

Non-Destructive Prediction of Moisture Content of Philippine *Coffea arabica* and *Coffea liberica* Green Beans Using Locally-Developed NIR Spectroscopy Instrument

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Abstract

Near-infrared (NIR) spectroscopy is a spectroscopic technique that uses the NIR region of the electromagnetic spectrum and analyzes the degree of absorption. It is widely used in agriculture to determine rapidly and non-destructively the internal or external qualities of agricultural products since traditional techniques are costly, labor-intensive and time-consuming. In green coffee beans, moisture content (MC) is one of the most important quality parameters; hence, rapid and reliable measurement of the attribute is essential. This study aimed to predict the MC of green coffee beans of four varieties of *Coffea arabica* (Typica, Caturra, Red Bourbon and Yellow Bourbon) and two accessions of *Coffea liberica* (Tolentino and Calabuso) using the local NIR spectrometer developed by previous researchers. Absorbance spectra of 100 samples for each variety/accession were collected and their MC was analyzed using the gravimetric method. The multivariate calibration models that considered 30 components in the region of water were established using R software by partial least squares (PLS) regression and validated using a separate set of samples. Results showed that the PLS regression model could develop multivariate models that can predict the MC of coffee beans. Among the six coffee samples, the calibration model for *C. arabica* – Typica obtained the highest cross-validated coefficient of determination ($R^2_{cv} = 0.8556$) with cross-validated root mean square error of prediction (RMSEPCv) of 0.6355 while *C. liberica* – Tolentino obtained the lowest R^2_{cv} (0.6169) with RMSEPCv of 1.128.

Keywords: partial least squares, root mean square error of prediction, multivariate calibration models, coefficient of determination

1. Introduction

Coffee (*Coffea* spp.) has been a valuable international trade commodity for which quality is of prime importance. It is an export-oriented crop earning around 20 billion US dollar a year. In 2019, the estimated annual income of the coffee sector as a whole was at least \$220 billion with about 100 million families around the world depending on it for their living (International Coffee Organization [ICO], 2019). Interest in coffee quality assessment is impelled by the need to supply the consumer with a consistently high-quality product at an affordable price. Indeed, quality is a major aspect of the modern coffee industry because high-quality product is a basis for success in today's particularly competitive market (Barbin *et al.*, 2014; Yusmanizar *et al.*, 2019a).

Chemical constituents of the roasted beans determine the quality of coffee as a beverage. Raw coffee beans contain a wide range of different chemical compounds, which react and interact each other at all stages of coffee roasting, resulting in greatly diverse final products (Ribeiro *et al.*, 2011). Moisture content (MC) is one of the most important quality parameters of green coffee beans. MC is one of the regulated quality standards of green coffee beans in most of the importing and exporting countries (Adnan *et al.*, 2017). MC beyond and away from the safety range of 8.0-12.5% based on fresh matter diminishes the bean quality and ruins its safety (Reh *et al.*, 2006; Pittia *et al.*, 2007; ICO, 2013). Green coffee beans above 12.5% are not allowed to be transported and traded (Van Hilten *et al.*, 2011). Moreover, MC below 8% causes shrunken beans and an unwanted appearance (Gautz *et al.*, 2008). However, beans with above 12.5% MC causes several consequences like microbial growth and altered sensorial quality of the final product; it also promotes mycotoxin formation causing risks to human health (Palacios-Cabrera *et al.*, 2004; Pardo *et al.*, 2005; Reh *et al.*, 2006). Several standards for reference, routine and rapid methods are already established in determining MC in green coffee (Reh *et al.*, 2006).

Sensory, mechanical and functional properties, nutritive values, and purity (presence of defects) are the parameters to be considered in food quality analysis. Traditional techniques used in food quality assurance analysis, namely chemical and Soxhlet method for crude fiber, starch, and oil; Kjeldahl method for protein; and gravimetric or oven drying method (hot-air oven or vacuum oven) for MC, are costly, labor-intensive and time-consuming. In coffee, sensory panel evaluation is still the ultimate tool in assessing its quality

which is laborious and not cost-effective. As a consequence, infrared spectroscopy is gaining attention since it is capable of resolving certain problems arising from traditional techniques (Siesler *et al.*, 2002; Rajput *et al.*, 2019; Yusmanizar *et al.*, 2019a).

