Canadian Science Publishing | Canadian Journal of Forest Research

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Journal:	*Canadian Journal of Forest Research*
Manuscript ID	cjfr-2024-0270.R1
Manuscript Type:	Research Article
Date Submitted by the Author:	09-Dec-2024

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Is the manuscript for consideration in a Special Issue or Collection?:	Recent Advances in Non-destructive Testing of Wood
Keyword:	Near Infrared Spectroscopy, eucalyptus, microfibril angle, non-destructive measurement, reflectance

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Nondestructive measurement of microfibril angle of wood by using near-infrared spectroscopy

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ABSTRACT

Fast-growing tree species, such as Eucalyptus, are used extensively in plantations for timber, but their mechanical properties are not well understood, especially the microfibril angle (MFA), which affects wood stiffness. MFA measurement is complex and expensive, but near-infrared spectroscopy (NIRS) offers a non-destructive alternative. This study aims to evaluate the effectiveness of NIRS in predicting MFA across different regions and environments. The results showed that NIRS could predict MFA, but the accuracy varied. In Brazil, higher prediction accuracy was observed when data from multiple regions were combined. In Laos, the presence of juvenile wood significantly decreased prediction accuracy. Combining data from multiple sites improved prediction accuracy, but decreased accuracy when juvenile wood was included. The study concludes that effective MFA prediction models must consider regional and environmental differences. Creating region-specific models is necessary for reliable wood quality assessment using NIRS. This research underscores the potential of NIRS as a practical tool for wood quality evaluation, highlighting the importance of accounting for factors such as wood maturity and environmental conditions in developing robust predictive models.

Keywords: Near Infrared Spectroscopy; eucalyptus; microfibril angle; non-destructive measurement; reflectance; wood property

INTRODUCTION

Fast-growing tree species are cultivated in plantations. As timber use shifts from natural forests to plantations, there are high expectations for utilization of wood due to increased growth and biomass production. These fast-growing species, such as *Eucalyptus* spp., have primarily been bred to supply pulp and fuelwood, making them less valuable as commercial frame timber for building materials. Consequently, there is currently limited information available on the mechanical properties of fast-growing species. It is necessary to measure wood physical properties such as stiffness, modulus of rupture, microfibril angle (MFA) and so on as using frame timber. In particular, MFA, which significantly affects physical properties, has rarely been investigated due to the difficulty of the measurement procedure and its time-consuming nature. Even if there were easy ways to measure parameters of wood quality, such as the Silvi Scan, the device is expensive and the number of devices and the countries that own them are limited.

MFA is related stiffness and physical properties of wood, such as elongation and contraction. Therefore, understanding MFA variation in wood is important for utilization of the wood; however, it can only be measured with expensive equipments or with time- consuming and laborious ways (Isik et al. 2011). That is why the data has not been collected for a variety of wood species, especially fast-growing hard wood.

To achieve rapid measurement of MFA in numerous wood samples, we focused on utilizing Near-infrared spectroscopy (NIRS). NIRS is a non-destructive measurement technique widely used in fields such as food science, agriculture, pharmaceutics, and forestry (Tsuchikawa et al. 2022, Pasquini et al. 2018, Bec et al.

2021). In the field of wood science, NIRS has been used to estimate cellulose and lignin content, as well as wood density and Young's modulus (Kelley et al. 2004, Ma et al. 2018). Hence, we hypothesized that simplifying and expediting the evaluation of wood quality using NIRS, we could accumulate a substantial amount of the data, and analyze the differences among wood species, growth regions, and patterns of wood quality distribution.

Several studies have attempted to predict MFA using NIRS (Schimleck et al., 2001, 2002, 2003, 2005; Jones et al., 2005). Previous NIR spectroscopic studies utilized MFA data provided by SilviScan analysis for calibration purposes. The MFA was determined by SilviScan using scanning X-ray diffraction with the X-ray beam approximately parallel to the growth ring. Schimleck (2007), determined MFA for each sample by measuring the relative width of 002 diffracted X-ray arcs from an X-ray beam on a tangential cross-section and utilizing a predictive model for the NIR. In this study, the MFA was determined using the modified Cave's method with X-ray diffractometry, where the X-ray beam was irradiated on the tangential section.

