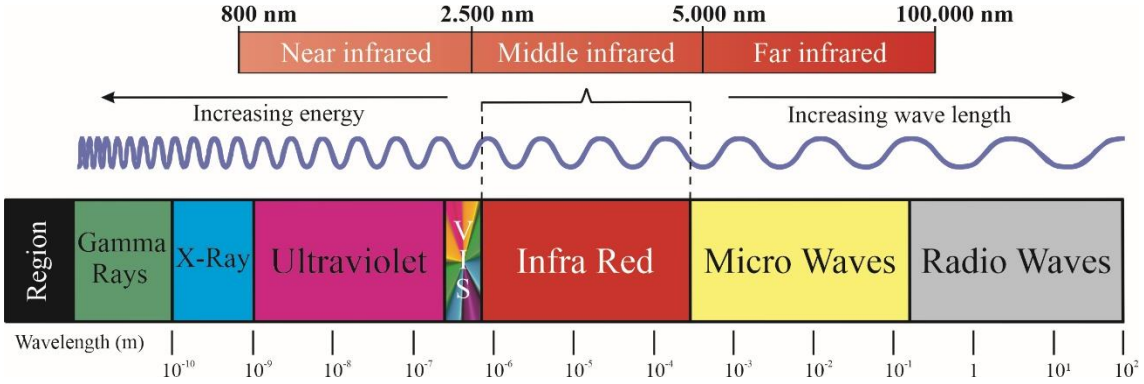
In this context, near-infrared spectroscopy has emerged as a technological alternative that has shown potential for predicting different wood properties in association with multivariate statistics, especially density and moisture content, as already indicated

in several studies (LIANG et al., 2019; BAROTTO et al., 2023; CHAMBI-LEGOAS et al., 2023; CICHOSZ et al., 2023).

3.3 Near-infrared spectroscopy and its applications

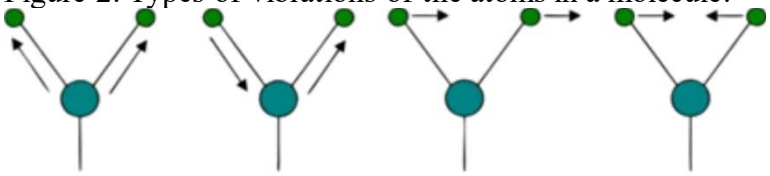
Near infrared spectroscopy (NIR) is based on the absorption intensity of electromagnetic radiation. From this, it is possible to analyze the interaction of the biological material with the organic bonds of the molecules constituting the sample (SHEPPARD et al., 1985), considering the near infrared region, which lies between visible light and infrared light, between 800 and 2500 nm (Figure 1).

Figure 1. Range of the electromagnetic spectrum of light with emphasis on the near infrared.



This interaction is associated with the vibrational patterns of the molecule's atoms and the spectral region, enabling the discrimination of different compounds with functional groups such as O-H, N-H and C=O (PASQUINI, 2003). According to Burns and Ciurczak (2008), the atoms in a molecule have numerous types of vibrations, mainly symmetrical and asymmetrical stretching, rocking and scissoring (Figure 2). In other words, if a molecule absorbs infrared radiation and the binding frequency is similar to the frequency of light, the NIR spectrum is obtained by recording the absorbance according to the wavelength of the incident light.

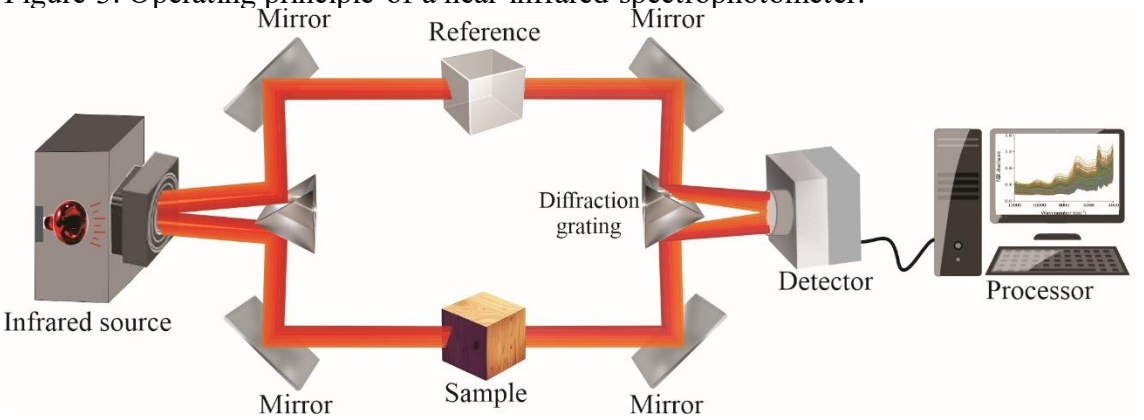
Figure 2: Types of vibrations of the atoms in a molecule.



Source: SKOOG et al. (2002).

The instruments that operate using NIR technology are made up of a light source with tungsten/halogen lamps, while the absorption measurements are made up of quartz or fused silica cells (SKOOG et al., 2002). In addition, the detectors are made from lead sulphide photoconductors (Figure 3). The NIR measurement modes are transmission, transflection and diffuse reflection. Diffuse reflection is the most widespread, in which the infrared light source is distributed on the sphere and directed towards the surface of the sample (PASQUINI, 2003).

Figure 3: Operating principle of a near-infrared spectrophotometer.



The first study using the NIR technique to gain prominence was led by Karl Norris in the 1960s in the United States. The researcher was looking for new methods of determining moisture in agricultural products and the results were promising (BOKOBZA, 1998). Today, in terms of applications, NIR spectroscopy has proven to be a suitable tool for managing raw materials and industrial products due to its accuracy of information, rapid data acquisition, non-invasive method that optimizes processes and minimal sample preparation (ESTOPA et al., 2017).

However, this technique also has some recommendations, such as the dependence on the calibration of predictive models, and it is necessary to use laboratory data to correlate with spectral signatures (MEDEIROS et al., 2023). In addition, the NIR sensor is sensitive to variations in atmospheric conditions (temperature and relative humidity) and the condition of the samples (surface and granulometry). These aspects, in turn, can cause variability and less predictability in the results (HEIN et al., 2009).

For the timber sector, research related to NIR has indicated that it is possible to simultaneously analyze different characteristics of wood through spectral signatures, such as the content of extractives, cellulose, hemicelluloses and lignin (MANCINI et al., 2020; AI et al., 2022), 2022), basic density (COSTA et al., 2018), moisture content (SANTOS

et al., 2020), mechanical strength (HEIN et al., 2009), plant fibre morphology (VIANA et al., 2009) and to categorize wood (RAMALHO et al., 2018; LIMA et al., 2022) and evaluate its products (COSTA et al., 2019; RAMALHO et al., 2019).

Table 1 shows the studies published in the literature using near-infrared spectroscopy to predict the basic density of wood in species of the *Eucalyptus* genus between 2018 and 2023. In short, the studies used benchtop NIR equipment and wood samples at equilibrium moisture, making it necessary to analyze the technique on wet raw material and on equipment that is easy to transport, such as portable equipment.

Table 1. Summary of work already carried out using NIR spectroscopy and basic density of *Eucalyptus* wood.

Authors	Species	Anatomical Plan	R^2
Miranda et al. (2023)	*Eucalyptus dunnii*	-	0,82
Benin et al. (2023)	*Eucalyptus benthamii*	-	0,68
Loureiro et al. (2022)	*Eucalyptus urophylla*	-	0,86
Amaral et al. (2021)	*E. urophylla* x *E. grandis*	Transversal	0,85
		Radial	0,70
		Tangential	0,53
Arriel et al. (2019)	*Eucalyptus* spp.	Transversal	0,77
Costa et al. (2018)	*E. urophylla* x *E. grandis*	Transversal	0,84
		Radial	0,78
		Tangential	0,75

3.4 Multivariate statistics associated with NIR

Near-infrared spectra contain a lot of complex information, making it necessary to use multivariate statistics to analyze the data. Principal component analysis (PCA) is the most widespread tool in qualitative evaluations, while partial least squares discriminant analysis (PLS-DA) and partial least squares regression (PLS-R) are widely used to develop categorical and prediction models, respectively (SANDAK et al., 2016).

PCA is a statistical method, used in chemometrics, which simplifies the information in a data set into a few variables, without significant loss of information in the sample (TIMM, 2002). The variables generated from the original variables are called principal components, with principal component 1 having a higher percentage of explanatory statistical information than principal component 2 and so on (NETO; MOITA, 1998). The analysis allows samples to be grouped according to their similarities,

using two-dimensional graphs, and its main characteristic is orthogonality, the latent variables obtained do not correlate with each other (THOMAS, 1994).

In PLS-DA, the model's algorithm aims to classify the variables by means of a linear separator; when there are more than two variables, the model's projection will be by a hyperplane in multidimensional space (BRERETON; LLOYD, 2014). This technique, used in the field of chemometrics, is suitable for identifying different groups of samples, while the regressions obtained by PLS are applied to predict continuous values in each of the categories. The main parameter indicating the efficiency of the model is the percentage of correct classification (GROMSKI et al., 2015).

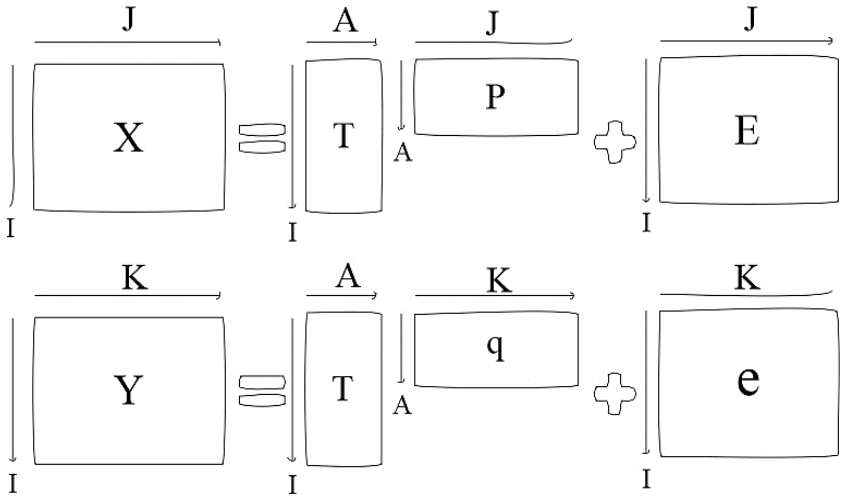
For building models with quantitative data, PLS-R is the most recommended analysis, as it predicts dependent variables from an independent set, correlating the variance of the Y matrix and X matrix data through their simultaneous and successive decompositions (BRERETON, 2003). The dependent variables are the values acquired in laboratory analyses and the independent variables correspond to the NIR spectra. As far as predictions are concerned, these are obtained by the latent variables, which have the best predictive performance in the model (ABDI, 2010).

3.5 Calibration methods

Multivariate calibration is a procedure that establishes the quantitative relationship between data obtained by an instrument (matrix X) and reference values estimated in the laboratory (matrix Y), with a view to developing prediction models (MARTENS; NAES, 1989). Several calibration methods are described in the literature, such as multiple linear regression (MLR), principal component regression (PCR) and partial least squares regression (PLS-R). However, PLS-R is the most common method to be applied to NIR spectra (FABER; RAJKO, 2007). PLS-R calibration allows the X and Y matrices to be broken down into two orthogonal sets, called scores and loadings (BERETON, 2003).

As shown in Figure 4, **T** corresponds to the matrix of scores, **P** to the loadings of the X matrix, **q** to the loadings of the Y matrix, **E** and e to the residuals of the decomposition of the X and Y matrices, respectively. Thus, **T** and **P** are close to the spectral data while **T** and **q** are close to the reference data, and the vector **T** of the scores of both data relates the two groups.

Figure 4: Decomposition of the X and Y matrices in the partial least squares regression analysis.



Source: BERETON (2003).

The parameters used as criteria for selecting PLS-R models are the coefficient of determination (R^2), which represents the correlation of the points on the calibration curve, the number of latent variables or principal components, the standard error values in calibration and validation, the root mean standard error (RMSE) and the deviation performance ratio (RPD), which assesses calibration accuracy (PASQUINI, 2003; BRERETON, 2003). Generally, 60% of the data is used for model calibration, while the remaining 40% is used for validation (MEDEIROS et al., 2023)

The main advantage of the PLS-R method is the accuracy of the models, as their statistical parameters do not change significantly when samples are added or removed from the calibration set (GELADI; KOWALSKI, 1986). This precision allows PLS-R models to be implemented in activities related to the production chain of large industries, making it possible to simplify their processes and reduce laboratory analysis costs, especially those that require daily assessments to monitor the quality of their products or raw materials (HEIN, 2008).

3.6 Optimization: Mathematical treatment, variable selection and *outlier* selection

In order to develop robust models, it is necessary to remove and/or reduce undesirable characteristics contained in the spectral signal, for which mathematical treatments are applied to the X matrix of the data (SWIERENGA et al., 1999). The main treatments for NIR spectral data are: multiplicative signal correction (MSC) and standard normal variation (SNV), which allow the removal of the additive baseline and the multiplicative effects of the signal. In addition, the first and second derivatives can be

used to reduce the effects of the additive and skewed baseline, respectively (HELLAND et al., 1995; SWIERENGA et al., 1998).

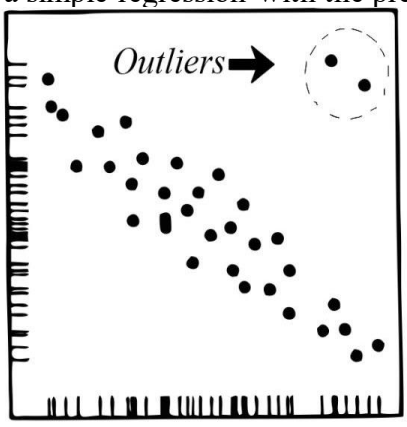
The selection of the number of latent variables, also known as PLS components, is a fundamental parameter for model optimization, since they determine the appropriate degree of a polynomial for calibration adjustment (FABER; RAJKO, 2007).

According to Barthus (1999), the recommended number of latent variables is the one that generates the least residual variation in validation. The use of a few latent variables produces more robust models that are easier to interpret, but when this number is too low, the information in the spectral data may not be properly exploited. On the other hand, when the number of variables is high, noise problems in the spectral signal can be caused to the model, increasing the error of the estimates.

Selecting the wavelength is yet another strategy for improving the predictive capacity of multivariate models. To do this, a spectral subset is used instead of the entire spectral range, especially if heteroscedastic noise is detected in the spectra (SWIERENGA et al., 1999). This selection results in a better model fit, reduces the size of the data, minimizes prediction errors and generates stability in the calibration (JIANG et al., 2002). Commonly, the spectral region of the first and second harmonics contain the most information that correlates with the variables of interest (LEARDI et al., 2002; GHASEMI et al., 2003).

Another option for improving the statistical parameters of the model is to remove *outliers* (ROUSSEEUW; LEROY, 2005), samples with gross errors and variable behavior within the data set are eliminated from the calibration process, since they are evident in the projection in a solitary way (Figure 5).

Figure 5 - Representation of a simple regression with the presence of *outliers*.



Source: ROUSSEEUW; LEROY (2005).

The selection of *outliers* contributes directly to the robustness of the model, increasing the probability of obtaining homogeneous information and grouping adjusted to the trend line. However, removing too many samples tends to reduce the representativeness of the data in less numerous matrices, and is not recommended in analyses where the number of samples is small (MARTENS; NAES, 1989).

Outliers can be detected by "leverage", a complementary quantity that analyzes the influence of a sample on the construction of the model. If the "leverage" measures are low, the sample has little contribution to the calibration; on the other hand, if the values are different from the calibration set, the sample probably has more influence on the model. In general, these samples are also easily detected by the scores graph (FERREIRA et al., 1999).

3.7 Validation methods

The model calibrated by PLS-R must be tested before its final application. This process is called validation, which consists of evaluating the consistency of the data and the model's ability to estimate the property of interest from similar samples, the replicability of the data. The testing process can be carried out using two methods, independent validation and cross-validation (PASQUINI, 2018).

The independent validation method refers to batches of external samples, a new set of data that was not used to build the model. This batch must present data and spectral signatures in the NIR with a variation range similar to the calibration data, in order to adjust the model's performance in relation to the parameters evaluated (WOLD, 1978). Thus, the calibrated model is applied to predict the value of the variable of interest in the validation set from the spectra obtained in the NIR (BURNS; CIURCZAK, 2008).

In cross-validation, the data for testing is taken from the calibration set itself, in which a sample is removed from the data set and the model is built by leaving it out, and its value is then predicted by the model. This procedure is repeated for all the samples in the set, ensuring more economical use of the data (SANDAK et al., 2016).

