Estimation of moisture content in Liberica coffee by using near infrared spectroscopy

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Abstract: Liberica coffee is a popular one in the global trade market beside Arabica and Robusta. Moisture content (MC) is one of the most important quality parameters of green coffee beans production and trading. NIR spectroscopy is one of the alternative methods for the rapid determination of chemical compounds in the products non-destructively. The purposes of this study are to determine the best calibration model, data transformation, and data pretreatment for moisture content of Liberica coffee. The ground coffee samples were measured by FT-NIRS in the wavelength of 1000-2500 nm, properly the moisture contents of the same samples were determined by the oven method. The obtained spectrums were transformed into absorbance (Log 1/R) and Kubelka-Munk (K/S) units. Data pretreatment, such as standard normal variance (SNV), second derivative (dg2), and multivariate calibration method such as a partial least square (PLS), were carried out to develop the best calibration model. Good accuracy for the prediction of moisture content of Liberica coffee green bean was obtained from the spectral data pretreated with dg2 and Kubelka-Munk(K/S) data transformation with the statistical evaluation values of r (0.87), RPD (2.05), consistency (99%) and CV (5.76 %)

1. Introduction
Indonesia is one of the biggest coffee producer and exporter countries all over the world. A coffee commodity is also Indonesia's fourth-largest foreign exchange earner next to palm oil, rubber, and cocoa. The total coffee plantation area of Indonesia is approximately 1.24 million hectares [1]. There are many types of coffee in the Indonesia coffee market, but in general, the common types are Arabica coffee, Robusta coffee, and Liberica coffee. Among them, Liberica coffee that can be grown on peatland has a unique characteristic and flavor. In Indonesia, Liberica coffee has been developed since the 1970s and then has widely spread in six areas; West Rangsang, Rangsan, Tebing tinggi Barat, Tebing tinggi Timur, and Merbau islands [2]. Present days, Liberica coffee can be found in Sumatra Island, especially in Tanjung Jabung Barat of Jambi Province. The total area of Liberica coffee was 3,028 ha, with a production of 1,214 tons in 2014 [3].

There are many quality parameters for coffee trading. Among them, moisture content has been regulated as one of the quality standards for coffee green bean by most of importer and exporter countries. The appropriate range of moisture content is 8.0 to 12.5% based on fresh matter. If the moisture content of coffee bean is above 12.5%, they are not allowed to be shipping and trading because higher moisture content can be accelerated microbial and fungal growth, which is leading to off-flavor and risk to human health. When the moisture content of the coffee bean is below 8%, it can be shrunken bean and unwanted physical appearance [4].

Therefore, an accurate and fast moisture content determination for the coffee bean is essential for the coffee sectors. The gravimetric method is widely accepted as a reference method to determine the moisture content for all coffee trading countries. Although the gravimetric method is effective for moisture content measurement, there are some inconveniences as sample preparation, time-consuming, and so on. Therefore, alternative methods for rapid determination are required, and one of
them is Near Infrared Spectroscopy (NIRS). NIRS is an increasingly growing non-destructive technique due to its rapidity, simplicity, safety, and so on. According to many references, it is already known that the prediction of moisture and chemical content by NIRS has been developed over the years for many types of specialty coffee [4, 5, 6, 7, 8, 9]. However, NIRS research for Liberica coffee has not been carried out yet. In this study, Standard Normal Variance (SNV) and Second derivative of Savitzky-Golay (dg2) were used as data pretreatment, and NIR spectroscopy was computed by the PLS calibration method. This research is aimed to develop the best calibration model, data transformation, and data pretreatment for moisture content of Liberica coffee.

2. Materials and methods

2.1. Material and Apparatus

Liberica coffee green beans (9.9% moisture content) obtained from the Indonesian Industrial and Beverage Crops Research Institute (BALITRI), Sukabumi were used as the main materials. NIR spectrometer type NIRFlex N-500 (BUCHI Labortechnic AG. Switzerland), LC-MS 2020 (Shimadzu, Kyoto, Japan), computer equipped with Unscramble software v 10.4 (CAMO, Norway) and Microsoft excel, grinder, Tyler sieve no.100 mesh, digital scales, and petri dish were the apparatus used in this study.

2.2. Sample Preparation

Liberica coffee green beans were ground with a grinder as size reducing and sieved by using Tyler sieve No. 100 mesh to get 150µm particle size. Fifty samples of coffee powder were obtained for NIR spectroscopy measurement.

2.3. NIRS Measurement

Fifty samples were used for NIRS measurement. First, 50g of ground coffee was weighted and spread into the petri dish until 1.2 cm depth of that petri dish. After that, NIRS measurement for these samples was conducted by type NIRFlex N-500 in the wavelength of 1000 – 2500 nm with the scan speed of 3 scans per second (Temperature around 22 – 25˚C) and then 150 spectral data were obtained. In this study, two types of reflectance spectral data: 150 spectral data and 50 spectral data were used. One hundred fifty spectral data from 3 times of scanning and 50 spectral data from the average of 3 times scanning results were obtained.