Previous research has demonstrated a strong correlation between the values of MFA measured by SilviScan and estimated by NIR in *Eucalyptus delegatensis* and *Pinus radiata* (Schimleck et al., 2001; Schimleck et al., 2002). While it is clear that MFA can be predicted using NIRS, adaptability of a model created for a specific site or tree species has not been investigated.

In a calibration model using NIRS, Wang et al. (1991) stated that calibrations created for certain products may not be useful to predict other products. In the field of fruit research, modelling errors can easily double when calibration models are applied to spectral datasets from different seasons or orchards (Nicolãı et al., 2007). These problems are less pronounced for products whose quality is stable, such as manufactured goods, but can be significant for plants and living organisms.

Similar to fruit, it is generally recognized that properties of wood vary based on factors such as the planting site and soil (Zobel et al. 1998, Kojima et al. 2009a). Therefore, a predictive model for the estimation of the properties of wood derived from a certain plantation may not be applicable as a general predictive model for other plantations. In other words, it is important to evaluate whether models that use near-infrared to predict the properties of wood grown in a particular region can be adapted to predict wood properties in different regions.

In a previous study, estimation of wood density, MFA, and stiffness of *Pinus taeda* (loblolly pine) using NIRS have been successfully applied to cores taken from various sites, with samples representative of a variety of sites and geomorphological regions in Georgia (USA) (Jones et at. 2005). These results suggest that general models for the estimation of wood properties of different species or site can be developed in the field of wood science. The above results suggest that wood qualities can be predicted using NIRS in the same way, regardless of sites where the samples are collected. However, it is necessary to examine whether the same predictions can be made in environments with significantly different growth habitats.

Based on the aforementioned context, our research focused on examining whether consistent predictions of MFA could be yield regardless of the planted location. In this study, we tested whether different prediction models can be adapted between regions with a different climatic division within an extensive area or regions with extremely variable planting environments within a narrow area. We conducted the following experiments using the MFA, which is related to the physical properties of wood such as stiffness and shrinkage.

1(Brazil): Eucalyptus trees planted in Brazil were used to examine the adaptability of the predictive model in regions with extensive climatic divisions. Brazil spans a vast north-south range, and the same tree species are planted across various latitudes and climatic zones, allowing us to investigate differences in planting locations over a wide range.

2(Laos): The adaptability of the prediction model was also examined using eucalyptus trees planted in Laos, where the planting environment changes significantly within small areas. In Laos, despite being in the tropics, there are many areas with poor river management, leading to some regions being flooded during the rainy season while others remain dry. This variability in the soil moisture environment enabled us to study planting areas with extremely different conditions.

3(Unified Model): Finally, we explored the possibility of developing a predictive model that could cover all the planting environment described in points (1) and (2) above.

MATERIALS AND METHODS

Sampling area and sample preparation

Wood sampling was conducted in Brazil and Laos to investigate the effects of different latitudes and climates in Brazil and the influence of habitat environments in Laos on NIR results. Detailed locations of the sampled sites are shown in Table 1.

At each site the test trees were selected to represent a wide range of diameters at breast height (DBH). For sample collection with a wide range of diameters, we utilized the existing diameter data of the plantation trees. We selected the trees with the largest and smallest diameters, and then chose the remaining samples to ensure an even distribution of diameters. This method ensured a balanced representation of trees with varying diameters. A rectangular specimen was collected at three-four cardinal points at breast height on each tree with a hand saw and chisel. This three-four cardinal sampling points method was used in order to account for the presence of reaction wood and variations in wood properties depending on the sampling points.

We cut off the sample as L:10mm R: 1mm T: 10mm to measure the microfibril angle (MFA) in the S_2 layer of the secondary wall.