The most recommended method is independent validation, as it provides more realistic results. However, cross-validation is useful when you have a limited number of samples or in the case of laboratory analyses that require expensive reagents to obtain the reference values for a particular property of interest (PASQUINI, 2003).

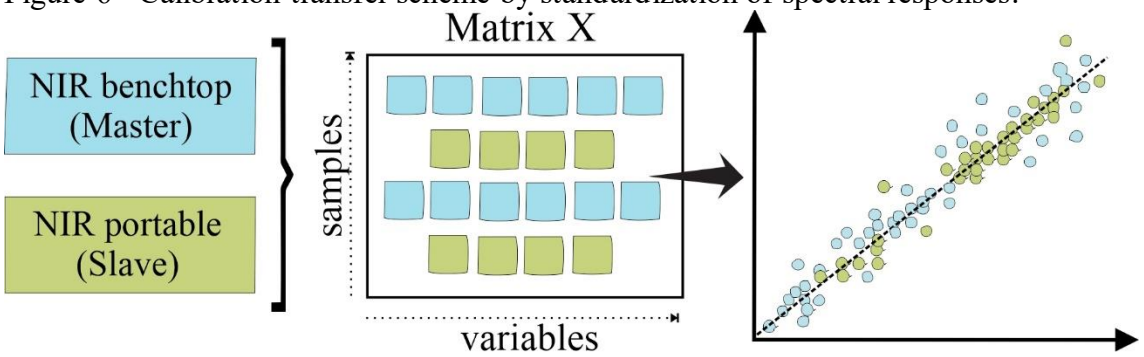
3.8 NIR spectra calibration transfer

Near-infrared (NIR) spectrometers vary in terms of the number of waves or absorbances measured, depending on the type of sensor (integrating sphere or optical fiber), device (benchtop or portable), model and manufacturer of the equipment (FEARN, 2001). Thus, the standardization of calibration models and the use of appropriate regression techniques have become fundamental to extracting more accurate information from the measured spectra. This, in turn, makes it possible to keep spectral responses similar between different pieces of equipment, given that reconstructing models can be costly and time-consuming (FORINA et al., 1995).

Calibration transfer is a procedure aimed at correcting and minimizing variations in instrumental responses between different pieces of equipment (WANG; KOWALSKI, 1992). The terminology "master" and "slave" is commonly used to explain the involvement between the equipment used in the transfer process, with the master equipment being responsible for the spectral acquisition of the calibration, with the initial analysis conditions, and the slave equipment as the receiver of the prediction model, represented by the new conditions (RUKUNDO et al., 2022).

Various methodologies have been recommended for performing calibration transfer, such as standardization of spectral responses, direct standardization (DS), piecewise direct standardization (PDS), reverse standardization (RS), univariate standardization and standardization of predicted values. In the standardization of spectral responses, the data matrix is made up of spectra from different samples, obtained from the "master" and "slave" equipment (Figure 6), does not require reference values, and the spectral responses are related by linear transformation (HONORATO et al., 2007).

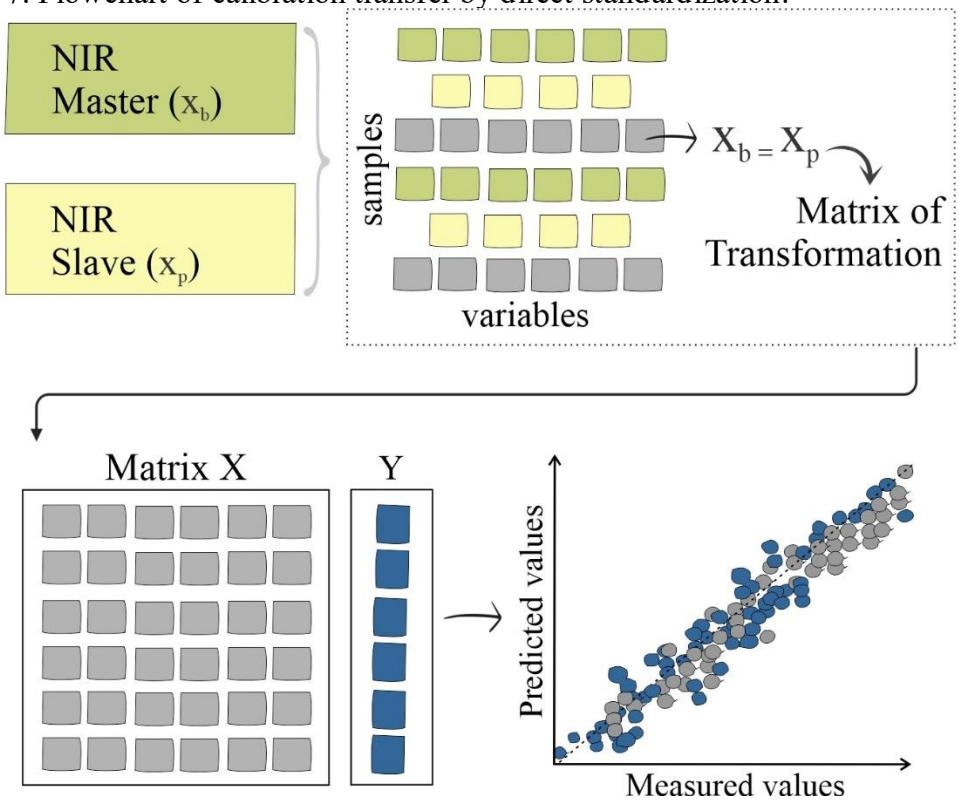
Figure 6 - Calibration transfer scheme by standardization of spectral responses.



Direct standardization (DS) is the method most commonly applied in calibration transfers (WANG et al., 1991). This method corrects the spectra of a given "slave" device,

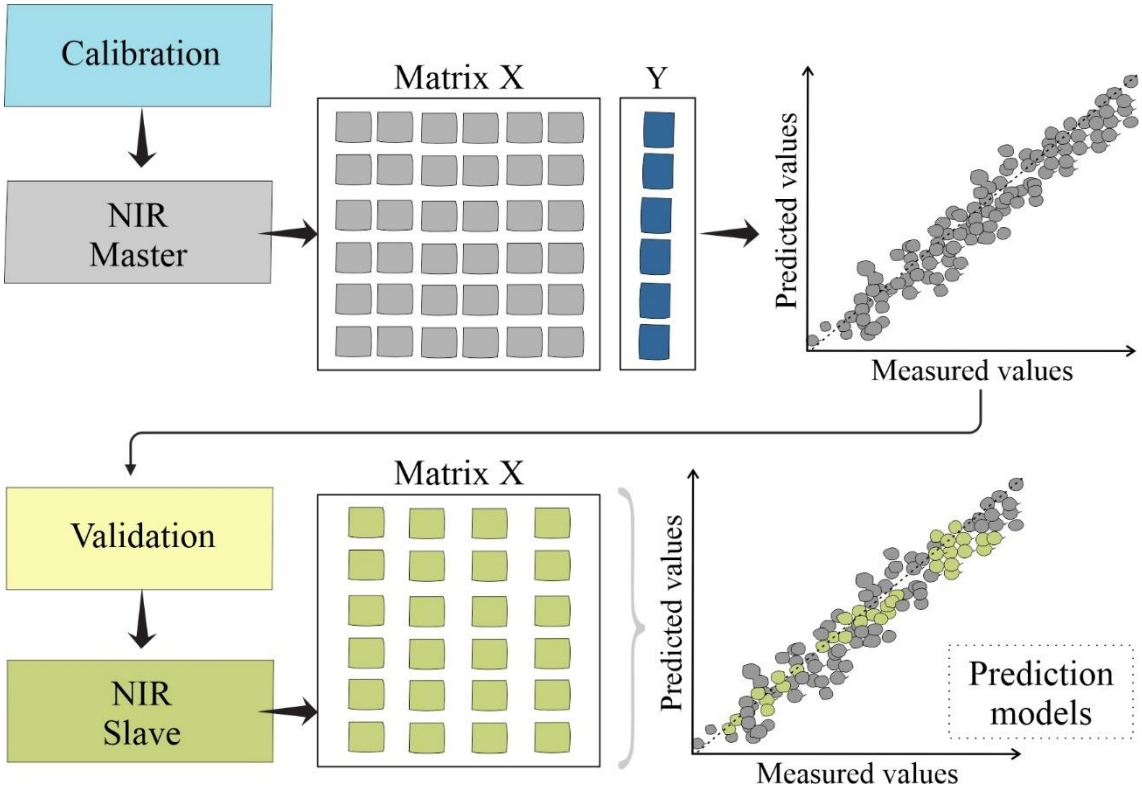
$\mathbf{X_p}$, so that they correspond to the spectra of the "master" device, $\mathbf{X_b}$. This correction is carried out using the $\mathbf{F(X_p = X_b)}$ matrix, known as the transformation matrix (Figure 7). If only a few wavelengths are selected, the procedure is called piecewise direct standardization (PDS). However, when the "slave" equipment has a lower resolution and acquisition range than the "master" equipment, the original spectra recorded on the 'master' spectrometer are standardized to be equivalent to those of the "slave" spectrometer. This is the reverse standardization (RS) method (RUKUNDO et al., 2022).

Figure 7. Flowchart of calibration transfer by direct standardization.



In the univariate standardization proposed by Shenk et al. (1985), for each wavelength of the "master" equipment, a correlation with the wavelength of the "slave" equipment is detected. The correlation between the wavelength of the "slave" device that most closely matches the wavelength of the "master" device is then calculated. After this wavelength correction, a spectral intensity correction is applied. The standardization of the predicted values follows the properties of the samples measured and estimated in the "master" and 'slave' equipment, using the calibration model built in the "master" equipment (Figure 8), and the data from the slave equipment to validate the information, with the results adjusted by regression equations (HONORATO et al., 2007).

Figure 8. Illustration of the calibration transfer of the predicted values.



Other approaches to facilitate the model transfer process can also be applied to the data matrix, such as selecting the main wavelengths that are associated with the properties of interest and building robust calibrations using mathematical pre-treatments to reduce spectral noise (FEARN, 2001).

In addition, Feudale et al. (2002) suggest that the calibrations should be updated before transferring between equipment, by including spectra recorded on different instruments in the data set, so that the model accounts for old and new experimental conditions and is valid for both situations.

In view of the above, it can be seen that calibration transfer is a useful tool for optimizing the prediction of variables obtained with different NIR devices. However, the number of studies on the calibration transfer of models based on NIR spectra applied to wood science and technology is scarce. Some varied reports of success have been reported by Zhang et al. (2022b), Rukundo et al. (2022), Schoot et al. (2022), Silva et al. (2017) and Hoffmann et al. (2016).

In the study by Zhang et al. (2022b), *Pinus nigra* wood density predictions were obtained from models transferred by direct piecewise standardization (PDS), with R^2 = 0.53 and RMSEP = 0.04 for wood with moisture ranging from 10% to 30%. In the study by Zhang et al. (2022a), different calibration transfer methods were analyzed between

four portable NIR spectrometers for predicting lignin in wood intended for pulp production. The most satisfactory results were obtained using direct standardization and direct standardization by parts, with R^2 greater than 0.79. With this, the authors reported promising performance of the calibration transfer between different NIR instruments.

4 GENERAL CONSIDERATIONS

In this first part of the thesis, the main topics related to the state of the art regarding the challenges in wood management and evaluation were presented, as well as the technological possibilities that have the potential to facilitate industrial processes. In this context, wood properties, near-infrared spectroscopy, multivariate statistics and the calibration transfer of predictive models are highlighted. In addition, the current scenario for the use of *Eucalyptus* wood was addressed, as well as the challenges and possible solutions for forest-based industries in optimizing production processes.

It can therefore be said that the use of NIR technology in wood science and technology is well-established and has shown promising results, from predicting physical properties such as density and moisture to estimating the contents of the wood's chemical constituents. However, most of the studies reported have considered the raw material at equilibrium moisture in a fully controlled laboratory environment, which is not always possible under industrial conditions. It should also be noted that all the studies used benchtop NIR equipment, with the integrating sphere spectral acquisition mode.

In this way, some limitations on the subject of this study were identified, as there is little in the literature on predicting the basic density of recently felled wood (saturated wood). In addition, the analysis of the potential of portable NIR equipment for predicting the physical properties of wood has not been identified in the literature, nor has the evaluation of calibration transfer between NIR spectrometers in forestry science. According to the literature survey, only Zhang et al. (2022) used calibration transfer and portable NIR spectrometers to estimate lignin content in wood. As a result, no work involving calibration transfer and the physical properties of wood has been carried out to date.

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SECOND PART - ARTICLES

ARTICLE 1

Estimation of the basic density of *Eucalyptus grandis* wood chips at different moisture levels using benchtop and handheld NIR instruments

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Abstract: With the increasing demand for productivity and quality in the forestry sector, near-infrared (NIR) spectroscopy is promising in the monitoring of wood properties, such as density. However, most predictive models are based on spectra acquired in wood at equilibrium moisture content using benchtop equipment. The objective of this study was to evaluate the performance of the NIR instruments in predicting the basic density of *Eucalyptus grandis* wood at different moisture contents. The wood chips were evaluated from saturated conditions (freshly felled) to hygroscopic equilibrium conditions using benchtop and portable NIR instruments. Principal component analysis (PCA) was performed to verify the behavior of spectral data, partial least squares discriminant analysis (PLS-DA) to classify density categories, and partial least squares regression (PLS-R) to develop predictive models. The moisture gradient was not the limiting factor for the statistical modeling. PCA discriminated 99.50% of the variation in the data, while the PLS-DA correctly categorized in the range of 0 to 94% the density classes. The models developed by PLS-R with the benchtop instrument showed a prediction coefficient (R^2) ranging from 0.79 to 0.85 and those with the portable instrument ranged from 0.77 to 0.82; the ratios of prediction deviation (RPD) were 2.20 and 2.45, respectively. Thus, NIR spectroscopy has shown potential application in wood under saturated conditions, regardless of the type of instrument. In the industrial context, the use of a portable NIR instrument could streamline wood characterization without the need for drying and transporting samples to the laboratories.

Keywords: Fiber saturation point, pulp and paper industry, multivariate statistics, quality control, real-time evaluation

1. Introduction

Eucalyptus wood is the main source of short fibers in the pulp and paper industry. In addition, because it is versatile, *Eucalyptus* wood is a raw material widely used in the production of charcoal, reconstituted panels, furniture, and sawn wood (IBÁ 2022). The properties of the raw materials are routinely evaluated by the industries to standardize the quality of their products, especially density, as it is a property that has a direct correlation with the physical-mechanical characteristics of the wood, with direct implications for the behavior, processing, application and transport of the wood (Costa et al. 2019; Amaral et al. 2020). For these analyses, the technologies used are desired to be fast and effective for optimizing their production processes as opposed to conventional methods for determining the properties of wood, which are time-consuming and expensive (Medeiros et al. 2023). For this reason, the use of technologies for wood selection and monitoring is an alternative to increase the efficiency of wood production (Arriel et al. 2019).

Near-infrared (NIR) spectroscopy is a technology based on the intensity of absorption of electromagnetic radiation to analyze the interaction of biological material with the organic bonds constituting the sample. This technique has been used in the forestry sector because it is non-destructive, reliable, and provides real-time responses (Sheppard et al. 1985; Ramalho et al. 2018). The information obtained from NIR spectroscopy is associated with the values of the properties determined in the laboratory. From this, statistical models can be developed to predict such properties; partial least squares regression (PLS-R) is the most commonly used model for analysis (Sandak et al. 2016; Amaral et al. 2021).

Among the wood properties, the density variation has a strong correlation with the spectral signature in the NIR (Hein et al. 2009). In the studies by Rosso et al. (2013), Costa et al. (2018), Arriel et al. (2019), and Amaral et al. (2021), models for estimating the basic density of *Eucalyptus* wood by NIR spectroscopy with a benchtop instrument showed adequate results for the selection of the genetic material in forestry companies.

However, most of the developed models are based on wood at equilibrium moisture content with the environment (~12% dry mass basis); this content varies depending on the region. The NIR application in wood samples with different moisture gradients is important to provide technically viable equipment for real situations, such as in the field, patio, or mat, where the moisture of the wood is not controlled. Since studies regarding this aspect are scarce and a gap in scientific knowledge exists, these aspects can potentially indicate a limitation of the use of NIR in adverse conditions.

Therefore, considering hypotheses such as the independence of basic density estimation from moisture content, the comparable precision between portable and benchtop devices, and the influence of wood anatomical planes on spectra, this study aims to evaluate whether moisture content affects basic density estimation, determine if portable and benchtop devices provide equivalent precision, and investigate the impact of wood anatomical planes on the spectral analysis of *Eucalyptus grandis* wood.