Near-infrared (NIR) spectroscopy (NIRS) is a spectroscopic method that uses the NIR region of the electromagnetic spectrum and analyzes the degree of NIR absorption. It is based on the absorption of electromagnetic radiation in the region ranging from 400 to 2500 nm. The radiation in the NIR, which interacts with samples, can be absorbed, transmitted or reflected, and captured by utilizing different measurement modes of the instrument. The NIR spectra comprise broad bands that correspond with the fusion of vibration modes (O–H, N–H, and C–H) and overtones of molecular vibrations (Wang, 2016). Hence, quality characteristics of samples can be measured by analyzing their NIR spectrum (Qiao *et al.*, 2015).

NIRS has obtained a wide acceptance as a tool in evaluating food quality (Rodriguez-Saona and Allendorf, 2011). It is recognized as reliable, low-cost and simple requiring low instrument maintenance (Jing *et al.*, 2010; Santos *et al.*, 2012; Krämer *et al.*, 2015; Qiao *et al.*, 2015; Tolessa *et al.*, 2016). A rapid and non-destructive technique in determining the internal or external qualities of agricultural products, NIRS requires minimal sample preparation, needs no reagents and produces no waste (Qiao *et al.*, 2015; Tolessa, *et al.*, 2016). Norris (1964) first used NIRS in agriculture to rapidly measure the MC in grains including the protein and fat in agricultural products (Davies and Grant, 1987; Nicolaï *et al.*, 2007; Lim *et al.*, 2014). Many studies have also presented the potential of NIRS as an alternative technique in coffee quality analysis. It has been used in differentiating Arabica from Robusta coffee (Kemsley *et al.*, 1995; Downey *et al.*, 1997; Esteban-Díez *et al.*, 2004, 2007; Buratti *et al.*, 2014), determining the defective from non-defective coffee beans (Craig, *et al.*, 2012, 2015), detecting possible coffee adulteration (Ebrahimi-Najafabadi *et al.*, 2012; Reis *et al.*, 2013), authenticating the coffee origin (Wang *et al.*, 2009), and in predicting the sensory and cup quality of roasted coffee beans (Ribeiro *et al.*, 2011; Tolessa *et al.*, 2016).

Applying suitable NIR spectral data, the partial least squares (PLS) regression model has emerged as the most popular multivariate calibration techniques in developing the model of coffee bean qualities (Pizarro *et al.*, 2007; Mo *et al.*, 2011; Santos *et al.*, 2012; Zhang *et al.*, 2013; Adnan *et al.*, 2017; Yusmanizar *et al.*, 2019b).

The locally-developed NIR spectroscopy instrument, which costs less than half of the cost of the commercial ones (4 to 5 million pesos), was used initially in predicting the moisture and crude protein of cracked corn and milled rice, and nitrogen content of rice leaves (Umali *et al.*, 2014). This study attempted to utilize the same instrument in predicting the MC in green coffee beans.

2. Methodology

2.1 Collection and Processing of Coffee Samples

Four varieties of *C. arabica* ripe coffee cherries, namely Typica, Caturra, Red Bourbon, and Yellow Bourbon were collected in January 2015 in La Trinidad and Atok, Benguet, Philippines. *C. liberica* accessions of Calabuso and Tolentino coffees were obtained in March 2015 from Tagaytay City, Cavite. The wet processing method was used to produce green beans.

2.2 Preparation of Coffee Samples for NIR Scanning

Green coffee beans obtained from wet processing of Arabica and Liberica were manually sorted to remove the defective beans. Afterward, 350 g of “good coffee beans” were sorted by size for 5 min using the standard 12” square wood sizing screens attached to the Ro-Tap Sieve Shaker. Only those samples that retained in sieve sized 18 and above were used in accordance with the Specialty Coffee Association (SCA) standards for grading of coffee beans. A total of 100 pieces of Petri dishes packed with 100 g of clean and sorted green coffee bean samples per variety/accession were prepared.