Samples from the different latitudes in Brazil

We studied *Eucalyptus grandis* W. Hill ex Maiden planted in three plantations in different latitudinal divisions, including tropical and subtropical climates in Brazil.

Sixty-nine wood samples were tested from an 18-year-old plantation in Açailândia in Brazil (B1). Eighty-five wood samples were collected from a 17-year-old plantation in Posto da Mata, Brazil (B2). Ninety-one wood samples were tested in a 16-year-old plantation in Porto Alegre, Brazil (B3). Detailed plantation information can be found in a previous paper (Kojima et al. 2009a).

Samples from the different habitat environment in Laos

The locations of our study areas are in central Laos. We studied *E. camaldulensis* × *E. deglupta* clones from three plantations; a non-flooded plantation, a slightly flooded plantation, and a flooded plantation, all planted in 2007. The slightly flooded plantation is not flooded above ankle height, but the flooded plantation is flooded to chest height in the rainy season. The three study plantations are located at almost the same latitude and climate zone; only their water condition differs.

Sixty-four wood samples were tested from a 3-year-old plantation in a non-flooded plantation (L1-NF), Paksuun, and in a flooded plantation (L2-F), Nong boua

PK in Laos. Fifty-two wood samples were tested from a 3-year-old plantation in a slightly flooded plantation (L3-SF), Nong boua PK in Laos. The plantation being in its third year of growth suggests that there is a high probability of it containing a significant proportion of juvenile wood.

Measurement of the microfibril angle in the middle layer of the secondary wall

Microfibril angle (MFA) in the S_2 layer of the secondary wall was determined for all test specimens. MFA in a thin tangential section (1mm thick) was determined with the modified Cave's method using X-ray diffractometry (CuKα line) (XD-D1w; Shimadzu Company Kyoto, Japan)(Cave 1966, Meylan 1967). The distribution of the diffraction intensity along the cellulose (200) arc was recorded by means of X-ray diffractometry and the "angle *T*" was determined according to Cave (1966), which is the basis for MFA determination by equation "MFA = 0.6×angle*T*". In the present study, we used raw MFA data obtained from our previous paper (Kojima et al. 2009a), collected in Brazil.

NIR device and spectra acquisition

For NIR measurement, we used the same samples used for the MFA measurements using X-ray diffractometry. The diffuse reflectance spectra of a spot (3 mm diameter) were collected at 8 cm^{-1} intervals over a range of 12000-4000 cm^{-1} (830-2500 nm) and the resolution was 8 cm^{-1} using a MATRIX-F spectrometer (Bruker Optics) equipped with an NIR fiber optic probe. The X-ray beam was irradiated on the tangential section, similarly to the NIR measurement. 32 scans were performed for each sample; these scans were averaged to give a single spectrum per sample. All the spectra were acquired at 20 ± 2°C and 65 ± 5% relative humidity.

Multivariable Analysis of the NIR Data Set

Statistical analysis of MFA for the training set and test set is presented in Table 2. The training set encompassed both high and low values for each parameter. Raw NIR spectra often exhibited baseline shifts or drifts due to variations in NIR measurements. Additional factors, such as instrument stability, temperature, humidity, or surface conditions of the sample, could also influence the spectra (Candolfi et al., 1999). Therefore, data pretreatment played a vital role in the analysis to eliminate such variations. For NIR measurement, pretreatment techniques were employed to mitigate these variations. In this study moving average smoothing (21points), baseline correction and second derivative calculation (Norris gap) were applied as the spectra pretreatment. Pretreated NIR spectra were utilized for subsequent analysis.

Partial least squares regression (PLSR) was employed for regression analysis. PLSR aimed to minimize the discrepancy between the target variable (MFA value) and the explanatory variable (NIR spectral data). The optimal number of factors used for each calibration was determined based on recommendations from the Unscrambler X software (version 10.1; Camo Software, Oslo, Norway). The data were assessed by examining the determinant coefficient (R^2 for the training set and R_{test}^2 for the test set) between the predicted and measured values. Additionally, the root mean square error of cross-validation (RMSECV) and the root mean square error of prediction for the test set (RMSEP) were statistically evaluated and compared for calibration and prediction models.