2. Material and methods

2.1. Sample collection and preparation

Eucalyptus grandis tree cultivated in 3 x 3 m spacing at 33 years of age from the experimental plantation at the Federal University of Lavras (Latitude: 21° 14' 30" S; Longitude: 44° 00' 10" W; Altitude: 919 m) was cut following the sampling recommendations provided in ASTM 5536 (ASTM 2017). Next, the stems with a commercial height of 26 meters were sectioned to obtain eight discs in different longitudinal positions (0%, DBH (1.30 meters), 15%, 30%, 45%, 60%, 75%, and 80%) for wood chip making.

A central (diametral) wood portion of each disc, comprising heartwood and sapwood, was removed with a band saw to manually produce wood chips with dimensions of approximately 30 × 30 × 4 mm (length × width × thickness). Chips were produced tangentially to the growth rings by hand using a chisel and mallet. The radial chips were made with the disc wedges, totaling 272 specimens (108 tangential + 164 radial). The chips were identified with a pencil according to the trees, disk, and position from which they were removed to represent the complete variability of the sampled trees.

2.2. Determination of basic density

The basic density was determined according to the ASTM D2395 standard (ASTM 2017). The mass was obtained on an analytical scale (accuracy of 0.001 g), and the volume was measured using the immersion method. Next, measurements were acquired via portable and benchtop NIR spectrometers. The specimens were placed on trays for air drying in a controlled environment at 25 °C and a relative humidity of approximately 60% until the samples reached the equilibrium moisture (~12% dry basis). At each 10% moisture loss, which was monitored by constant weighing, the acquisition of mass and spectra in the chips was performed (Fig. 1), with each measurement

corresponding to a drying stage, totaling 10 stages, with the last stage referring to the anhydrous mass. After reaching equilibrium moisture content, the samples were placed in an oven with a temperature adjustment of 103 ± 2 °C until the dry mass was obtained.

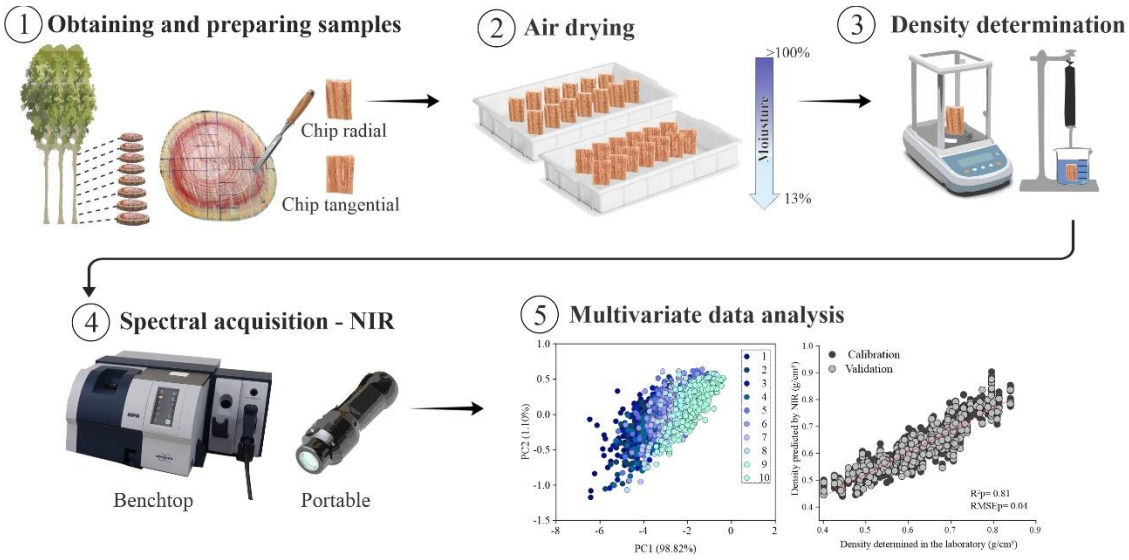

Fig. 1. Flowchart of the study stages.

2.3. NIR Spectral acquisition

The spectra were collected directly on the chip surface using benchtop and portable NIR instruments. The benchtop instrument was a Fourier transform (FT) NIR spectrometer (MPA, Bruker Optik GmbH, Ettlingen, Germany) with its software OPUS v. 7.0. The spectra were recorded by diffuse reflection from the integration sphere. The spectral range used for the calculations was 1112 to 2500 nm (9000 to 4000 cm^{-1}) with a resolution of 8 cm^{-1}, resulting in 1300 spectral variables. Sixteen (16) scans were performed on each wood chip, and then the averages were calculated and compared with the standard to obtain the absorption spectrum of the specimen. Background compensation was performed every 10 min of spectral acquisition and the light leaking from the MPA window was protected.

The portable instrument was an On-site MicroNIR (Viavi Solutions Inc., CA, United States); it was directly set in reflectance mode on the surface of the wood chip. The acquisition range was from 950 to 1650 nm (10526 to 6060 cm^{-1}) with a resolution of 5.6 nm, with the generation of 125 spectral variables. Each spectrum had an average of 16 scans.

Using the point-and-shoot technique, a dark scan and a reference scan were performed approximately every 10 minutes, and the data was collected using the software SpectralSoft Solutions (Viavi Solutions Inc., CA, USA).

A total of 2720 spectra were collected on each piece of equipment, in the benchtop NIR instrument and the portable NIR instrument. The NIR data were associated with the wood density data, and multivariate models were developed.

2.4. Multivariate data analysis

The spectral data were analyzed by multivariate statistics in their original form and with mathematical treatment. Unscrambler v.9.7 software (Camo Software, NJ, USA) was used to perform principal component analysis (PCA) and partial least squares regression analysis (PLS-R). PCA was applied to analyze the grouping of the data according to the wood surfaces and the drying stages, while PLS-R was performed to develop the models for predicting the basic density of the wood by cross-validation (*leave-one-out*) and independent validation (*test set*).

For this purpose, eight latent variables were used to compare the results, and for cross-validation, the data selection method was randomized with eight segment numbers. In the *test set*, the spectra were separated into calibration and validation sets, using 60% of the data to calibrate the models and 40% to validate them, according to the data selection process proposed by Medeiros et al. (2023).

Partial least squares discriminant analysis (PLS-DA) was performed by cross-validation using the software Chemoface version 1.65 (Nunes et al. 2012) to classify the wood density. The density categories for the correlation with PLS-DA were determined based on INDEA (2011); here, very low density was considered to be <0.40 g cm^{-3}, low density was considered to be between 0.40 and 0.55 g cm^{-3}, average density was considered to be between 0.55 to 0.75 g cm^{-3} and high density was considered to be > 0.75 g cm^{-3}.

The ratio of prediction deviation (RPD) was obtained as the ratio between the standard deviation of the values determined in the laboratory and the standard error of the cross-validation. The pretreatments of standard normal variation (SNV) and first and second Savitzky–Golay derivatives with 13 points and 3 polynomials were applied to the spectra to optimize the models. The PLS regression models were evaluated by the root mean square error (RMSE), coefficient of determination (R^2), ratio of prediction deviation

(RPD), percentage of correct answers, and the effectiveness of the mathematical treatment.

3. Results and discussion

3.1 Monitoring of water desorption

The desorption of water in the wood showed uniformity according to the drying stages, even though each stage takes a different number of days to achieve the same water reduction (~10%). This uniformity is associated with the arrangement of the anatomical elements, chemical composition, and origin of the woody material. The maximum moisture values were approximately 100% for the radial chips (Fig. 2A) and approximately 130% for the chips obtained with tangential cuts (Fig. 2B). This variation in the moisture value per chip type could be explained by the order of production of the specimens; specifically, the tangential chips were prepared after the radial chips and were kept packaged for a longer time to avoid exposure to air. In the interval between the drying stages 9 and 10, an abrupt decrease in moisture was observed, which was expected due to obtaining the dry weight, in which the chips were subjected to artificial drying.

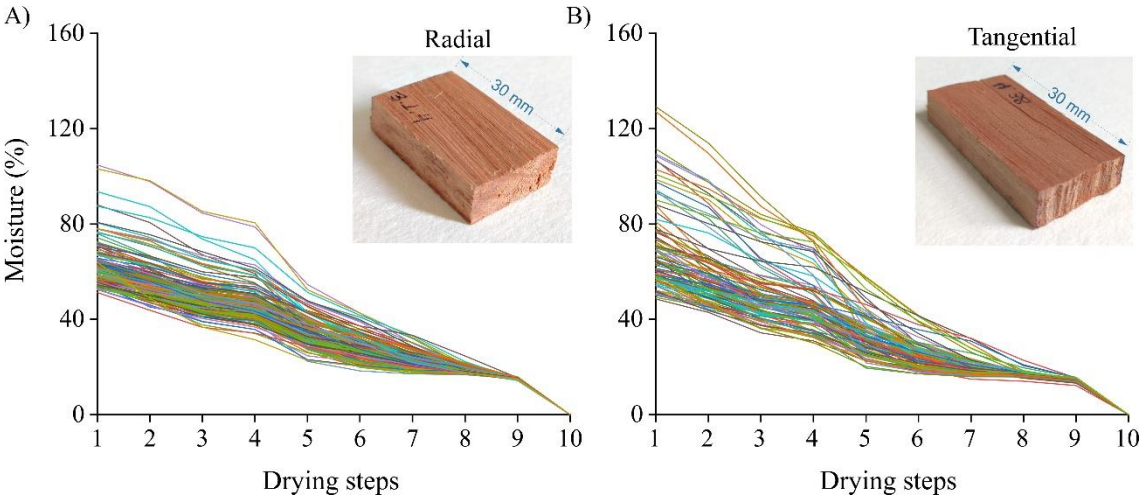

Fig. 2. Moisture reduction of the *Eucalyptus grandis* wood chips obtained from sections in the radial (A) and tangential (B) planes.

These desorption results by the drying step could be explained by the increase in the vapor pressure of the interior of the anatomical elements towards the atmospheric air. As a result, the mass flow of the residual free water was accelerated, which was more

easily released until the fiber saturation point (FSP) was reached (Mascarenhas et al. 2020); here, the moisture of the wood was reduced by approximately 28~30% (Fredriksson et al. 2023). Hereafter, impregnation water was exclusively present and required more energy to be removed because the water-cellulose hydrogen bonds, which are stronger than the water–water bonds, needed to be broken to promote the diffusion of water vapor through the layers of the cell wall.

Specifically, in addition to the different moisture levels that wood can present, the mechanisms of free water and impregnation water need to be considered as a variation factor for density estimates since the types of electrostatic interactions between the molecules and the hydroxyl groups of cellulose and hemicelluloses are distinct (Thybring and Fredriksson 2021; Murr et al. 2022); for example, these interactions can influence the spectral signatures obtained with NIR.

This trend was reported by Amaral et al. (2020), who determined the *Eucalyptus* wood moisture content (approximately 80%) and observed the variations in the correlation factors with the spectral data. The water desorption patterns observed in this study were in agreement with other studies performed with wood and wood derivatives (Moretti et al. 2020; Lima et al. 2022a; Medeiros et al. 2023).

The amplitude of the moisture gradient is important for the prediction of the basic density of wood since the statistical models are trained to cover different moisture conditions in the same sample or different samples of the wood under study. Thus, the fitted model becomes more robust in predicting this property in woods with different moisture contents. According to Honorato et al. (2007), the addition of different sources of information in the data matrix causes the results to be more realistic and has greater applicability in the industrial environment.

3.2. Spectral signatures in the NIR

The chips obtained from the tangential and radial planes showed similar mean spectral profiles at different moisture levels (Fig. 3A). The minimum absorbance observed was 0.3, and the maximum was 0.9 for the benchtop instrument, indicating a low influence from the wood surface in the spectral acquisition.

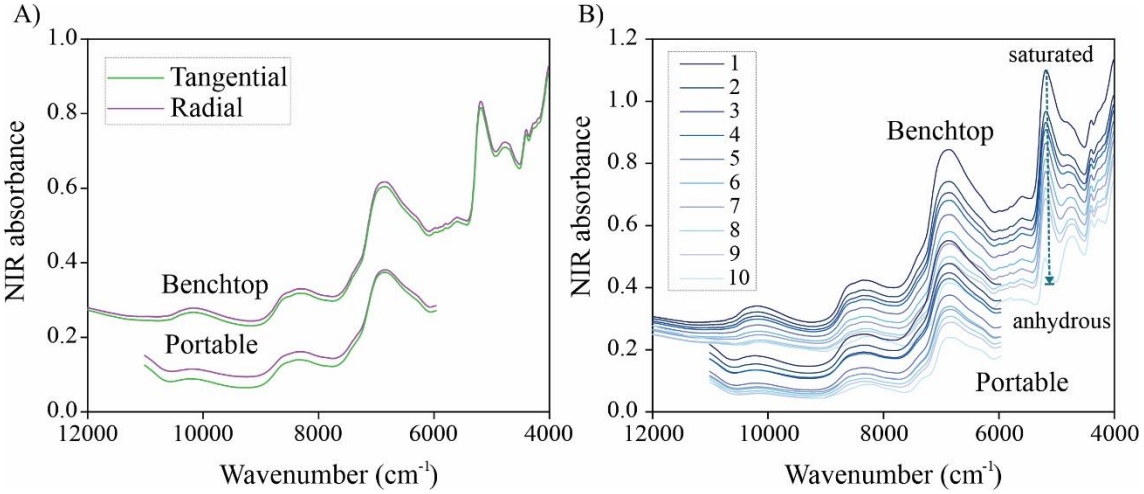

Fig. 3. Mean raw spectral signatures of the chips in different anatomical planes (Fig. 3A) and by drying stage (Fig. 3B) in *Eucalyptus grandis* wood obtained with benchtop and portable NIR instruments.

Based on the analysis of the spectral signatures by type of instrument, the portable NIR had the lowest absorbance values, ranging from 0.1 to 0.4, both in the average signatures by type of surface (Fig. 3A) and in the signatures by drying step (Fig. 3B); this result was reasonable due to the type of lamp, luminous power, radiation intensity and acquisition technology (optical system), which were different from benchtop instrument; the benchtop system obtained spectral acquisition by an integrating sphere and had superior characteristics in the emission of electromagnetic radiation and the wavelength sweep.

From Fig. 3B, the absorbance in the NIR spectra decreased as the water desorption occurred in the wood; this process started at the saturated condition (step 1), reached equilibrium with the environment (step 9), and ended in the anhydrous condition (step 10), with a total of 272 spectra per drying step. This behavior of the signatures according to the water content was in agreement with the results found by Medeiros et al. (2023), who analyzed the desorption of water in cellulosic pulp with a benchtop NIR instrument.

The main absorbance peaks were in the waveband of 6800 cm^{-1} in the spectra from the benchtop NIR instrument and in the waveband of 5200 cm^{-1} in the spectra from the portable instrument. These spectral regions were related to the variation in moisture in the wood. According to Nisgoski et al. (2016), Costa et al. (2019), and Amaral et al. (2020), moisture intensified the interaction of electromagnetic radiation with the vibrations of hydroxyl groups, increasing the absorbance and information content of the

material. For this reason, the absorbance decreased in increasing order of the drying steps in the spectral signatures.

The bands from 7000 cm^{-1} to 6800 cm^{-1} were attributed to the amount of cellulose, hemicelluloses, and lignin that corresponded to the main constituents of wood (Schwanninger et al. 2011). Specifically, the spectral signatures obtained for *Eucalyptus* wood under different moisture conditions were by reports in the literature for wood.

3.3. Principal component analysis

Similar to the spectral signatures, in the PCA scores obtained by first derivative spectra, the wood surfaces showed no visual difference regardless of the anatomical planes of the wood, since the variables obtained for each type of instrument remained superimposed (Fig. 4A and 4B). Corroborating these results, Costa et al. (2018) analyzed NIR spectra in the three anatomical planes of the wood and reported the same overlap of variables in the PCA plots considering the radial and tangential planes.