2.3 Laboratory Analysis of MC

The gravimetric method was used to measure the actual MC of the coffee samples. A total of 100 samples of 15 g per variety/accession were oven-dried at 100 ± 5 °C for 24 h. The weight loss of the samples was then measured. The wet basis MC of the green bean was computed using Equation 1.

$$MC = \frac{W_w - W_d}{W_w} \times 100 \quad (1)$$

where:

W_w = weight of sample before drying

W_d = weight of sample after drying

2.4 Development of Spectral Signature of Coffee Beans through NIR

The locally-developed CvSU NIR spectrometer (Figure 1), made by Umali *et al.* (2014), comprises a rotating sampling cup attached to a stepper motor and spaced with the light source attached to a predetermined distance. It has a servo motor that holds the probe reference with a reference material attached to it. The method includes placing the sample into the sampling cup which is rotated by the stepper motor. The PircPlus software is used to make a command from the main computer. The incident light passes through the sample and reflects the sensor through a fiber optic cable. The sensor that is connected to the main computer processes the detected light signal and creates spectra. It then sends the created spectra to the main computer which interprets and saves the data. The instrument works within the wavelength range of 950-1650 nm.

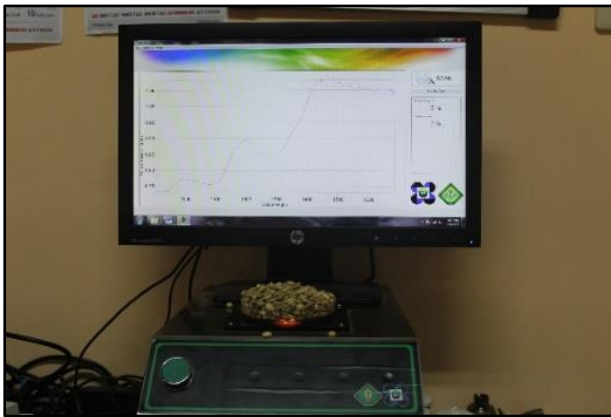


Figure 1. The locally-developed CvSU NIR spectroscopy instrument

To ensure the models' ability to predict the MC of future samples per variety/accession, 100 samples were scanned through the NIR instrument to develop the spectral signatures of the coffee bean variety/accession. Two sets of data were prepared per variety/accession. The first 80 sets of data were utilized in developing the calibration models (or prediction models for MC)

while the remaining 20 sets of data were used to validate the prediction models.

2.5 Development of Multivariate Calibration Model

Multivariate calibration models in predicting the MC of green coffee beans, considering 30 components of the region of water, were developed using the first 80 sets of data of each variety/accession by the PLS regression model. The PLS regression model was used in the study since it is commonly used in chemometrics to establish regression models for physical and chemical properties from spectral data (Santos *et al.*, 2012). The optimal number of latent variables for each prediction model was estimated with the leave-one-out (LOO) cross-validation method. To determine the optimal model among the calibration models developed, the values of the coefficient of determination in calibration (R^2) and the coefficient of determination in cross-validation (R^2_{cv}) were evaluated. The values of the root mean square error of prediction from cross-validation ($RMSEP_{cv}$) in all the models were evaluated to assess the PLS model error.

2.6 Validation of the Prediction Models

The remaining 20 sets of spectral signatures of the scanned samples were used to validate the prediction models for MC. Prediction values from developed models were compared with the actual MC values which were obtained through the gravimetric method by air-oven drying. To assess the reliability of the models for predicting the MC of green beans, the percent error was computed using Equation 2.

$$\text{Percent error} = \frac{|M_v - P_v|}{M_v} \times 100 \quad (2)$$

where:

M_v = measured/actual value

P_v = predicted value

3. Results and Discussion

3.1 Measured MC of the Coffee Samples

The measured MC of the Arabica and Liberica coffee samples obtained through air-oven drying is shown in Table 1. It can be observed that the measured MC of the coffee samples ranged from 8.65 to 19.60%.

Table 1. Range of measured MC of the coffee samples

Coffee samples	Range of MC (%)
<i>C. arabica</i> – Red Bourbon	11.00 – 18.67
<i>C. arabica</i> – Typica	8.65 – 17.33
<i>C. arabica</i> – Caturra	10.00 – 16.00
<i>C. arabica</i> – Yellow Bourbon	10.00 – 13.33
<i>C. liberica</i> – Calabuso	11.71 – 19.60
<i>C. liberica</i> – Tolentino	10.30 – 16.61

3.2 Spectral Signatures for MC of Coffee Samples

The spectral signature for MC of the different varieties of Arabica and accessions of Liberica coffees using the locally-developed instrument is shown in Figures 2 and 3, respectively. Note that the spectral wavelength of water occurs at 1450 wavelength.