RESULTS AND DISCUSSION

Prediction accuracy

Calibration tests were performed to predict values for samples unrelated to the sample used to develop the calibration. Table 3 presents a comprehensive overview of the outcomes obtained from predictions made on the distinct test set. To gauge the quality of the predictions, determinant coefficient of the prediction (R^2_p) was computed, reflecting the extent to which the variation in the independent set was estimated by the calibration.

When training samples were employed from each specific plantation site to construct prediction models, the prediction accuracy for each plantation site was (B1) $R^2 = 0.46$, (B2) $R^2 = 0.44$, (L1-NF) $R^2 = 0.28$ and (L3-SF) $R^2 = 0.34$. However, it was not feasible to predict the MFA for B3 and L2-F. Conversely, when training samples from all countries were used to create prediction models for each country, the prediction accuracy for each plantation site was (B1) $R^2 = 0.49$, (B2) $R^2 = 0.49$ and (L1-NF) $R^2 = 0.17$. However, it was not feasible to predict the MFA for B3, L2-F and L3-SF.

Figure 1 shows the average of (a) all raw spectra, (b) all pretreated NIR spectra after smoothing, baseline correction, and second derivative calculations, and (c) regression coefficients from PLS model used for prediction. The regression coefficients of the PLS model used for the prediction were derived from the training set. Although the average results are shown, the results for Brazil and Laos exhibit a similar trend. Regression coefficients can be used to determine important spectral regions that are responsible for the correlation with MFA. It is obvious in the absorbance spectrum that at least three peaks exist at wavelengths of 1916, 2092 and 2361 nm. A highly negative impact and positive impact on the calibration model were found at these wavelengths, indicating that their absorbance was highly correlated with MFA. The absorption bands at these wavelengths can be attributed to chemical components such as cellulose and water (Table 4) (Schwanninger et al., 2011). As reported in a previous study (Okuyama et al., 1994), MFA is strongly dependent on cellulose content. Therefore, cellulose composition has a relatively high influence on the calibration model, as indicated by the regression coefficients at wavelengths of 2092 and 2361 nm.

The different latitudes

Utilizing samples gathered from different latitude regions in Brazil, the predictive precision exhibited a lower accuracy in the B3 plantation, while demonstrating higher accuracy in other locations. In Porto Alegre (B3), we observed an NIR to predict the MFA (Table 3). The MFA values displayed minimal variation across the three plantations in Brazil.

Impact of data variability on estimation

To ascertain the underlying cause, we conducted an examination of data variability within each plantation site (Table 2). The outcomes revealed that the B3 plantation exhibited less data variability in comparison to the other plantations, potentially influencing the diminished accuracy in predictions. Previous research has indicated that wood quality tends to remain relatively stable within a specific plantation, independent of the growth rate (Kojima et al. 2009a, 2009b, 2009c). In essence, if the prediction model were exclusively created for a single plantation, the data variability would be minimal, consequently leading to poorer accuracy. To achieve highly accurate MFA predictions, it is deemed necessary to develop a prediction model incorporating data samples collected from diverse plantations, thereby enhancing the variability within the dataset.

The different habitat environment

Utilizing samples collected from different environments in Laos, the predictive accuracy proved to be significantly low for each plantation site, particularly in L1-NF and L3-SF (Table 3). Similar to the findings in Brazil, the MFA values displayed minimal variation across the three plantations in Laos. While the Brazilian plantation, aged over 16 years, had reached the commercial harvest stage, the Laos 3-years-old plantation was still below the harvest age and predominantly consisted of juvenile wood. It is postulated that the inferior accuracy of the data collected in Laos can be attributed to the higher prevalence of a substantial amount of juvenile wood, compared to Brazil. The data was scattered due to the presence of juvenile wood, but the accuracy of the MFA forecast did not improve.