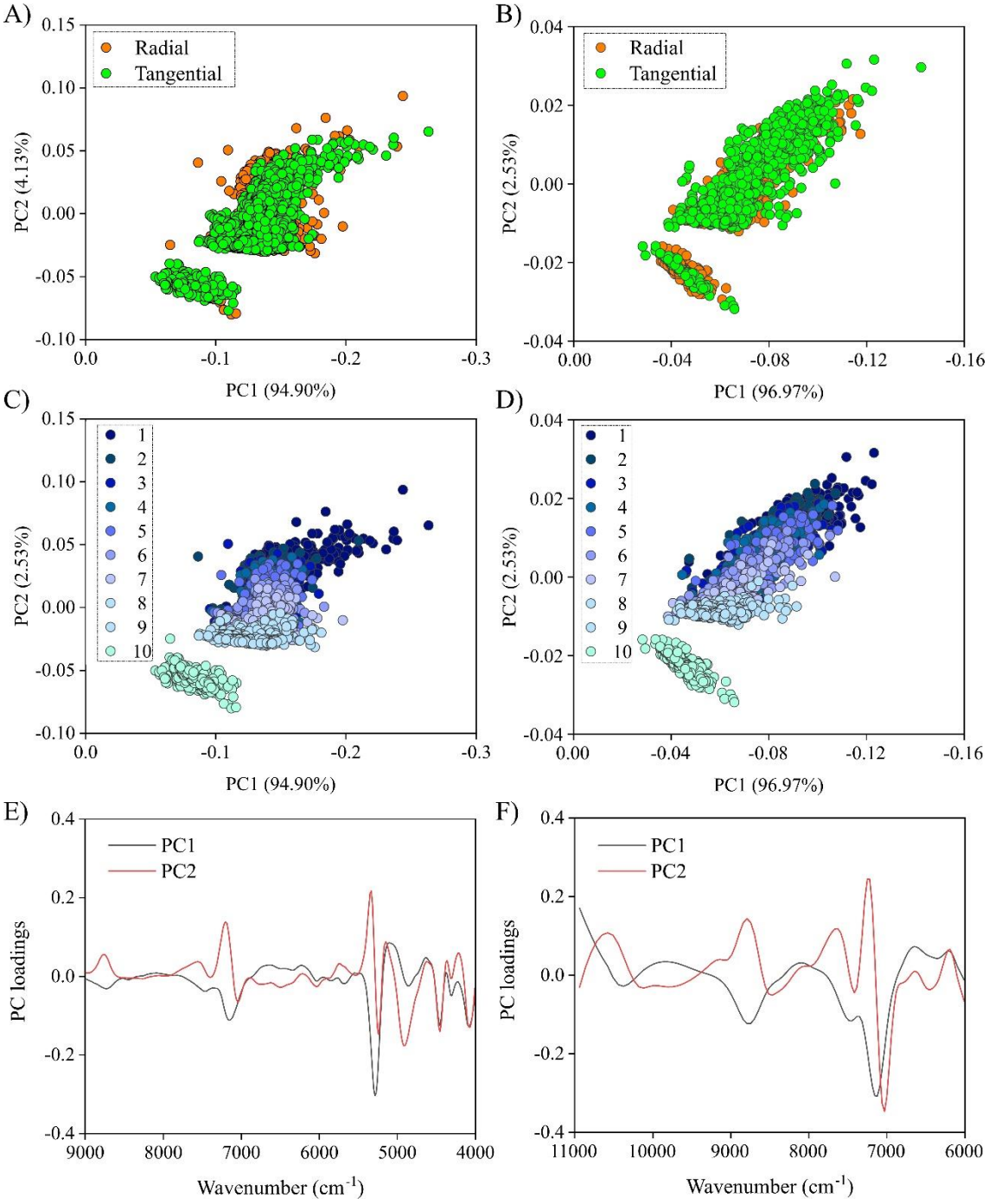

Fig. 4. PCA scores and PC loadings by wood surface and drying stage for the spectra treated with the first derivative from the benchtop NIR (A, C, and E) and the portable NIR instrument (B, D, and F).

Although there are differences in the arrangement of cells in the different anatomical planes of wood, the chemical composition of wood does not significantly vary (Rowell 2013). This may explain the results because the NIR spectra are associated with

the vibrational patterns of the chemical groups present in the molecules that compose the wood.

However, in some cases, the greater exposure of a particular group of cells may influence the spectral signatures. For example, in woods that contain vessel elements filled with tyloses and the presence of radial and axial parenchyma with mucilaginous or oily content, peaks are potentially recorded in spectral bands more characteristic to a particular anatomical plane to the detriment of others (Braga et al. 2011; Karlinasari et al. 2021). In the present study, these effects were not observed, probably because the *Eucalyptus* wood contained heartwood and sapwood with low concentrations of extractives and other substances of secondary metabolism (Arisandi et al. 2023).

The chip drying stages followed a grouping trend according to moisture levels, starting in saturated conditions until reaching equilibrium with the environment (Fig. 4C and 4D). In the stage with more humid wood, greater dispersion of the samples was observed than in the drier stages. This trend was observed in the studies by Medeiros et al. (2023) in cellulosic pulp and by Amaral et al. (2020) in the examination of wood chips.

These variations were explained by the heterogeneity of the wood because, at moisture levels above the FSP, the amount of water varied depending on the inter- and intracellular spaces (Gezici-Koç et al. 2017). Since the examined samples were from different pith-bark and base-top positions, variations in the proportions of juvenile wood and mature wood, late wood and early wood, and, consequently, wood porosity were very likely. The development of models to discriminate the drying stage of wood is important in decision-making in forest-based industries because moisture directly interferes with several productive sectors, such as the production of pulp, furniture, panels, and charcoal.

In the principal component analysis loadings plots, the reflectance peaks were similar to those in the spectral signatures. Regarding the performance of the instruments, the portable NIR showed higher values than the benchtop NIR, in which the sum of principal component 1 and principal component 2 discriminated 99.50% of the information. In the benchtop NIR, 94.90% of the explanation of the data variation was represented by component 1, which was close to the portable instrument. Using PCA, Amaral et al. (2020) indicated that the discrimination of the drying steps in wood with benchtop NIR spectra was higher than 99.85%; these results were similar to those of this study. However, there are no reports of the use of portable NIR instruments for this purpose, highlighting the unprecedented nature of this information.

3.4. Partial least squares discriminant analysis

Basic density classification models for different wood moisture contents were fitted by PLS-DA based on the raw spectra collected from the NIR instruments (Table 1). In the confusion matrix, for the "very low" density class, no prediction success was achieved, regardless of the instrument used. For the "medium" class, success was obtained on the order of 93% and 94% for the benchtop NIR and portable NIR instruments, respectively.

Table 1. Confusion matrix of the partial least squares analysis for the classification of wood density at different moisture contents in the two NIR instruments.

		Classification of wood density						
		Very low	Low	Mean	High	N of samples	%Success	Total
Real class								
Benchtop NIR Instrument								
Predicted class	Very low	0	30			30	0	78%
	Low		336	323		659	51	
	Mean		126	1664		1790	93	
	High			199	35	234	15	
Portable NIR Instrument								
Predicted class	Very low	0	30			30	0	75%
	Low		329	330		659	50	
	Mean		106	1684		1790	94	
	High			200	34	234	14	

A possible understanding of these results can potentially be based on the explanations by Kollmann and Côté (1968). The authors mentioned that low-density wood has cells with thinner cell walls and more pits. This, in turn, can induce multisurface reflectance, thus reducing the potential of electromagnetic radiation to capture the pertinent information. The presence of water can also influence this result because water vapor is capable of generating variations in the spectral signal due to the instability of the molecular geometry.

The success rate of the model developed with the benchtop instrument was 78%, which was slightly higher than that of the portable instrument with a 75% accuracy. Thus, the two instruments were able to classify the basic density of wood. Notably, the choice of instrument is influenced by the application environment and the nature of the sample, directly in the field or in the laboratory. In the study by Lima et al. (2022b), PLS-DA models for wood basic density classification were adjusted and their reported

classification success rates were higher than 90%. However, the studied wood was at the equilibrium moisture content (~12%).

For the adjusted models to be promising for controlling the quality of raw materials in forestry companies, it would be necessary to expand the database with a greater range of variation, since the accuracy of the models was around 70%. Even so, although the statistical parameters in the "very low" and "low" moisture classes have shown inferior performance in predicting density, there is potential for using this tool to predict moisture in different packaging conditions of wood particles. This is in line with what was presented by those in the wood pulp and paper processing production chain, the ranges of density values that must be monitored are between 0.50 and 0.75 g cm-³, falling within the range indicated in this study. Thus, even though the classification has less predictive capacity for the lower classes, our results fell within the moisture range that industries monitor wood in the pulping process and standardization of the final product.

3.5. Partial least squares regression

The models fitted by PLS-R to predict the basic density of the wood at different moisture levels exhibited coefficients of determination ranging from 0.77 to 0.86 in the different validations (Table 2).

Table 2. Statistical parameters of the PLS-R models to predict the basic density of *Eucalyptus grandis* wood at different moisture levels.

Instrument	Surface	Treatment	R^2cv	RMSEcv	R^2p	RMSEp	RPDcv
Benchtop	RD	untreated	0.817	0.038	0.810	0.053	2.20
	TG		0.794	0.054			
	RD	1°D	0.833	0.036	0.831	0.037	2.45
	TG		0.857	0.045			
	RD	2°D	0.798	0.040			
	TG		0.831	0.049			
	RD	SNV	0.797	0.040			
	TG		0.796	0.054			
Portable	RD	untreated	0.815	0.038	0.813	0.038	2.33
	TG		0.771	0.057			
	RD	1°D	0.780	0.041	0.833	0.048	2.44
	TG		0.827	0.049			
	RD	2°D	0.777	0.042			
	TG		0.815	0.051			
	RD	SNV	0.788	0.041			
	TG		0.815	0.050			

R^2c - coefficient of determination for calibration; RMSEc - root mean square error for calibration; R^2cv - coefficient of determination for cross-validation; RMSEcv - root mean square error for cross-validation; R^2p - coefficient of determination for prediction; RMSEP - root mean square error for prediction; RPDcv - the ratio of performance to deviation for cross-validation. RD – Radial; TG – Tangential. 1D - first derivative; 2D - second derivative; SNV - standard normal variate.

In the spectra without mathematical treatment, the most appropriate statistical parameters were obtained for the radial surface on the benchtop instrument, with a coefficient of determination of 0.81 in the cross-validation. The error associated with the models followed similar values for RMSEcv and RMSEp, demonstrating uniformity in the data matrix. This consistency was observed in the cross-validation and independent validation, in which the correlation values did not differ from each other.

When applying mathematical treatment to the spectra, the models showed an increase in the coefficient of determination, and the tangential surface began to exhibit the best statistical values for both instruments and validation methods. The first derivative was the most efficient mathematical treatment, except for the radial surface in the portable instrument. In the performance deviation relationship (RPD), the models maintained low variation, with values between 2.20 and 2.45, meeting the predictions cited in the literature (Williams and Sobering 1993; Fujimoto et al. 2007). The prediction coefficient of the models and the deviation performance ratio were selected according to the lowest error associated with the correlation for each piece of equipment and surface.

Regarding the type of instrument, the models showed low influence, but the portable NIR exhibited a slight reduction in the statistical parameters in certain correlation adjustments. According to Santos et al. (2020), this decrease in the performance of the models could be explained by the spectral readout area since the integrating sphere was approximately 10 mm in diameter, while the optical system had a point area, with less representation of the wood surface. However, both instruments were efficient in predicting the basic density of wood at different humidities.

Amaral et al. (2021) predicted the basic density of *Eucalyptus* wood at equilibrium moisture with original spectra in a benchtop NIR instrument; their data showed R^2cv= 0.70, RMSEcv= 0.4 and RPDcv= 1.83 on the radial surface of the wood, and the data treatment with the first derivative showed improvements in the models. Siesler et al. (2008) recommended the use of mathematical treatments in spectral data in

the NIR to minimize noise in the spectral signal and indicated that the first derivative was one of the main treatments because it reduced the systematic displacement of the baseline.

Vimal et al. (2014) used NIR spectroscopy to estimate the basic density of *Eucalyptus tereticornis* wood in the green condition (30% moisture) and evaluated the spectral acquisition mode (integrating sphere and optical fiber) and the effect of the radial and tangential surface on the model calibration. The best coefficients of determination were obtained on the radial surface and on the integrating sphere, which corroborated the results of this study.

Fig. 5 shows the plots of the PLS-R validation of the data with first derivate mathematical treatment to predict the basic density of *Eucalyptus grandis* wood at different moisture levels, and the property correlation was performed with the values determined in laboratories via conventional methods and predicted via NIR technology with the benchtop and portable instruments and the scattering trends for the radial and tangential surface of the wood.

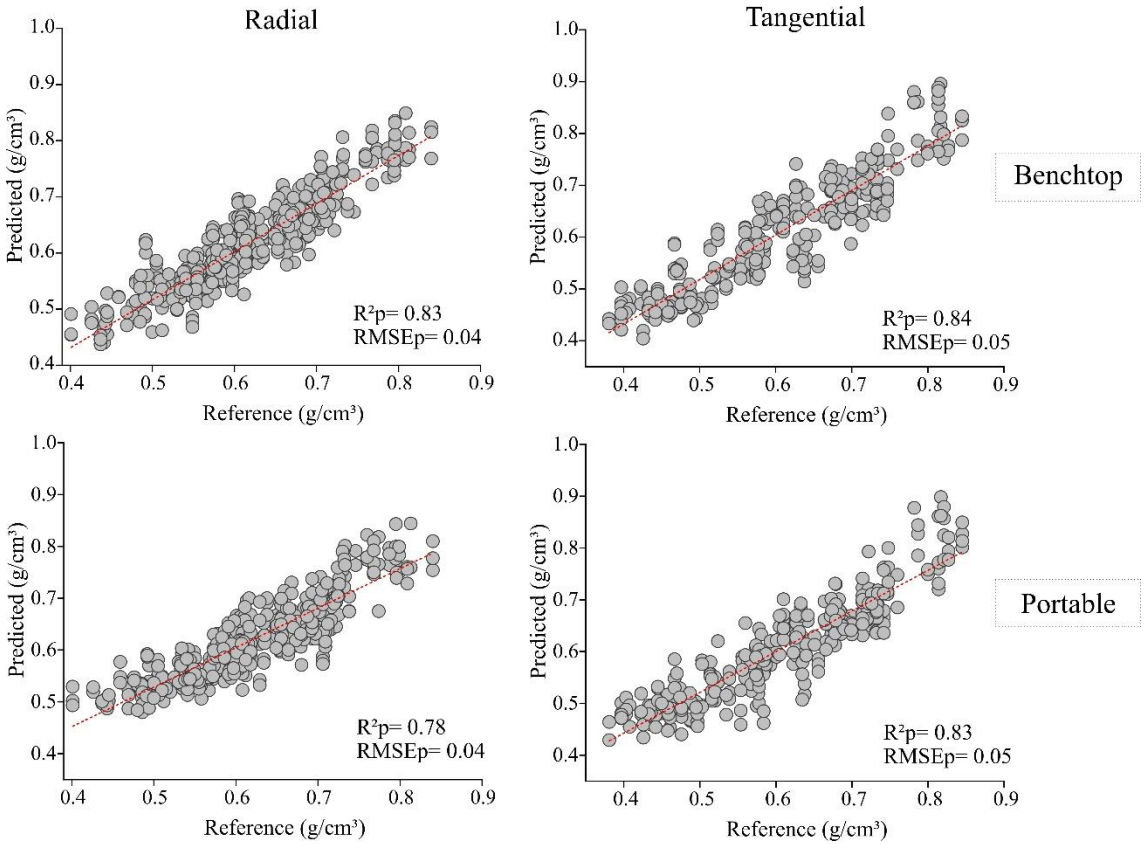

Fig. 5. Plot of the PLS-R validations treated with first derivative to predict the basic density at different moisture contents by type of instrument and wood surface.

Visually, the effect of the instrument and the wood surface was negligible; however, when analyzing the predicted values, a reduction in the coefficient of determination was observed on the radial surface with the benchtop and portable NIR. Costa et al. (2018) reported that the spectral reading on the radial surface included information from several years of wood formation, while in the tangential direction of the shaft, the representativeness of the wood formation was from a shorter period, which agreed with our model results. In the study by Costa et al. (2019), models developed with a benchtop NIR instrument to predict wood moisture in different water proportions also showed adequate prediction values, corroborating our basic density results for wood.

Based on the analysis of the coefficient of determination of the PLS-R models by drying stage, the wood moisture did not affect the R^2 values of the cross and independent validations, regardless of the instrument or surfaces, reinforcing the potential of NIR technology for industrial use (Fig. 6). The error associated with the PLS-R models showed uniform variation, with values between 0.02 and 0.06 g/cm³, in the cross-validation and the prediction.