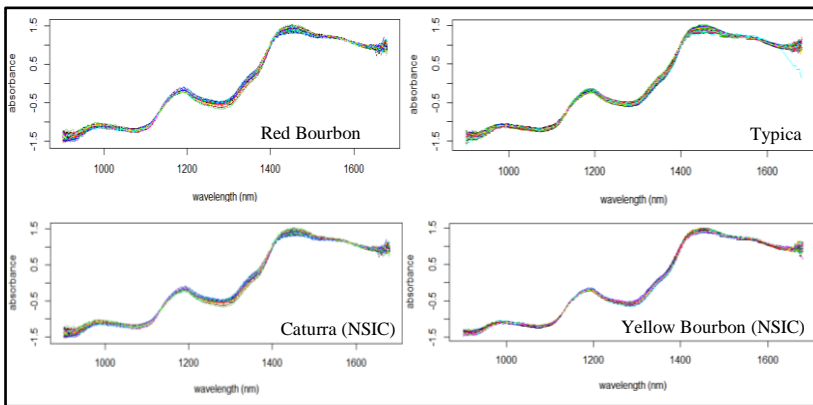


Figure 2. Spectral signatures for MC of *C. arabica* varieties

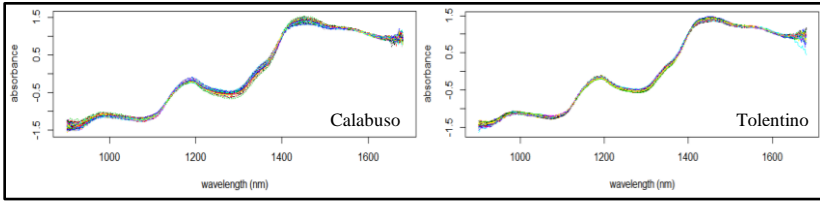


Figure 3. Spectral signatures for MC of *C. liberica* accessions

3.3 PLS Multivariate Calibration Models for the Prediction of MC

Table 2 presents the PLS multivariate calibration and the cross-validation of the prediction models for the MC of *C. arabica* and *C. liberica* coffees. The R^2 of the samples for calibration ranged from 0.96 to 0.99. Results of the cross-validation among the samples show that the $RMSEP_{cv}$ ranged from 0.3903 to 1.128 and the R^2_{cv} ranged from 0.6169 to 0.8556. Among the six coffee varieties/accessions, the model for MC of *C. arabica* – Typica obtained the highest R^2_{cv} (0.8556) with $RMSEP_{cv}$ of 0.6355 while *C. liberica* – Tolentino obtained the lowest R^2_{cv} (0.6168) with $RMSEP_{cv}$ of 1.128. This implies that the locally-developed NIR instrument is suitable in predicting the MC of *Coffea arabica* varieties and *Coffea liberica* accessions.

Table 2. PLS calibration, cross-validation and test results for the prediction of MC of coffee samples

Coffee samples	Calibration		Cross-validation		Validation		
	R^2	RMSEP	R^2_{cv}	$RMSEP_{cv}$	Measured (ave)	Predicted (ave)	% Error
<i>C. arabica</i>							
Red Bourbon	0.9901	0.1563	0.6752	0.9005	14.03	14.27	1.71
Typica	0.9992	0.0467	0.8556	0.6355	11.53	12.58	9.11
Yellow Bourbon	0.9997	0.0158	0.7918	0.3903	11.67	12.57	7.71
Caturra	0.9930	0.1412	0.6645	0.9758	12.51	12.95	3.52
<i>C. liberica</i>							
Calabuso	0.9871	0.1868	0.6803	0.9285	15.29	14.20	7.13
Tolentino	0.9567	0.3792	0.6169	1.128	13.05	13.14	0.69

The goodness of fit of the multivariate calibration model for each coffee variety of *C. arabica* (Red Bourbon, Typica, Caturra, and Yellow Bourbon) and accession of *C. liberica* (Calabuso and Tolentino) is displayed in Figures 4, 5, 6, 7, 8, and 9, respectively.