Samples from all sites

The relationships between measured values and NIR-estimated values are shown in Figure 2. A prediction model for MFA was employed, incorporating data from both Brazil and Laos, to compute the NIR-estimated MFA values for Brazil and Laos (Figure 2 A). In the case of Brazil, the MFA estimation was performed using the entire Brazilian dataset (Figure 2 B), while for Laos, the estimation was conducted utilizing the complete Laos dataset (Figure 2 C). The correlations between measured values and NIR-estimated values are $R^2 = 0.53$, $R^2 = 0.62$, and $R^2 = 0.48$, figure 2, A, B, and C, respectively. The prediction model for MFA, incorporating data from both Brazil and Laos, was employed to derive the NIR-estimated MFA values for Brazil and

Laos. The correlations between the measured values and NIR-estimated values were R^2 = 0.50 for Brazil and R^2 = 0.45 for Laos (Table 3).

In this study, we collected wood samples from numerous sites across various locations in Brazil, enabling the comprehensive MFA prediction model that encompassed the entire dataset from Brazil. Remarkably, this consolidated model demonstrated higher accuracy compared to the individual plantation-specific prediction models within Brazil. This result can be attributed to the increased variability present in the comprehensive dataset from Brazil. Likewise, when all samples from Laos were analyzed, the prediction model yielded superior accuracy compared to the individual plantation-specific models (Table 3).

Schimleck et al, (2002, 2005) reported a strong correlation in samples containing juvenile wood with an MFA exceeding 20 degrees. However, in this study, the observed correlation was not as strong as reported by Schimleck et al, (2002, 2005) in any case. Schimleck et al, (2002, 2005) estimated the measured MFA using SilviScan and conducted X-ray irradiation on the radial surface. In contrast, in this study, MFA was measured using X-ray diffraction and performed X-ray irradiation on the tangential surface. Furthermore, in addition to these differences, the plantation environment in this study consisted of a mixture of distinct individuals, which could potentially impact the detected correlations.

CONCLUSIONS

The results of this study highlight the feasibility of utilizing near-infrared spectroscopy (NIRS) as a reliable method for predicting wood quality, particularly the microfibril angle (MFA). The predictive potential of NIRS was demonstrated by developing a robust MFA prediction model using wood samples collected from various regions in Brazil and Laos. In the different latitude regions of Brazil, the variability of the data had a noticeable impact on the accuracy of the predictions. As a result, the prediction model that incorporated data from multiple regions exhibited higher accuracy compared to models based solely on individual plantations. On the other hand, in different habitats in Laos, the presence of juvenile wood emerged as a significant factor contributing to decreased prediction accuracy.

When combining data from both Laos and Brazil, the prediction accuracy of the model decreased. This decline is primarily attributed to the issue of prevalent juvenile wood in the Laotian samples. Consequently, it became evident that, in order to develop an effective prediction model, it is imperative not to rely solely on a single site but rather create separate models accounting for the immaturity of the timber, while acknowledging the necessity of incorporating variation within the dataset.

Hence, it is evident that in order to develop an effective prediction model, it is imperative to account for the variability of the data and avoid relying solely on a single region. Furthermore, the presence of juvenile wood necessitates the creation of distinct prediction models. These findings highlight the importance of considering data variability, in addition to factors such as age or diameter, when evaluating wood quality using NIRS. To enhance and broaden the scope of the prediction models, it will be essential to gather data from a broader range of wood species and growth regions.

It can be suggested that when predicting the properties of biological samples such as wood, increasing the number of sample locations may enhance the accuracy of the prediction. However, it is crucial to ensure that conditions, such as the number of years of growth, are consistent.

ACKNOWLEDGEMENTS

We greatly appreciate the assistance of Flosul (Porto Alegre), Aracruz cellulose S.A. (Posto da Mata), Companhia Vale do Rio Doce (Açailândia) and Oji Forest & Product Co. (Laos) in random order, for their contribution to the sample preparation. This work was supported by a JSPS Grant-in-Aid (No. 21K20597) and the FFPRI Support Program for researchers with family obligations.