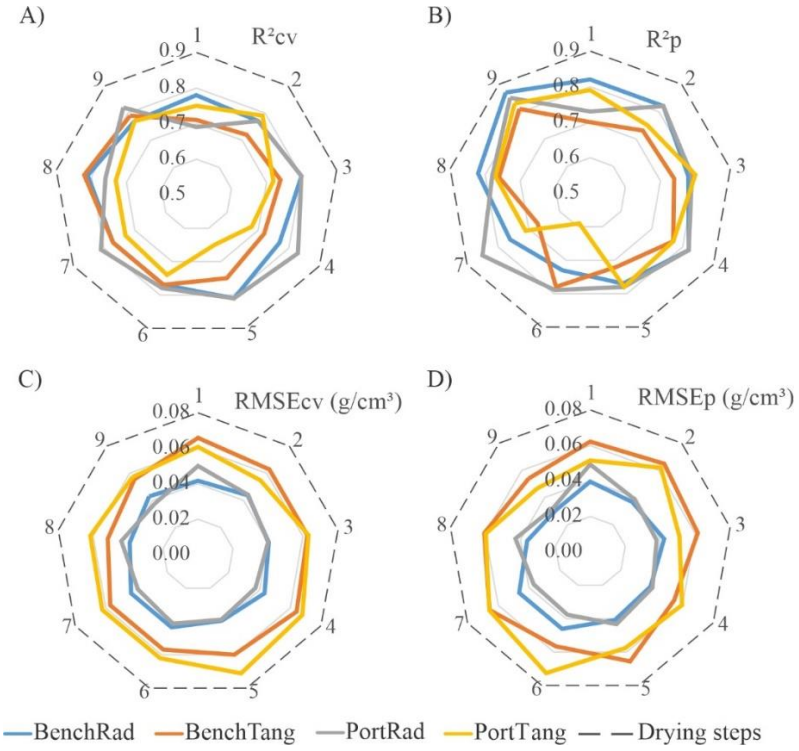

Fig. 6. Graphs of the prediction of the basic density per drying stage (stage 1 - wetter to stage 9 - drier) in different surfaces and instruments in the cross-validation (A) and in the independent validation (B) and their respective errors (C and D).

The radial surface maintained the best statistical performance in all models, and the tangential surface presented reduced values. This answers one of the hypotheses outlined in this research, considering that for the wood under study, the anatomical plane can contribute to improving estimates. This can be seen more clearly when analyzing the most significant statistical parameters observed in drying step 9; here, the chips were at equilibrium moisture content with the environment (~12%). However, the difference in values between the drying stages was practically insignificant because the robustness of these models was suitable for practical field and laboratory applications.

Arriel et al. (2019) estimated the basic density of *Eucalyptus* wood at equilibrium moisture using benchtop NIR and reached $R^2cv= 0.55$ and $RMSEcv= 0.26$ for the spectra treated with the first derivative and collected on the transverse surface. In the same way, Costa et al. (2018) predicted the basic density with $R^2p= 0.73$ and $R^2p= 0.76$ on the tangential and radial surfaces, respectively.

Thus, the radial surface had the most reliable results, in agreement with the results of this study. This could be explained by the fact that the radial section of *Eucalyptus* wood was characterized by greater exposure of the fiber mass, which was highly correlated with wood density (Pirralho et al. 2014). The tangential section had part of its exposed area covered by voids in the lumens of the radial parenchyma, causing discontinuity of the woody mass. Thus, this discontinuity in the tangential section could potentially cause a reduction in the spectral interaction with the cell wall of the fibers.

By observing the global model plot and its β-coefficients for the basic density of *Eucalyptus grandis* wood in the NIR instrument, verification of the predictive accuracy for the models was fairly possible, and the model accuracy in different wood moistures and surfaces was maintained (Fig. 7).

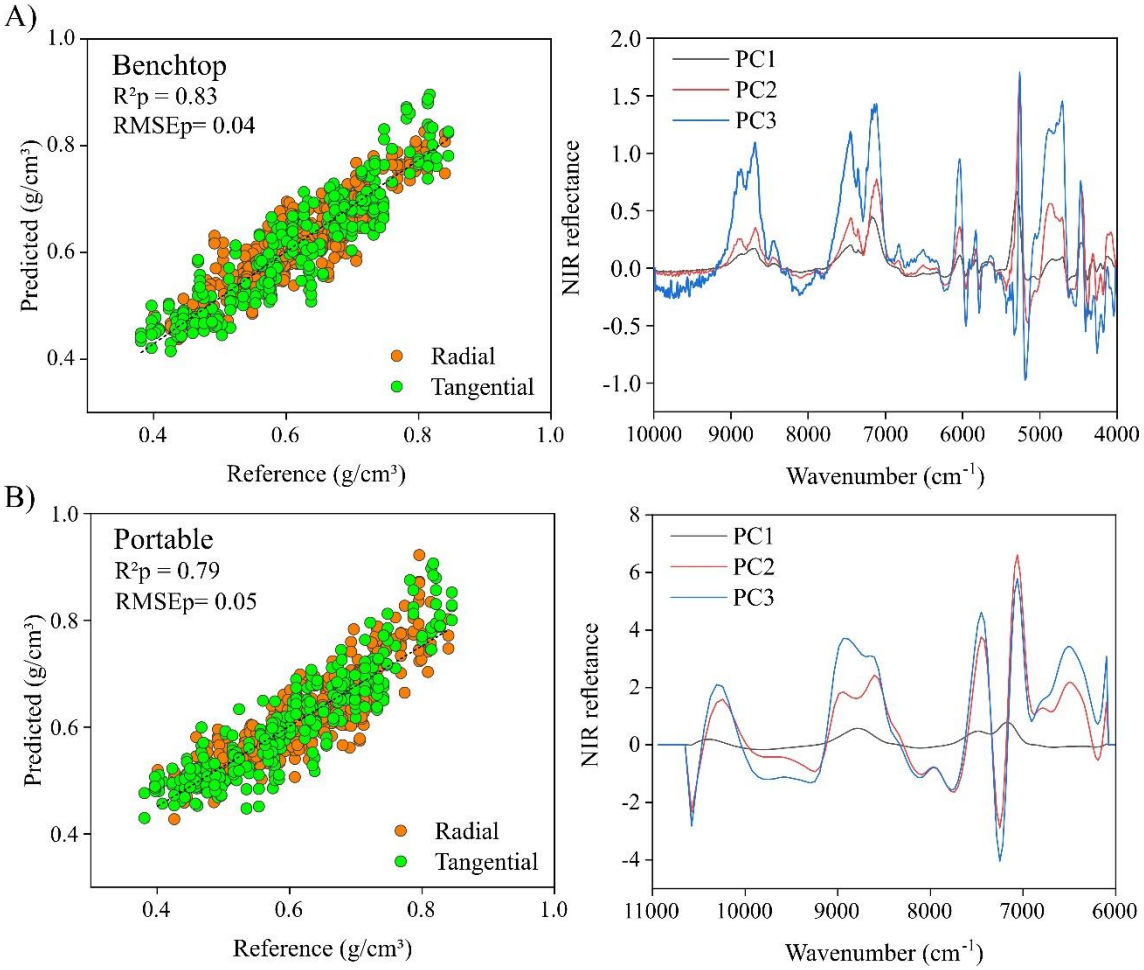

Fig. 7. Global model for the basic density of *Eucalyptus grandis* wood in benchtop (A) and portable (B) NIR instruments, with spectra treated with first derivative.

In the β-coefficients of the global model, the most significant refletance peaks were in the band 5500 cm^{-1} and the range of 6800 to 7200 cm^{-1}, for benchtop and portable equipment, respectively. Medeiros et al. (2023) developed a global model for the prediction of cellulosic pulp moisture at different moisture contents and pulp types using benchtop NIR, and the results were also promising, with an R^2 of 0.95, RMSEp of 0.1 g/cm^3 and β-coefficients with peaks in the band 5500 cm^{-1}, with reflectance NIR values ranging from -1.50 to 2.25. Honorato et al. (2007) report that the global model enhanced the validation ability to predict the property of interest due to possible variations in data analysis.

Visually, the PLS-R correlations of the instruments were similar, with equivalent linear trend adjustments and minimal differences in the prediction values. Thus, in the results of the present study, the proposed objectives were achieved because the instruments demonstrated equivalent performance, with the main difference being in their

handling. Thus, the benchtop and portable NIR instruments could be used to estimate the basic density considering different moisture contents and wood surfaces.

It is worth noting that this study has limitations regarding species diversity, as in this work only *Eucalyptus grandis* wood was considered. Additionally, the storage and drying conditions of the wood under study were standardized, unlike what occurs in sawmills and wood storage yards, where the raw material is eventually exposed to photodegradation, oxidation, and deterioration by xylophagous agents.

Therefore, it is necessary to carry out more studies considering the sources of variation to better understand the influence of these factors on the predictions of wood technological parameters, especially density. Considering that this parameter varies between species, and between different positions of the trunk (pith-bark and base-top), it is influenced by management and silvicultural treatments, varies between growth rings, and is also influenced by nutritional parameters and water availability. This, in turn, will reflect different spectral responses that can influence the obtaining of predictive models from NIR spectroscopy.

4. Conclusion

The PLS-R models developed with the NIR spectra showed adequate coefficients of determination (R^2 of 0.77 to 0.85) to predict the basic density of *Eucalyptus grandis* wood at different moisture levels, and the performances of the models were not affected by moisture content. Thus, based on these models, the basic density could be fairly estimated in woods recently cut or after the desorption of water in industrial yards, contributing to the optimization of production processes.

The radial surface of the sample, with the spectra without mathematical treatment, was the most suitable for spectral collection, regardless of the NIR instrument used. The instruments showed equivalent application potential. However, the portable NIR instrument has a lower cost, and its operation is more dynamic than that of the benchtop instrument. Thus, based on the results from this study, the acquisition of reliable estimates with different NIR spectrometers was possible.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data Availability

Data will be made available on request.

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ARTICLE 2

Impact of moisture and NIR sensors on calibration transfer between predictive models of *Eucalyptus grandis* wood density

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Abstract: The aim was to evaluate the potential for model transfer between NIR equipment for predicting the basic density of wood at different moisture. Samples of *Eucalyptus grandis* wood were produced. Basic density was determined by the ratio between anhydrous mass and saturated volume. Benchtop and portable near-infrared spectra were collected on freshly cut wood, at the fiber saturation point and at equilibrium moisture. A script was developed in the R software to make the NIR equipment matrices compatible, totaling four matrices: two with raw dimensions and two with standardized dimensions. PCA and PLS-R analyses were carried out to evaluate the data and obtain the models, which were validated using the leave-one-out and test set methods. Calibration transfer was applied to analyze the models under different instrumental variations. The models developed in each matrix showed R^2 ranging from 0.74 to 0.92 and RMSE from 0.03 to 0.06 g/cm³, showing potential for predicting density from raw and compatible data. In the calibration transfer, the R^2p reached 0.93 and the RMSEp 2.83 g/cm³. The FSP moisture models showed the best statistical parameters, and the calibrations of the benchtop equipment are more promising for transfer to other equipment.

Keywords: Wood free water, wood impregnation water, portable and benchtop NIR, cell wall, fiber saturation point, wood drying.

1. Introduction

Artificial intelligence-based tools associated with equipment that enable obtaining information about materials in a non-destructive manner have demonstrated great potential for assessing the quality of raw materials in forestry industries [1]. One of the most promising technologies is near-infrared (NIR) spectroscopy, through which it is

possible to estimate different properties of wood and its derivatives [2,3]. The main advantages of this technique are speed, minimal sample preparation and reliability of information [4].

NIR spectral data acquisition is typically performed with benchtop equipment, which requires a controlled environment, reducing usability in industrial settings or forestry yards [5]. An alternative to this problem is the use of small portable devices, which are lightweight, operable with one hand, and can be carried for field measurements [6]. However, these devices have lower resolution and a shorter wavelength range, which consequently reduces their analytical capacity when compared to benchtop equipment [7].

In view of this, efforts are being made to standardize the performance of models developed on portable devices so that they are equivalent to benchtop calibrations. This is because generating and testing the performance of calibrations is an expensive and time-consuming procedure. As a result, several approaches to transfer NIR calibrations from one device to another have been implemented [8,9,10]. These methodologies aim to correct the effect of instrumental variation in the collected spectra before transferring the calibration between NIR sensors.

However, there are very few studies on calibration transfer for predicting wood properties. For this purpose, only Zhang et al. [6] obtained models from calibration transfer between models developed in portable NIR equipment to estimate the lignin content in pulp of *Pinus massoniana*, *Cunninghamia lanceolate*, *Acacia*, *Eucalyptus* and *Poplar*. The best results obtained by the authors were achieved when they used direct standardization and direct standardization by parts, with R^2 greater than 0.79.

In addition, the studies available in literature do not meet the daily demands of industries that handle large quantities of wood, since they are performed with benchtop NIR equipment and with the wood at equilibrium moisture content. These conditions are completely different from those in pulp and paper mills or reconstituted wood panel factories, where the material is generally stored in stacks with high heterogeneity, variability in the exposure of anatomical planes, wide moisture gradients and uneven contact with atmospheric air.

These conditions, in turn, generate differences in moisture and density between wood samples, which makes it impossible to use classical methods to quantify these parameters and, at the same time, require instruments that can be used in the field with

the same estimation accuracy as laboratory equipment, but the number of studies in the literature that address this approach is scarce.

For example, the research conducted by Medeiros et al. [11] monitored water desorption in different bleached and unbleached pulps of *Eucalyptus* sp. and *Pinus* sp. Medeiros et al. [12] also addressed the feasibility of estimating the density of *Eucalyptus* sp. wood under different moisture conditions using benchtop and portable NIR equipment. In both studies, the authors found that the NIR technique at different moistures and using different sensors is very promising for estimating the parameters of wood products with potential for industrial application.

Even so, there is a clear lack of information in the literature on calibration transfer for estimating wood properties in conditions other than the laboratory environment, especially for wood density, which is one of the most important properties in monitoring the quality of raw materials for forest-based industries, given the limitations of classic methods for determining wood density, which include factors such as high cost, time-consuming processes, susceptibility to operational errors, the need for robust sampling in certain cases and the difficulty of obtaining parameters in real time.

Thus, the innovation of this work lies in the expansion of scientific knowledge on how to carry out the calibration transfer technique between models adjusted from different NIR equipment to estimate wood density in different moisture conditions. Another innovative aspect is the use of portable equipment, which is more suitable for field applications, allowing flexibility, mobility and agility in data collection outside the laboratory environment.

In view of the above, it is necessary to develop a standardized method for evaluating calibration transfer performance in advance to optimize the selection of NIR equipment for wood product applications. To this end, multivariate statistics are used to find a prediction of the performance of a handheld device using existing (primary) benchtop NIR spectra [5].

In this context, considering the hypothesis that moisture does not influence density estimates and that calibration transfer techniques can maintain the predictive capacity of models generated from benchtop NIR equipment to the detriment of portable equipment, the objective of this study was to evaluate the potential of calibration transfer between models adjusted with benchtop and portable NIR equipment for predicting the basic density of *Eucalyptus grandis* wood at different drying stages.

2. Material and Methods

2.1 *Obtaining and preparing samples*

According to the criteria established in ASTM D 5536-17 [13] and considering a commercial height of 26 m, discs were removed from *Eucalyptus grandis* trees along the trunk. The trees were approximately 33 years old and were grown in a spacing of 3 m x 3 m in an experimental plantation at the Federal University of Lavras (Latitude: 21º 14' 30" S; Longitude: 44º 00' 10" W; Altitude: 919 m). A central baguette containing heartwood and sapwood was removed from each disc. Using a band saw, 95 wooden cubes measuring 30 mm × 30 mm × 30 mm (length × width × thickness) were produced from this material, rigorously oriented in terms of the transverse, longitudinal-radial and longitudinal-tangential planes. These cubes were properly identified by their relative position for each tree used.

2.2 *Determination of basic density and moisture on a dry basis*

The basic density was determined according to standard D2395-17 [14]. After saturation of the wood cubes (green), they were weighed on an analytical balance and subjected to spectral readings using portable and benchtop NIR. The wood cubes were then arranged on trays for air drying in a climate chamber at a temperature of ~25 °C and relative humidity of ~60% until reaching the equilibrium moisture - EMC (~12%). The samples were then sent to air circulation at a temperature of 103 °C ± 2 °C to obtain anhydrous mass. The moisture of the material was monitored by successive weighings, considering a difference of ~10% moisture loss at each drying stage.

2.3 *NIR spectral acquisition*

At each drying stage described, starting from the green condition (above PSF), spectra were obtained directly on the cross-sectional surface of the cubes, using benchtop and portable devices (Fig. 1).