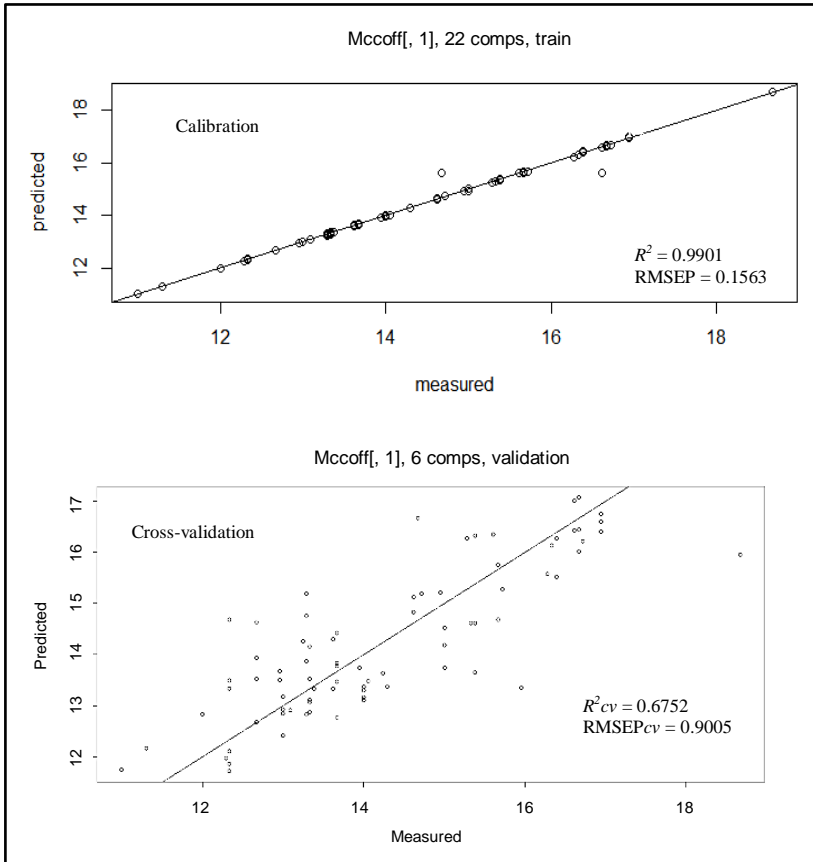


Figure 4. The goodness of fit of the multivariate calibration model for determining the MC of *C. arabica* – Red Bourbon

3.4 Prediction of MC

The calibration models were tested using the remaining 20 sets of spectral signatures of each green coffee bean variety/accession to predict MC of green coffee beans. The percent error (Table 2) of the predicted values for MC of Arabica and Liberica coffees ranged from 1.71 to 9.11% and 0.69 to 7.13%, respectively.

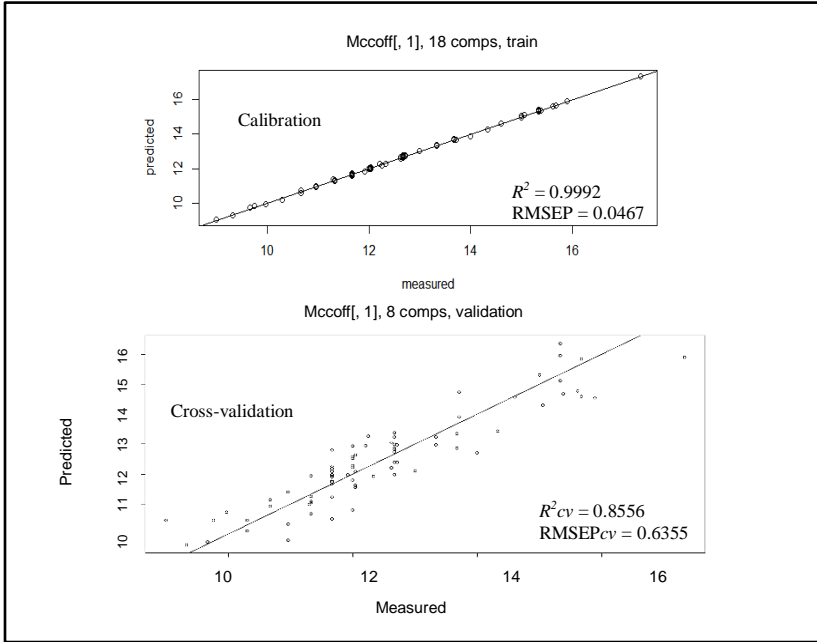


Figure 5. The goodness of fit of the multivariate calibration model for determining the MC of *C. arabica* – Typica