Data availability

Data generated or analyzed during this study are provided in full within the published article.

Competing interests statement

The authors declare there are no competing interests.

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Tables

Table 1. Detailed geographical coordinates of the sampled sites, along with the corresponding tree ages and the total number of trees sampled.

Table 2. Range of wood properties for training set and test set for each plantation.

Table 3. Statistical analysis of MFA values for both the training set and the test set, categorized by each wood district. The prediction coefficient of determination (R^2) was calculated based on the predictions made specifically for the distinct test set.

Table 4. Assignment of absorption bands in the NIR region. Peak number corresponds to those in Figure 1.

Figure caption

Figure 1 (a) Average of all raw spectra, (b) Average of all pretreated NIR spectra by smoothing, baseline correction, and second derivative calculations. (c) regression coefficients from the PLS model used for prediction.

Figure 2 Relationships between measured MFA and NIR-estimated MFA. MFA prediction model, integrating data from both Laos and Brazil, was employed to determine the estimated MFA using near-infrared spectroscopy (NIRS) for (A) Brazil and Laos. For (B) Brazil, the MFA estimation utilizing the complete Brazilian dataset was conducted, while for (C) Laos, the estimation was carried out using the entirety of the Laos dataset.

sample No.	Country	city	latitude and longitude	climate	tree age	number of sample trees	number of samples
B1		Acailandia	5º5'S, 47º39'W	tropical	18	18	69
B2	Brazil	Posto da Mata	18º05'S, 39º33'W	tropical	17	21	85
B3		Porto Alegre	30º05'S, 51º10'W	subtropical	16	22	91
L1-NF		Paksuun (non-flooded)	18º19'N, 104º02'E	tropical	3	16	64
L2-F	Laos	Nong boua PK (flooded)	18º19'N, 104º02'E	tropical	3	16	64
L3-SF		Nong boua PK (slightly flooded)	18º19'N, 104º02'E	tropical	3	13	52

Table 1

780x118mm (144 x 144 DPI)

		MFA(°) for training set					MFA(°) for test set				
		Average	Min	Max	STDEV	sample number	Average	Min	Max	STDEV	sample number
Brazil	B1	14.3	9.6	24.6	3.1	56	13.9	10.2	18.0	2.5	13
	B2	12.4	7.8	22.2	2.7	69	11.8	8.4	16.2	2.5	16
	B3	9.3	7.2	13.8	1.3	73	9.5	7.8	13.2	1.4	18
	Brazil all	11.8	7.2	24.6	3.2	198	11.5	7.8	18.0	2.8	47
Laos	L1-NF	11.0	8.4	17.2	1.9	52	11.5	8.4	14.8	2.1	12
	L2-F	16.9	10.9	25.6	3.8	52	17.0	11.6	24.8	3.8	12
	L3-SF	11.7	8.0	17.6	2.0	42	12.4	9.2	16.0	2.0	10
	Laos all	13.3	8.0	25.6	3.9	146	13.7	8.4	24.8	3.7	34
ALL		12.4	7.2	25.6	3.6	344	12.4	7.8	24.8	3.4	81

Table 2

184x49mm (300 x 300 DPI)

Training set					Test set		
		Number of factors	R2	RMSE		R2	RMSEP
Brazil	B1	1	0.94	3.45	B1	0.46	1.78
	B2	2	0.95	2.69	B2	0.44	1.78
	B3	2	0.99	1.14	B3	NA	1.53
Laos	L1-NF	1	0.97	1.93	L1-NF	NA	2.13
	L2-F	2	0.98	2.94	L2-F	NA	3.1
	L3-SF	1	0.97	1.94	L3-SF	0.34	1.57
Brazil	Brazil all	2	0.95	2.64	B1	0.49	1.73
					B2	0.49	1.70
					B3	NA	1.64
					Brazil all	0.62	1.68
					Los all	0.19	3.27
Laos	Laos all	2	0.96	2.69	L1-NF	NA	2.12
					L2-F	0.17	3.32
					L3-SF	NA	2.24
					Laos all	0.48	2.63
					Brazil all	NA	2.80
ALL	ALL	2	0.95	2.84	Brazil all	0.50	1.91
					Laos all	0.45	2.69
					ALL	0.53	2.28