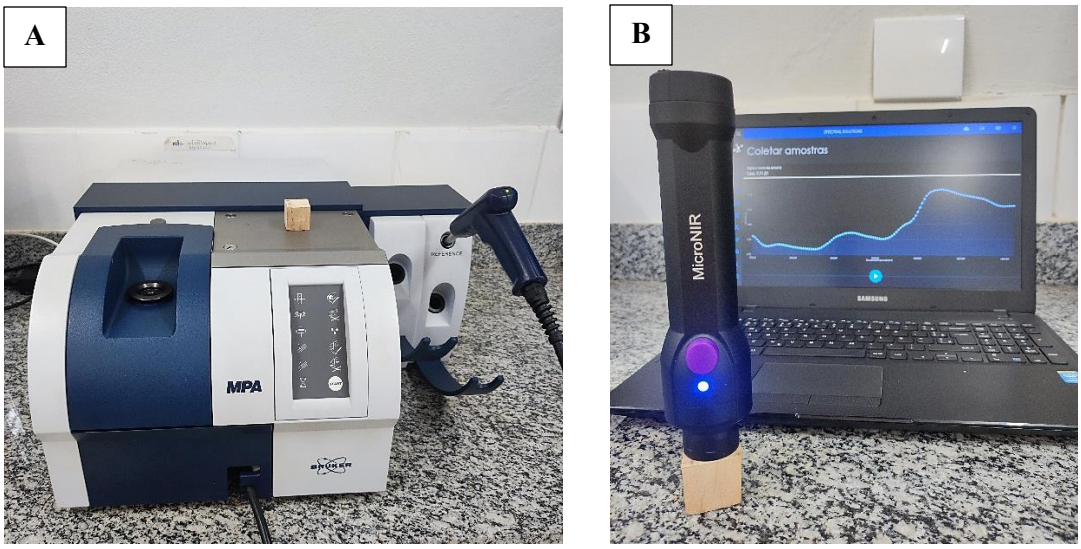

Fig. 1. Benchtop (A) and portable (B) near-infrared spectrometers used to collect spectral data from *Eucalyptus grandis* wood samples.

Benchtop: the stationary equipment was an FT-NIR spectrometer (MPA, Bruker Optik GmbH, Ettlingen, Germany) with OPUS v.7.0 software. Spectra were recorded by diffuse reflection from an integrating sphere, with the reference background taken every 30 minutes. The spectral range was from 800 nm to 2500 nm (12500 cm^{-1} to 3999 cm^{-1}) with a resolution of 8 cm^{-1}, resulting in 2203 spectral variables, but only 125 variables were used in the development of the models to standardize the sensors. For this, 16 scans were performed on the wooden cube and then the averages were calculated and compared with the standard to obtain the absorption spectrum of the sample.

Portable: The portable MicroNIR On-site equipment (Viavi Solutions Inc., CA, USA) was used. The device was controlled and data manipulated using the SpectralSoft Solutions software interface (Viavi Solutions Inc., CA, USA). The MicroNIR was set to reflectance mode and used directly on the surface of the cubic wood samples. The acquisition range used was 950 nm to 1650 nm (10526 cm^{-1} to 6060 cm^{-1}) with a resolution of 5.6 nm, generating 125 spectral variables. Thus, each spectrum represents an average of 16 scans. Using the "point and shoot" function, a dark scan and a reference scan were performed approximately every 10 min.

In total, 285 spectra were collected with the portable and bench NIR device separately, with 95 spectra from each moisture condition (Green, FSP and EMC). The spectral data was associated with the basic density of the wood and multivariate models were developed.

2.4 *Compatibility of data matrices and calibration transfer*

Preliminarily, it was necessary to match the data matrices because the NIR spectra produced by the benchtop and portable devices are different in at least three aspects: 1) spectral range, 2) spectral resolution, and 3) starting point, as shown in Table 1.

Table 1

Characteristics of the benchtop and handheld NIR sensors used in the study.

Parameter	Benchtop	Portable
Spectral Range	800 nm to 2500 nm (12500 cm^{-1} to 3999 cm^{-1})	950 to 1650 nm (10526 cm^{-1} to 6060 cm^{-1})
Spectral Pass	1.07 nm / 8 cm^{-1}	6.2 nm / 36 cm^{-1}
Starting Point	1110 nm / 9010 cm^{-1}	950 nm / 10526 cm^{-1}
Ending Point	2500 nm / 3999 cm^{-1}	1650 nm / 6060 cm^{-1}
Number of Variables	1300	125

In this study, the idea was to record spectra on the same material and make the spectral matrices of the equipment compatible so that the models could be validated using spectra from different sensors. Using the R 4.4.0 software [15] and the rchemo package [16] to standardize the data, a script was developed to interpolate the spectral data between the benchtop and portable devices, so that the spectra started from the same point and had the same structure (range, resolution and starting point).

Initially, spectral acquisition was performed on each sensor separately. In the benchtop NIR equipment, the matrix with 2203 variables, the standard sensor dimension, was obtained. However, for studies with wood, the information is considered between the wave range 9010 to 3999 cm^{-1}, totaling 1300 variables (B 1300). From the B 1300 matrix, 125 variables compatible with the portable sensor (B 125) were selected and, through mathematical interpolation, using the R script, the B-P 125 matrix was reached, which corresponds to the 1300 matrix standardized with the dimensions of the portable equipment. Next, the P 125 matrix was obtained, referring to the raw data from the portable sensor, with 125 variables. From this matrix, using the R script, the P-B 1160 matrix was acquired, so that the portable sensor matrix becomes equivalent to the benchtop NIR. Finally, from this matrix, he selected 125 variables, obtaining the matrix P-B 125.

The number of spectral variables was adjusted to 125 to make the most of the information from the portable NIR spectrometer, which had the narrowest spectral range

(Fig. 2). In addition, adjusting the other matrices to 125 variables is a way of testing the effect of the low resolution on the quality of the spectra from the bench equipment and of understanding the behavior of the model when the spectral information is drastically reduced to just 125 variables. Thus, the matrices that were obtained by mathematical interpolation are considered to be compatible or corrected, on the contrary they are the original matrices, originating from the crude spectra.

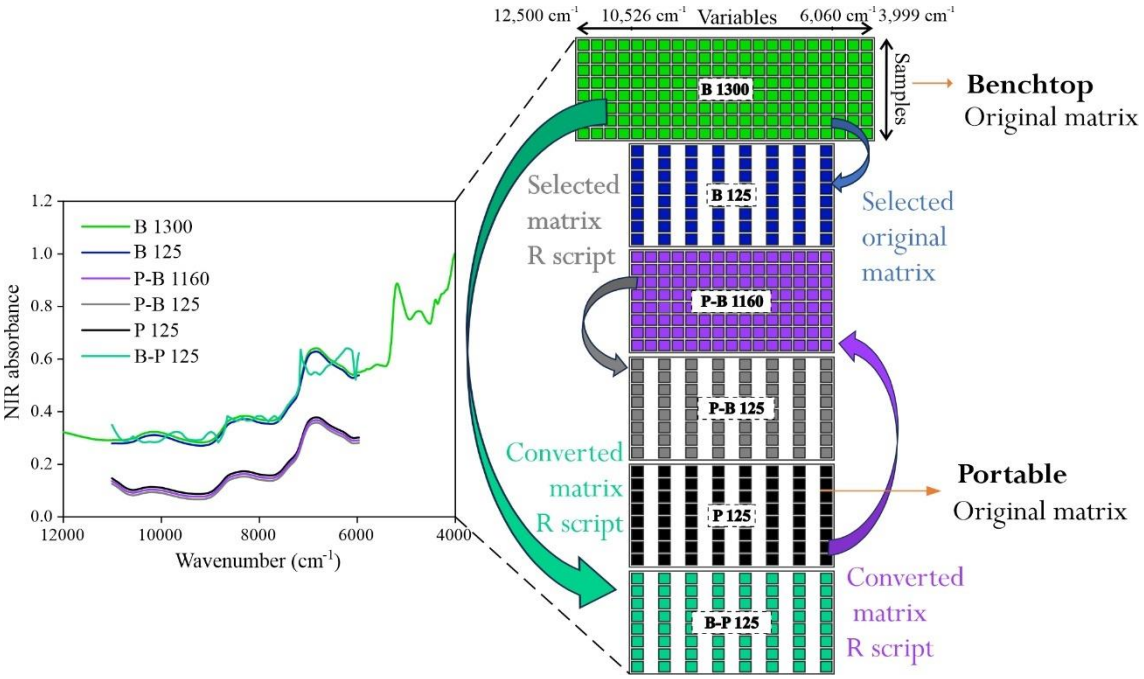

Fig. 2. Standardization of matrices from NIR equipment. Where: B 1300= raw data from the bench equipment. B 125= raw data from the bench equipment with 125 selected variables. P-B 1160= data from the portable equipment expanded to be equivalent to the bench equipment by the R script. P-B 125= data from the P-B 1160 matrix reduced to 125 variables. P 125= raw data from the portable equipment. B-P 125= data from the bench equipment converted to be equivalent to the portable equipment by the R script.

To transfer the calibration, three experimental approaches were carried out: matrix correction, variable selection and external validation. Matrix correction was carried out to match the size of the spectral data, given that the matrix of the benchtop equipment is more robust than that of the portable equipment. Variable selection was then applied to match the wavelengths of each NIR device, reducing variations in spectral resolution, and the selection was made manually among the 125 variables using a spreadsheet.

The objective of external validation was to evaluate the prediction performance of the models in relation to the spectra acquired from secondary sensors. In this approach, a partial least squares regression (PLS-R) model was developed using spectra measured on benchtop equipment, which was validated using spectra acquired on portable equipment and vice versa. In this way, it was possible to analyze the prediction error caused by the difference between the spectra of the two NIR sensors.

2.5 *Multivariate data analysis*

The spectral data were analyzed using multivariate statistics in their raw form and with the mathematical treatment of the first derivative of Savitzky-Golay with 13 smoothing points and a third-degree polynomial. Chemoface software version 1.65 [17] was used to perform principal component analysis (PCA) and partial least squares regression analysis (PLS-R). PCA was applied to explore the clustering of the data according to the raw and compatibilized matrices, while PLS-R was used to develop models for predicting basic wood density by leave-one-out and test set validation, as well as for calibration transfer.

For this, the number of latent variables was adjusted according to the needs of the model, and in the cross-validation the data selection method was randomized with six numbers of segments. In the test set, the spectra were separated into calibration and validation batches, using 60% of the data to calibrate the models and 40% to validate them, according to the data selection proposed by Medeiros et al. [11].

The ratio of performance to deviation (RPD) was obtained from the ratio between the standard deviation of the values determined in the laboratory and the standard error of the cross-validation. The criteria for selecting PLS regression models were assessed by reducing the root mean square error (RMSE) and maximizing the coefficient of determination (R^2).

3. Results and discussion

3.1 *Basic density and moisture of wood*

The basic density and moisture values of *Eucalyptus grandis* wood are shown in Fig. 3. Density ranged from 0.35 to 0.85 g/cm³, with the largest number of samples

concentrated between 0.50 and 0.55 g/cm³, corresponding to medium density wood (Fig.3A). The density value is directly influenced by the proportion of fibers and pores in the wood, as well as by the type of wood and external factors such as silvicultural treatments [18].

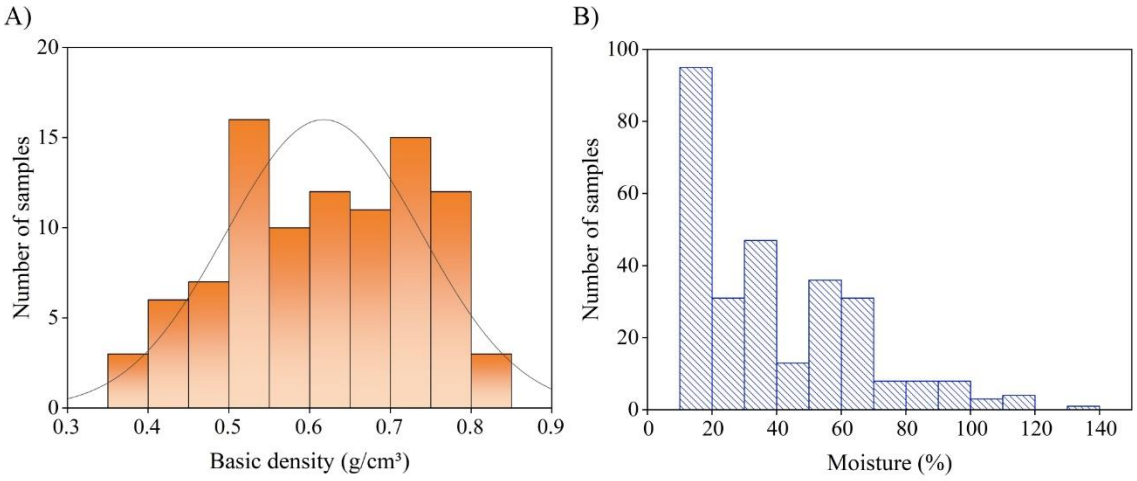

Fig. 3. Histogram of density (A) and moisture (B) of wood *Eucalyptus grandis*.

In the pulp and paper industry, these average values are recommended due to the wood delignification stage for transformation into cellulose pulp, where optimizing the cooking time increases the final pulping yield and reduces the amount of reagent used in the process. In addition, the viscosity parameters and the physical, mechanical and optical properties of the product are standardized [19]. This variability in density is important for the development of prediction models, as it broadens the sectors in which they can be applied and can be used in the paper and panel industries and in the energy sector to obtain charcoal.

Regarding wood moisture, a maximum value of 140% (green condition) and a minimum of ~12% (EMC) were observed, with FSP around 30% (Fig. 3B). The largest number of samples were found in the equilibrium condition with the environment. This trend refers to the water desorption of all samples, both reaching the same moisture range. In the green condition, however, the moisture values fluctuated due to the difference in the pith-bark and base-top directions. Gezici-Koç et al. [20] reported that, regardless of the position, the loss of impregnation water occurs only after the desorption of free water, and drying below the FSP occurs more quickly.

The range of moisture values was considered satisfactory for generating realistic information in the statistical models, which were superior or equivalent to studies in the

same segment. Amaral et al. [2] developed moisture prediction models for *Eucalyptus* chips with a water variation range between 80 and 0%. Santos et al. [21] included moisture from 130 to 0% to estimate the water content in *Eucalyptus urophylla* x *Eucalyptus grandis* hybrid wood, which achieved robust models, with R^2p reaching 0.96. Medeiros et al. [11] used moisture between 200 and 0% to predict cellulose pulp moisture using benchtop NIR, obtaining high prediction values (R^2p= 0.98).

However, Kollmann and Côté [22] warn that wood with very low density and high moisture conditions can cause multi-surface reflectance and water vapor in the spectral signal, respectively. As a result, model parameters can be compromised due to these factors.

3.2 *NIR spectral signature*

Analyzing the spectral signatures, differences were observed between the NIR spectrometers throughout the band, even after correcting the matrices obtained from the benchtop and portable equipment (Fig. 4A). This characteristic may be related to sensor technology, such as the type and power of the light, the intensity of the radiation and the spectral acquisition system [12]. The portable equipment showed absorbance values of between 0.1 and 0.3, which are incipient when compared to the benchtop equipment, with an absorbance range of between 0.3 and 0.7. This result is explained by the fact that benchtop equipment has a device that allows greater penetration of electromagnetic radiation, providing more information and consequently a greater range of absorbance.

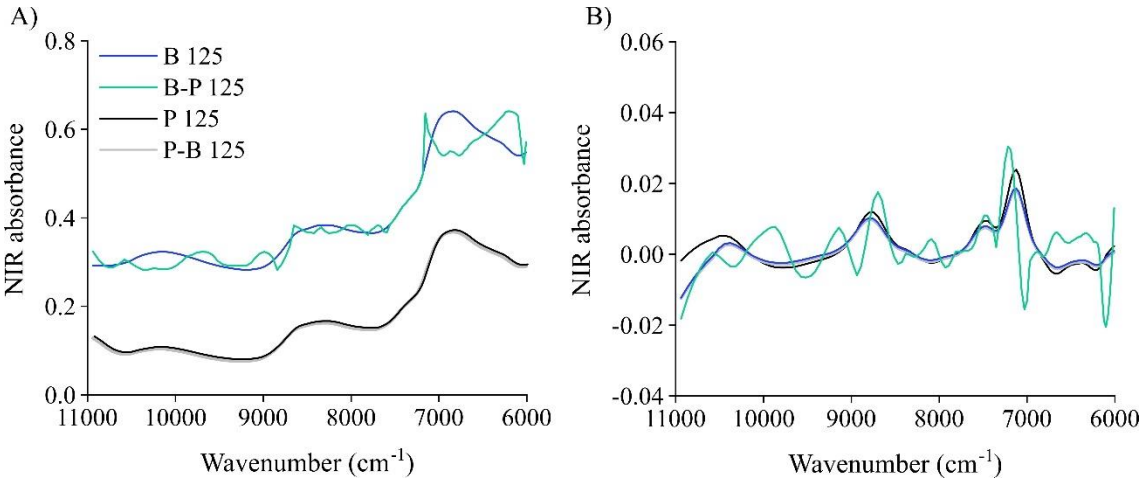

Fig. 4. Average spectral signature of the NIR matrices with the raw data (A) and treated by the first derivative (B) under different moisture conditions (green, fiber saturation

point, and equilibrium moisture). Where B 125= raw data from the bench equipment with 125 selected variables. B-P 125= data from the bench equipment converted to be equivalent to the portable equipment by the R script. P 125= raw data from the portable equipment. P-B 125= portable equipment data expanded to be equivalent to benchtop equipment by R script, with 125 variables subsequently selected.