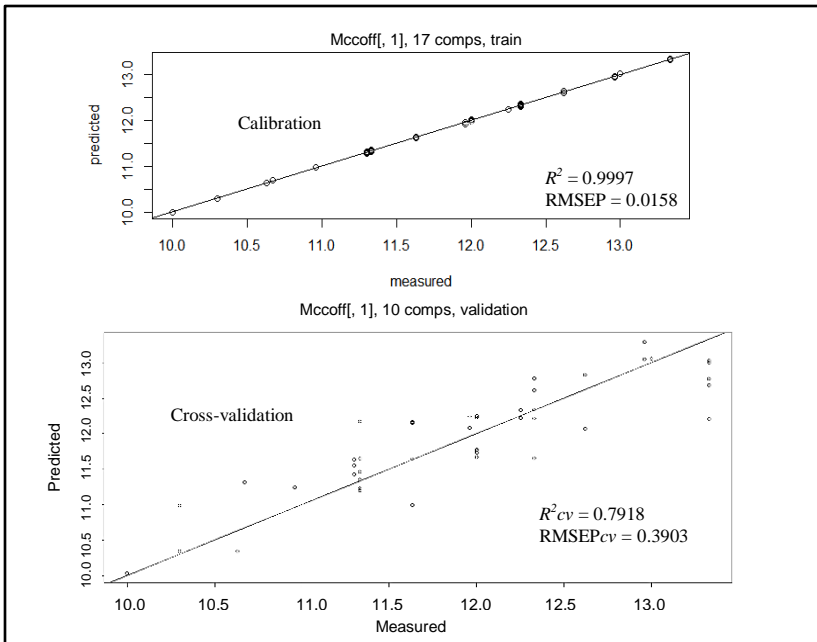


Figure 6. The goodness of fit of the multivariate calibration model for determining the MC of *C. arabica* – Yellow Bourbon

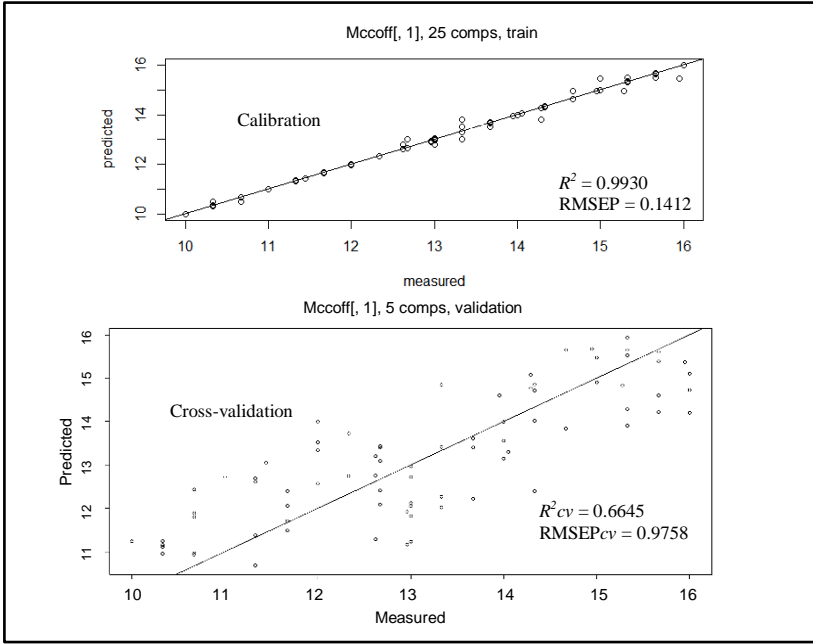


Figure 7. The goodness of fit of the multivariate calibration model for determining the MC of *C. arabica* – Caturra