Table 3

178x129mm (300 x 300 DPI)

peak number	wavelength(nm)	component	Bond vibration
1	1916	Water	O-H antisymmetric, stretching vibration, +O-H deformation vibration of H_2O
2	2092	Cellulose	O-H and C-H deformation vibration, +O-H stretching vibration
3	2361	Cellulose	O-H deformation vibration or C-H deformation vibration,+C-H stretching vibration or C-H_2 stretching vibration

Table 4

608x177mm (144 x 144 DPI)

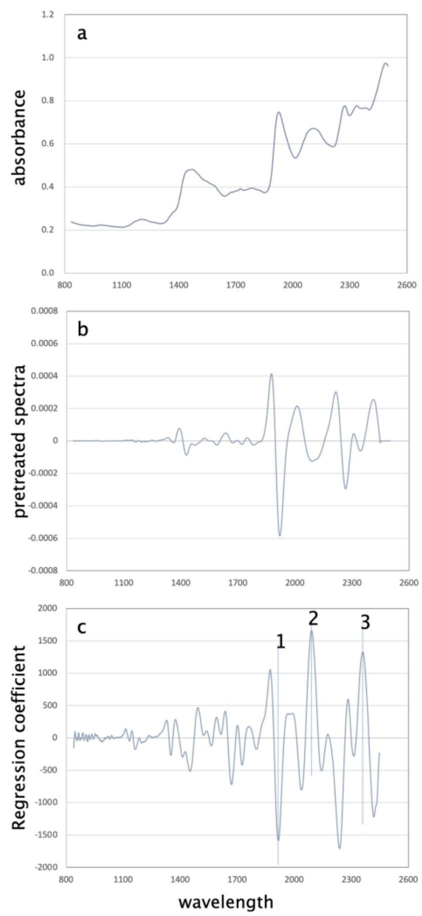

Figure 1. (a) Average of all raw spectra, (b) Average of all pretreated NIR spectra by smoothing, baseline correction, and second derivative calculations. (c) regression coefficients from the PLS model used for prediction.

175x268mm (300 x 300 DPI)

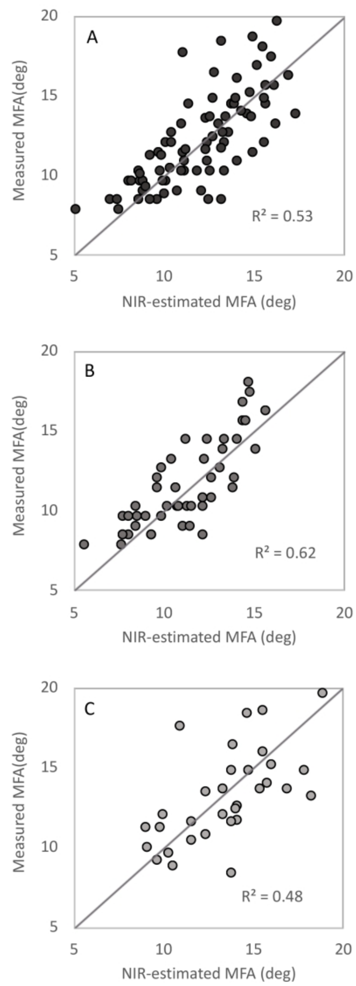

Figure 2. Relationships between measured MFA and NIR-estimated MFA. MFA prediction model, integrating data from both Laos and Brazil, was employed to determine the estimated MFA using near-infrared spectroscopy (NIRS) for (A) Brazil and Laos. For (B) Brazil, the MFA estimation utilizing the complete Brazilian dataset was conducted, while for (C) Laos, the estimation was carried out using the entirety of the Laos dataset.

209x297mm (300 x 300 DPI)