In the spectral profile of the benchtop NIR matrix corrected to become equivalent to the portable equipment, variations were also seen along the wavelength, mainly between the 7300 cm^{-1} and 6000 cm^{-1} bands (Fig. 4B). In this case, the variations can be explained by the significant reduction in the number of variables, causing irregular peaks according to wavenumber. However, the profile of the spectra of the matrices obtained by the portable equipment was well standardized, even though the portable NIR matrix was increased in terms of the number of variables to make it compatible with the benchtop equipment, which is positive, given that the accuracy of the estimates of the variable of interest was maintained.

When applying the first derivative to the data, the signatures showed similar trends throughout the spectral range, with the exception of some peaks present in the corrected benchtop sensor matrix, which can also be explained by the superior electromagnetic radiation characteristics of the benchtop equipment, which is a parameter that is difficult to standardize.

In the raw and mathematically treated signatures (Fig. 4A and 4B), the main absorbance peak was in the 7000 cm^{-1} range, which is attributed to the constituents of the wood, such as the amount of cellulose, hemicelluloses and lignin [23]. Specifically, bands between 7000 cm^{-1} and 6287 cm^{-1} refer to the crystalline and amorphous regions of cellulose, peaks from 6900 to 6850 cm^{-1} to extractives and the phenolic OH groups of lignin, wavelengths between 5200 and 5050 cm^{-1} to water in the wood and in the 8370 cm^{-1} range the CH and CH_3 groups [24]. Despite the variations observed between the instruments and mathematical procedures for refining the information, the spectral signatures obtained for *Eucalyptus grandis* wood under different moisture conditions were in agreement with other studies in the literature [1,2,12].

3.3 *Principal component analysis (PCA)*

In the exploratory analysis of the spectra by type of NIR matrix, the effect of wood moisture had little influence on the discrimination of the data, in which the high presence of water reduced only 1% of the explanation of the principal components (PC) in the green condition with the raw data (Table 2). However, when evaluating the contributions of each PC in FSP and EMC moisture, it can be seen that the variations were negligible, with the individual and total values being similar, especially for the models without mathematical treatment.

Table 2

Contribution (%) of the principal components (PC) of the raw spectra and mathematical treatments by NIR sensor for *Eucalyptus grandis* wood in different moisture conditions: green, at fiber saturation point (FSP) and at equilibrium moisture (EMC).

Moisture	Treatment	PC1	PC2	Total
Green	Untreated	95	2	97
	1D	66	15	81
FSP	Untreated	97	1	98
	1D	86	6	92
EMC	Untreated	97	1	98
	1D	82	8	90

The PCA model for discriminating NIR matrices with the highest explanatory percentage was achieved with the raw spectra (PC1= 97% and PC2= 1%). Thus, the first derivative treatment did not show any improvement for this specific analysis, even though this treatment showed the best effect among the other spectral processing treatments, with standard normal variation and second derivative. However, even though first derivative models show a reduction in data explanation, they may be more realistic for practical use in industries. Arriel et al. [4] developed PCA models to discriminate the source of spectral acquisition (integrating sphere and optical fiber) and achieved satisfactory results with the raw data, with PC1 = 99.38% and PC2 = 0.48%.

In the PCA scores, for the data of the B1300 and P125 matrices treated by first derivative, some samples were projected in isolation (Fig. 5A and 5C), which can be explained by the greater amount of water in some samples, due to the lower density. However, as water desorption occurs in the wood, the samples are projected more

homogeneously and with low dispersion, since the water content tends to be more homogeneous among the samples as they approach the equilibrium moisture content. In the PC loadings, for the benchtop and portable NIR, peak intensity was detected around the bands 5128 cm^{-1}, 6896 cm^{-1}, 8333 cm^{-1} and 10300 cm^{-1} (Fig. 5B and 5D), which correspond to the absorption range of liquid water in the NIR [25,26].

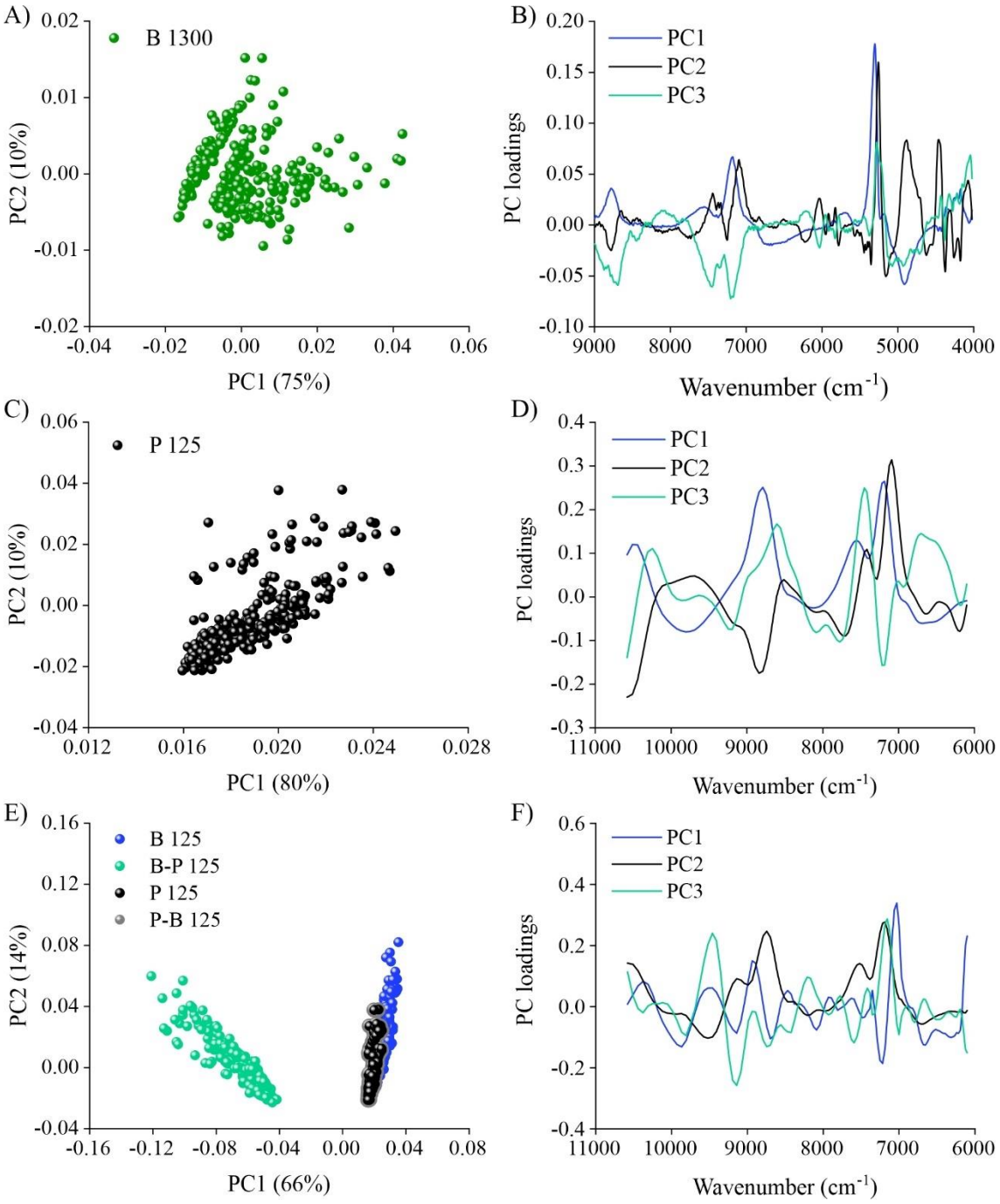

Fig. 5. PCA scores (A, C, E) and PC loadings (B, D, F) of NIR matrices treated with first derivative. Where B 125= raw data from the bench equipment with 125 selected variables. B-P 125= data from the bench equipment converted to be equivalent to the portable

equipment by the R script. P 125= raw data from the portable equipment. P-B 125= portable equipment data expanded to be equivalent to benchtop equipment by R script, with 125 variables subsequently selected.

In the global PCA, which includes the four matrices under different moisture conditions, the raw matrices of the benchtop and portable equipment overlapped the portable NIR matrix compatible in benchtop NIR (Fig. 5E), with the data treated by the first derivative. However, the benchtop NIR matrix converted in portable NIR data did not show the same trend, which may be an effect of the variations observed in spectral signatures (Fig. 4). In the study by Rukundo et al. [7], overlapping PCs were found to be a reliable indicator of spectral data standardization, indicating that calibration could be transferable to another NIR spectrometer.

In the sum of PC1 + PC2, the total contribution of the global PCA was 80%, which is considered low when compared to the models developed at just one moisture level, as shown in Table 2. However, global models are indispensable, as they include different situations that may be necessary in the use and useful life of the calibration. In the PC loadings graph, the reflectance values showed positive and negative values, ranging from -0.3 to 0.3, with sharp peaks in the 9200 cm^{-1} and 7300 cm^{-1} wavebands, according to the spectral signatures. This tendency for the new variables to be similar to the samples has also been reported in the literature [11].

3.4 *Partial least squares regression (PLS-R)*

When analyzing the data from each matrix separately and at different moistures, it was possible to obtain robust models with cross-validation (R^2cv) and independent (R^2p) coefficients of determination greater than 0.777 and 0.744, respectively (Table 3). Thus, the effect of the water condition in the wood had little influence on the performance of the models, although it is common for free water to alter the spectral reading due to the greater solubilization of extractives and minerals present in the lumen.

Table 3

Statistical parameters of PLS-R models for estimating the basic density of wood at different moisture levels using NIR sensors.

Matrix	MC	Treatment	R²cv	RMSEcv	R²p	RMSEp	RPDcv	LV
B 125	Green	untreated	0.785	0.056				
		1D	0.796	0.055	0.780	0.058	2.182	10
	FSP	untreated	0.915	0.036	0.865	0.037	3.370	8
		1D	0.902	0.039				
	EMC	untreated	0.808	0.054				
		1D	0.800	0.055	0.826	0.052	2.182	5
	All	untreated	0.806	0.053	0.744	0.060	2.286	10
		1D	0.803	0.053				
B-P 125	Green	untreated	0.818	0.053	0.837	0.050	2.268	7
		1D	0.802	0.054				
	FSP	untreated	0.929	0.033	0.901	0.031	3.712	8
		1D	0.895	0.040				
	EMC	untreated	0.777	0.059				
		1D	0.800	0.054	0.830	0.051	2.222	6
	All	untreated	0.804	0.053	0.744	0.060	2.285	10
		1D	0.796	0.054				
P 125	Green	untreated	0.849	0.047				
		1D	0.873	0.043	0.864	0.046	2.793	6
	FSP	untreated	0.868	0.044	0.897	0.044	2.723	8
		1D	0.867	0.045				
	EMC	untreated	0.795	0.055				
		1D	0.802	0.054	0.783	0.057	2.226	4
	All	untreated	0.785	0.055				
		1D	0.805	0.053	0.820	0.050	2.286	7
P-B 125	Green	untreated	0.861	0.047				
		1D	0.885	0.042	0.861	0.046	2.886	7
	FSP	untreated	0.884	0.042				
		1D	0.855	0.046	0.870	0.046	2.606	3
	EMC	untreated	0.787	0.055				
		1D	0.812	0.054	0.782	0.057	2.247	4
	All	untreated	0.785	0.056				
		1D	0.805	0.053	0.803	0.053	2.286	7

R²cv - coefficient of determination for cross-validation. RMSEcv - root mean square error for cross-validation. R²p - coefficient of determination for prediction. RMSEp - root mean square error for prediction. RPDcv - ratio of performance to deviation for cross-validation. LV - latent variables. 1D - first derivative. B 125= raw data from the bench equipment with 125 selected variables. B-P 125= data from the bench equipment converted to be equivalent to the portable equipment by the R script. P 125= raw data from the portable equipment. P-B 125= portable equipment data expanded to be equivalent to benchtop equipment by R script, with 125 variables subsequently selected. MC= moisture content.

The models developed with the matrices from the portable equipment (P 125 and P-B 125) showed the best statistical parameters in the cross-validation. For independent validation, the highest R^2 values and the lowest error values associated with the models were obtained for the matrix from the benchtop equipment corrected for the portable one (B-P 125). This shows that the four matrices have the potential to be used to adjust the basic density predictive models, as well as the feasibility of applying the conversion script developed to generate the new compatible matrices.

Similar to the PCA, wood under FSP conditions showed the best results in the PLS-R, although the models including all moistures were also satisfactory in terms of statistical parameters, especially with the B125 matrix. Substantial variations were observed in the RMSE of the models (0.033 to 0.060 g/cm³), indicating a difference between the data matrices. In terms of the ratio of performance to deviation (RPD), the models had a minimum value of 2.182, maximum of 3.712 and average of 2.535, meeting the requirements of the literature [27,28]. As for the treatment of the data, the first derivative showed improvements for some parameters, but with minimal difference from the raw data.

Amaral et al. [29] also developed models for predicting the basic density of *Eucalyptus* wood based on NIR spectroscopy with benchtop equipment and equilibrium moisture (~12%). These authors evaluated different mathematical treatments, and the first derivative showed the best statistical parameters for the models, with R^2cv= 0.86, RMSEcv= 0.254 g/cm³ and RPD= 2.69. Medeiros et al. [12] generated models for predicting basic density at different moistures using benchtop and portable NIR and obtained minimal differences between the results from each sensor, indicating that the choice of equipment can be determined by the application environment, laboratory or field.

In the models including the spectra of the four NIR matrices, the wood in the FSP condition showed R^2p and RMSEp values of around 0.844 and 0.045 g/cm³, respectively (Fig. 6B). In other words, these parameters indicate that there was a strong correlation between the values predicted by the NIR and those determined in the laboratory.

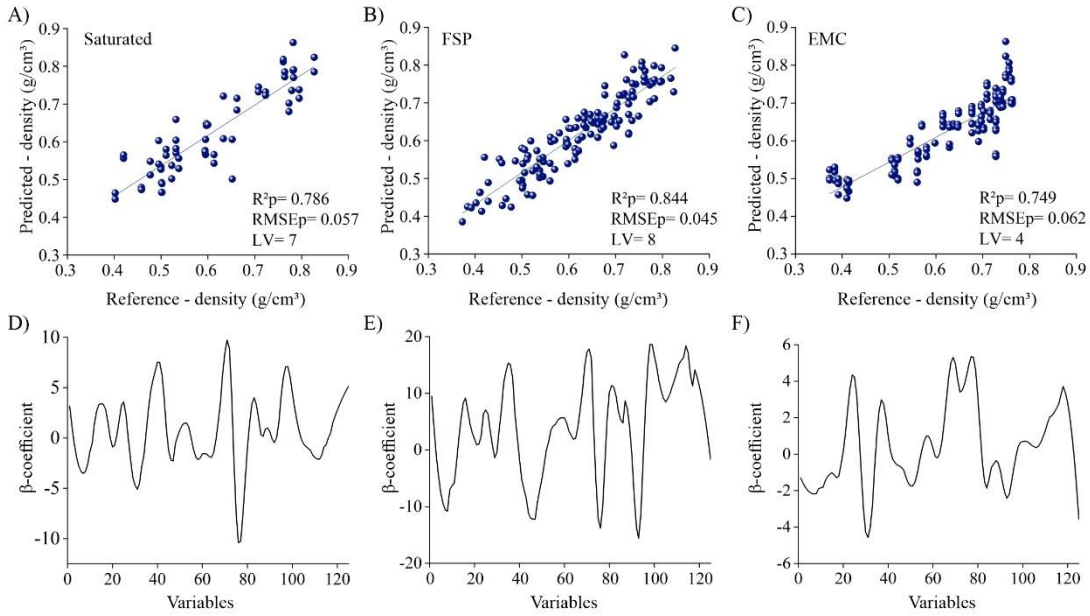

Fig. 6. Model validation plots include the four NIR matrices in different moisture conditions (A, B, C) and their respective β-coefficients (D, E, F).

The models obtained for wood in the EMC condition and above FSP had the lowest R^2p values, respectively. However, the RMSEp values for the model obtained in EMC were ~8% higher than those obtained above FSP. These results are probably related to the behavior of electromagnetic radiation due to the high-water content above the FSP, which can cause oscillations in the spectral signal. In the EMC condition, the absorbance shows a tendency to decrease, as the lower water content minimizes the vibrations of the hydroxyl groups.

In the global model, with the different matrices and moisture conditions, the prediction coefficient was adequate, with R^2p= 0.765 (Fig. 7). This approach can save time and costs on the development and implementation of new static models in NIR instruments, whether benchtop or portable. However, new model optimization methods are essential to reduce RMSEp, which can improve the stability of spectral signals between devices.