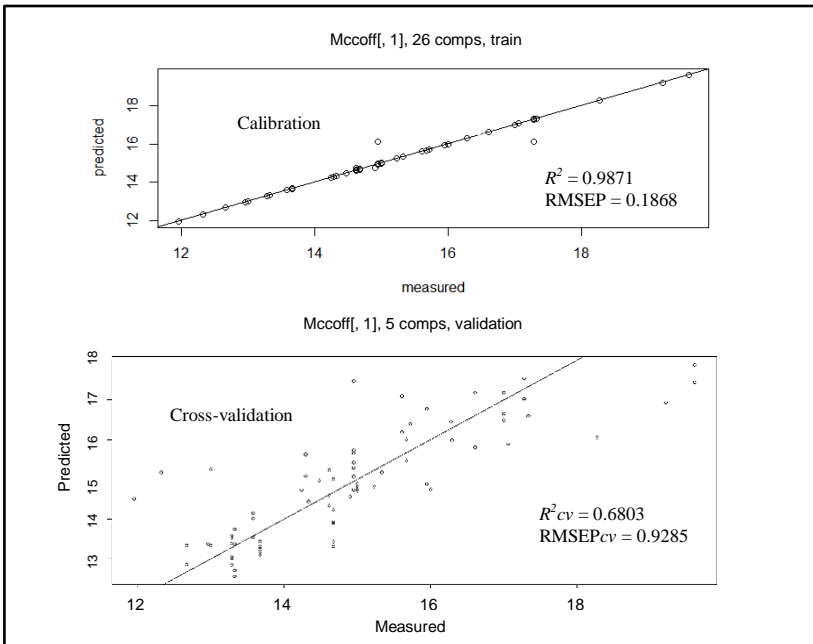


Figure 8. The goodness of fit of the multivariate calibration model for determining the MC of *C. liberica* – Calabuso

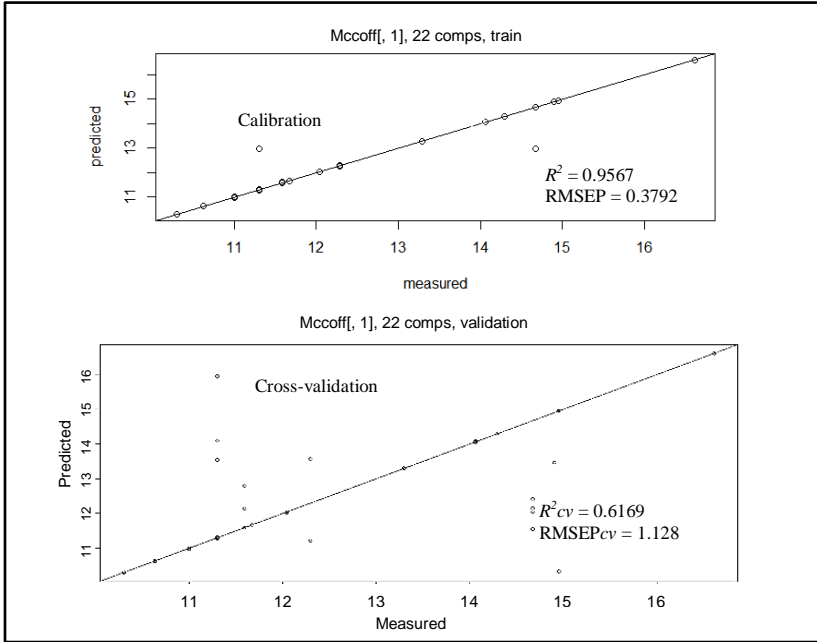


Figure 9. The goodness of fit of the multivariate calibration model for determining the MC of *C. liberica* – Tolentino

4. Conclusion and Recommendation

The study proved that the locally-developed CvSU NIR spectroscopy instrument is a valuable tool providing a non-destructive method in predicting the MC of Philippine *Coffea arabica* and *Coffea liberica* green beans. This technique also validated a rapid prediction of the MC in green beans. The calibration model for *C. arabica* – Typica coffee demonstrated the best performance in predicting the moisture of green beans as evidenced by its high R^2_{cv} (0.8556) with $RMSEP_{cv}$ of 0.6355.

The calibration and prediction models developed in the present study may be used to craft a software that will be installed in the NIR instrument for the non-destructive measurement of the MC of green coffee beans. It is also recommended that calibration and prediction models of other coffee quality parameters such as percent purity, color, and caffeine content be conducted as these properties greatly affect the coffee's overall cup quality.

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