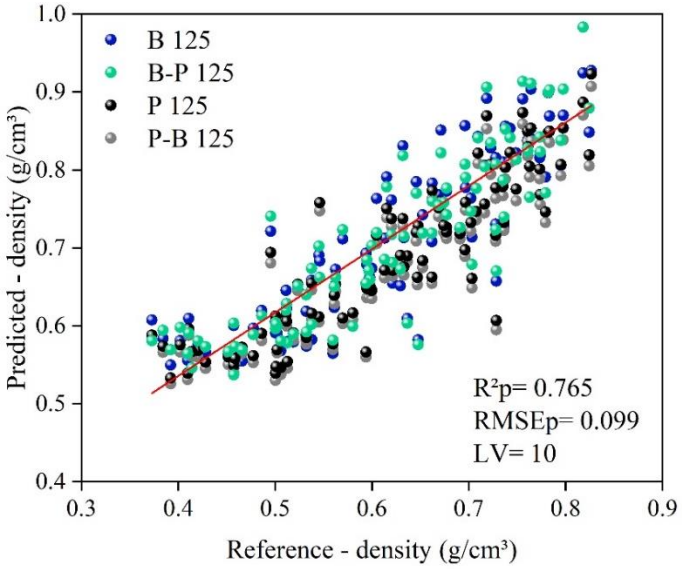

Fig. 7. Global model based on first derivative of NIR spectra for predicting wood basic density. Where B 125= raw data from the bench equipment with 125 selected variables. B-P 125= data from the bench equipment converted to be equivalent to the portable equipment by the R script. P 125= raw data from the portable equipment. P-B 125= portable equipment data expanded to be equivalent to benchtop equipment by R script, with 125 variables subsequently selected.

Global models are considered complex and more statistically accurate due to the larger size of the database and the inclusion of different sources of variation, which is why it is important to develop them in large-scale analyses for industrial application [8]. Medeiros et al. [12] when developing global models for estimating the basic density of wood at different moistures and NIR sensors, obtained values of R^2= 0.83 and RMSEp= 0.04 g/cm³ for the benchtop equipment and R^2= 0.79 and RMSEp= 0.05 g/cm³ for the portable equipment, considering green moisture up to EMC, corroborating this study.

3.5 *Calibration transfer between NIR sensors*

The calibrations developed on the benchtop equipment, by moisture level, showed considerable prediction parameters after testing independent batches of data obtained from the secondary sensor (Table 4). When validating the models in each spectral matrix, the highest coefficients were in the EMC moisture with the data without mathematical treatment, although the lowest errors were seen in the FSP moisture.

Table 4

Statistical parameters of models calibrated with benchtop NIR data and validated in raw data matrices and compatible by the R script.

MC	Treatment	Cross-validation		Test set validation					
		B 125		P 125		P-B 125		B-P 125	
		R^2cv	RMSEcv	R^2p	RMSEp	R^2p	RMSEp	R^2p	RMSEp
Green	untreated.	0.785	0.056	0.590	0.238	0.592	0.235	0.068	0.228
	1D	0.796	0.055						
FSP	untreated	0.915	0.036			0.656	0.089		
	1D	0.902	0.039	0.209	0.276			0.115	0.867
EMC	untreated	0.808	0.054	0.732	0.505	0.734	0.489	0.675	0.240
	1D	0.800	0.055						

R^2c - coefficient of determination for calibration. RMSEc - root mean square error for calibration. R^2p - coefficient of determination for prediction. RMSEp - root mean square error for prediction. 1D - first derivative. B 125= raw data from the bench equipment with 125 selected variables. P 125= raw data from the portable equipment. P-B 125= portable equipment data expanded to be equivalent to benchtop equipment by R script, with 125 variables subsequently selected. B-P 125= data from the bench equipment converted to be equivalent to the portable equipment by the R script. MC=moisture content.

Evaluating the performance of each matrix, it can be seen that the data from the portable equipment converted into a benchtop (P-B 125) showed the highest coefficients of determination for predicting basic density (0.592 to 0.734), as well as the lowest error values associated with the models (0.089 to 0.489 g/cm³), indicating that the data standardization method is promising. Close values were achieved in the portable equipment matrix (P 125), while the least desirable parameters were observed in the standardized bench matrix for portable (B-P 125). As for the mathematical treatment of the first derivative, this showed improvements only in the model validated with the P 125 and B-P 125 matrix in the FSP moisture condition.

Similarly, Table 5 presents the validation values of the portable equipment at different moisture in the matrices with the raw data and compatible by the R script. In this transfer, the results showed higher values in the coefficient of determination when compared to the validation of the benchtop equipment, especially when validating in the P-B 125 matrix, which showed a minimum value of 0.877. However, the RMSE values were higher in the B-P 125 matrix, ranging from 0.600 to 2.835 g/cm³. This behavior can be attributed to the wave range and resolution of each sensor, since the portable sensor requires more complex standardization to make it compatible with the benchtop sensor database, while the benchtop equipment requires a large reduction in the number of

variables to be transformed into a portable one, generating opposite performance between the sensors.

Table 5

Statistical parameters of models calibrated with portable NIR data and validated in raw data matrices and compatible by the R script.

MC	Treatment	Cross-validation		Test set validation					
		P 125		B 125		P-B 125		B-P 125	
		R^2cv	RMSEcv	R^2p	RMSEp	R^2p	RMSEp	R^2p	RMSEp
Green	untreated	0.849	0.047			0.902	0.038	0.049	0.644
	1D	0.873	0.043	0.448	0.338				
FSP	untreated	0.868	0.044			0.920	0.034		
	1D	0.867	0.045	0.735	0.331			0.049	2.835
EMC	untreated	0.795	0.055	0.485	0.521			0.175	0.600
	1D	0.802	0.054			0.877	0.043		

R^2c - coefficient of determination for calibration. RMSEc - root mean square error for calibration. R^2p - coefficient of determination for prediction. RMSEp - root mean square error for prediction. 1D - first derivative. P 125= raw data from the portable equipment. B 125= raw data from the bench equipment with 125 selected variables. P-B 125= portable equipment data expanded to be equivalent to benchtop equipment by R script, with 125 variables subsequently selected. B-P 125= data from the bench equipment converted to be equivalent to the portable equipment by the R script. MC=moisture content.

For these models, the treatment of the first derivative provided improvements in the matrix parameters, and in the bench matrix (B 125), the coefficient of determination reached 0.448 and the error 0.338 g/cm³ in the green condition. On the other hand, in the FSP condition, the coefficient reached 0.735 and the RMSEp 0.331 g/cm³ with the application of the derivative. Honorato et al. [8] and Siesler et al. [30] report that the first derivative is the main mathematical treatment in NIR spectra, as it eliminates the systematic displacement of the baseline.

Studies on calibration transfer between NIR equipment in wood science and technology are still scarce. However, Zhang et al. [6] applied direct standardization, slope/bias correction, direct piecewise standardization and canonical correlation analysis methods to evaluate the performance of calibration transfer between four portable NIR spectrometers, with the aim of predicting lignin content in wood, and concluded that direct standardization is the most suitable for maintaining model robustness, which confirms the potential of the transformation matrices in this study, which present a methodology similar to direct standardization.

Li et al. [31] calibrated a PLS model built with spectra measured on primary equipment and validated it with spectra from a secondary instrument to predict the nitrogen content in *Nicotiana tabacum* leaves. The authors concluded that the method can be efficient for practical applications when primary equipment is not available, corroborating the results found in this study, which also applied the same calibration transfer method. Rukundo et al. [7] also developed a model for estimating nitrogen content with a benchtop NIR spectrometer, but for grasses, and reported that it could be successfully transferred to two spectrometers, one benchtop and one portable.

The best models after the calibration transfer were obtained in the FSP moisture condition and with the data without mathematical treatment, which were selected based on the lowest error associated with the models, as shown in Fig. 8. One hypothesis for the observed behavior, in which the estimates perform better under certain moisture conditions, such as at the fiber saturation point or at equilibrium moisture, may be related to wood sampling, such as the region of the tree from which the wood was collected. In addition, it may be associated with the thickness of the cell wall, since adult woods with thicker cell walls tend to provide high spectral information due to the greater amount of chemical groups capable of interacting with electromagnetic radiation. In contrast, juvenile woods with thinner cell walls have less potential for spectral interaction.

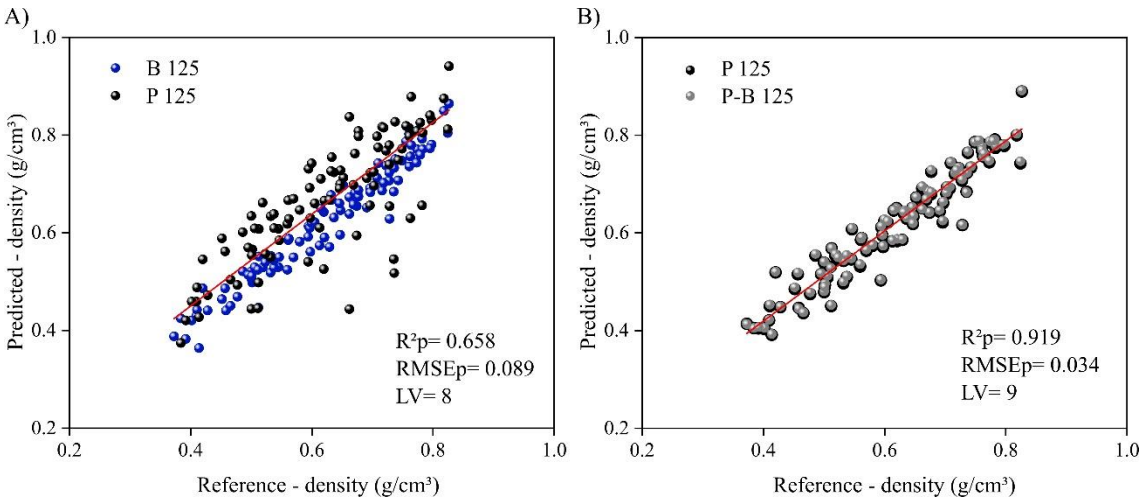

Fig. 8. Plot of the best models after calibration transfer for predicting the basic density of wood in the condition of moisture at the fiber saturation point and with the raw data.

The model with the highest performance when calibrating with the benchtop sensor data was seen when validating with the P 125 matrix, with the raw data from the

equipment, without the need for matrix standardization methods, where the dispersion shows the differences between the equipment, but also the closeness of the prediction trends (Fig. 8A). In the calibration developed on the portable equipment, the most robust model was when it was validated with the P-B 125 data, the matrices being of primary origin, which is why the information in the PLS regression overlapped (Fig. 8B).

Thus, the benchtop sensor showed the highest transfer capacity, presenting promising results even with data from different sources, sizes and spectral resolutions. This superior performance can be attributed to the higher signal-to-noise ratio (S/N) of this device, a factor that directly influences the effectiveness of transferring calibration models. Benchtop equipment with a higher S/N generates cleaner, better quality spectra. On the other hand, portable sensors have lower S/N, which results in noisier spectra and, consequently, less analytical robustness.

Finally, global models for calibration transfer, including all the possible sources of variation evaluated in this study, were tested for their performance (Table 6). Analyzing the values by moisture range, the best parameters remain in the FSP condition, especially for the data without mathematical treatment. On the other hand, the models covering green moisture, FSP and EMC showed greater robustness with the spectra treated by first derivative.

Table 6

Statistical parameters of the calibration transfer by the update method between the original and corrected NIR matrices.

MC	Treatment	Cross-validation		Test set validation							
		Global		B 125		P 125		P-B 125		B-P 125	
		R^2cv	RMSEcv	R^2p	RMSEp	R^2p	RMSEp	R^2p	RMSEp	R^2p	RMSEp
Green	untreated	0.816	0.051	0.824	0.040						
	1D	0.814	0.051			0.838	0.057	0.905	0.042	0.888	0.039
FSP	untreated	0.856	0.045	0.933	0.037	0.839	0.040	0.906	0.041		
	1D	0.845	0.047							0.780	0.061
EMC	untreated	0.807	0.053								
	1D	0.815	0.052	0.825	0.062	0.850	0.050	0.755	0.051	0.787	0.061
All	untreated	0.718	0.064	0.696	0.065						
	1D	0.741	0.061			0.735	0.062	0.805	0.053	0.714	0.063

R^2c - coefficient of determination for calibration. RMSEc - root mean square error for calibration. R^2p - coefficient of determination for prediction. RMSEp - root mean square error for prediction. 1D - first derivative. B 125= raw data from the bench equipment with 125 selected variables. P 125= raw data from the portable equipment. P-B 125= portable equipment data expanded to be equivalent to benchtop

equipment by R script, with 125 variables subsequently selected. B-P 125= data from the bench equipment converted to be equivalent to the portable equipment by the R script. MC=moisture content.

When evaluating the validation matrix, it can be seen that the B 125 data showed the lowest error associated with the models, as well as the highest coefficient of determination. However, in general, all the combinations proved to be viable for predicting the basic density of wood. Medeiros et al. [11] applied this same calibration transfer method, but for moisture prediction regardless of the type of cellulose pulp, considering only benchtop equipment. Thus, the calibration transfer had an R^2p greater than 0.839, showing satisfactory performance for estimating different moistures in any type of pulp.

According to Ni et al. [32], calibration transfer promotes the compatibility of calibration spectral data applied in validation, which allows the model to be applied in different practical situations, in the case of this study, for predicting basic density between NIR equipment. Thus, as a result, the potential for using the developed models as an alternative to overcome the deficiencies of large production processes stands out, even if they do not present the same performance as the primary equipment. In addition, the methodology presents the advantage of using databases already obtained and stored by industries, avoiding redoing the complete acquisition of new spectra, which optimizes time and reduces costs.

The models can help overcome the main challenges faced in corporate environments, especially those that make it difficult to obtain wood density quickly. In addition, they help to develop new approaches applicable to other relevant estimates, such as lignin and extractive content. They also serve as a basis for qualitative analyses, such as phenotyping genetic materials in breeding programs, species identification, traceability and monitoring of forest products, as well as non-timber applications, including oils, tannins, bark, among others.

It is recommended that further studies be carried out with adult and juvenile wood in different moisture conditions, including spectral collection using both NIR sensors, in order to compare the performance of the models in the face of sampling variations. This approach will make it possible to analyze the effect of porosity in terms of the amount of capillary water, which can interfere with the spectral signal and introduce multiple variation factors into the model. In addition, it is recommended that new species be included to assess the impact of genetic differentiation on the spectral properties of wood.

Finally, the prospects are that these models for predicting the basic density of wood will be applied under natural drying conditions, such as in piles of logs or industrial chips, as they represent practical scenarios for using and processing wood. In this way, the performance of the models can be assessed in a more realistic way and in line with the operational demands of the forestry sector.

4. Conclusions

The basic density of wood using benchtop and portable NIR spectrometers could be estimated at different moisture levels, both with the matrices obtained directly from the sensors and with the matrices standardized by the R script developed in this study.

Wood moisture caused variations in the predictive capacity of the PLS-R models, with the fiber saturation point being the most favorable condition for developing the models in the individual matrices, while in the calibration transfer the equilibrium moisture with the environment was the most suitable for maintaining the robustness of the models.

In the calibration transfer, the model developed with the benchtop equipment showed the best performance to be transferable to the portable equipment due to the lower error values associated with the models, especially with the data without mathematical treatment. It is therefore recommended that the instrument responsible for developing the calibration should be the benchtop device and the instrument receiving the model should be the portable device.

Credit author statement

Dayane Medeiros: Conceptualization, Methodology, Validation, Writing– Original draft, Writing – Review & Editing. Felipe Batista: Formal analysis, Methodology, Writing: Review & Editing. Adriano Mascarenhas: Visualization, Investigation, Writing: Review & Editing. Gilles Chaix: Software, Data Curation, Supervision, Investigation. Paulo Hein: Funding acquisition, Project administration, Resources, Writing: Review & Editing.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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PART THREE - GENERAL CONSIDERATIONS

NIR spectroscopy combined with multivariate statistics is able to categorize and predict the basic density of wood regardless of moisture conditions. The anatomical plan of the wood influences the fit of the models built to estimate basic density.

Calibration transfer is more effective when the model is developed from the spectral data matrix acquired with benchtop NIR equipment and under PSF moisture conditions.

The models developed to determine density have the potential to be used in the forestry industry, helping to monitor and standardize the quality control of planted wood.

It is recommended that further studies be carried out with adult and juvenile wood in different moisture conditions, including spectral collection using both NIR sensors, in order to compare the performance of the models in the face of sampling variations. This approach will make it possible to analyze the effect of porosity in terms of the amount of capillary water, which can interfere with the spectral signal and introduce multiple variation factors into the model. In addition, it is recommended that new species be included to assess the impact of genetic differentiation on the spectral properties of wood.

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