2.4. Water content measurement

After NIRS measurement, water content measurement of the samples was done by using the gravimetric method, according to AOAC, 2000 [10]. Firstly, the dish and its lid were dried in the oven at 105˚C for 3 h and then transferred to the desiccator to be cold. Dish and lid were weighted after cooling. Secondly, 3g of the coffee sample was weighed and placed in the dish. The dishes with the samples were placed in the oven and dried at 105˚C for 3 h. After drying, the dish with partially covered with lid was transferred to the desiccator to be cold. The dish and sample were reweighted after cooling, and the moisture content of samples was calculated by equation (1).

\[
\text{Moisture(\%)} = \frac{W_1 - W_2}{W_1} \times 100
\]

Where \( W_1 \) = weight of the sample before drying (g)
\( W_2 \) = weight of the sample after drying (g)
2.5. **NIR Data Processing and Analysis.**

The reflectance spectrums were transformed into absorbance (log 1/R) and Kubelka Munk Model (K/S). In this study, standard normal variance (SNV), second derivative (dg2), and combination of both were applied as NIR data pretreatment to develop the best prediction for moisture content of Liberica coffee. Samples were divided into calibration and validation samples that were 102 and 48 in 150 spectral data and 34 and 16 samples in 50 spectral data, respectively. The calibration and validation for NIR data processing and chemical contents were carried out by using Partial Least Square (PLS). Accuracy of NIRS method for moisture content prediction of Liberica coffee was determined from the result of coefficient correlation (r), Standard Error of Calibration set (SEC), Standard Error of Prediction set (SEP), Coefficient of Variation (CV) and Ratio of Performance to Deviation (RPD) \[5\]. NIR data processing and analysis were carried out by using the unscrambler software V10.5 trial version (CAMO, Norwegia).

3. Results and discussion

3.1. **NIR Spectra and Moisture Content of Liberica Coffee Beans.**

The original absorbance spectra for moisture content of Liberia coffee are shown in figure 1. There are two kinds of absorbance spectra, which are obtained by the data transformation of Absorbance (Log 1/R and) and Kubelka Munk Model (K/S). According to Budiastra et al. [6], the absorbing peak of spectra for moisture content can be generally found in the wavelength 980nm, 1450 nm, and 1940 nm. However, it may be needed to do data pretreatment methods to determine the moisture content of Liberica coffee.

![Figure 1. NIR absorbance spectra for moisture content of Liberica coffee green bean](image)

The results obtained from the gravimetric method were used as reference data for moisture content table 1. According to ASEAN stan 31: 2013 [11], the moisture content of coffee green bean should not be more than 13%. In this case, although the moisture content of coffee green bean before grinding was 9.9%, the average moisture content of Liberica coffee green bean powder was 4.91% because of the evaporation process during grinding. It is lower than the moisture content of Arabica Bondowosono coffee green bean powder 5.43% [5]. This is due to the origin, variety, and some physiological activities during sample processing.
Table 1. Moisture content of Liberica green bean powder

| Process    | Range (%) | Mean (%) | Standard Deviation (%) |
|------------|-----------|----------|------------------------|
| Calibration| 3.85-6.13 | 4.91     | 0.57                   |
| Validation | 4.27-5.47 | 4.83     | 0.305                  |

3.2. Results of calibration and validation.

Tables 2 and 3 show the best calibration and validation model obtained from 50 spectral data and 150 spectral data by using the PLS method. Table 2 is for the results using 50 spectral data, and a good calibration model can be found in the spectral data pretreated with dg2, which is data transformed by Kubelka-Munk (K/S) method. In this model, PLS factor 3 indicated a good result as correlation coefficient r (0.84), RPD (1.86), consistency (76%), and CV (6.12 %). In table 2, all of the spectral data except dg2 pretreated spectra were attained poor accuracy prediction model for moisture content because dg2 can reduce linear baseline and increase spectra resolution. Table 3 shows the results obtained from data transformation and data pretreatment using 150 spectral data. In table 3, the best calibration model can be found in the spectra as the correlation coefficient r (0.87), RPD (2.05), consistency (99%) and CV (5.76 %), which pretreated with the second derivative of Savitzky-Golay (dg2) by Kubelka-Munk data transformation.

Table 2. Result of Calibration and Validation for moisture content by PLS (50 spectral data)

| Data Transformation | Data Pretreatment | Factor | r    | SEC (%) | SEP (%) | CV (%) | RPD     | Consistency (%) |
|---------------------|-------------------|--------|------|---------|---------|--------|---------|-----------------|
| Absorbant           | original          | 4      | 0.67 | 0.4194  | 0.4400  | 8.53   | 1.35   | 95              |
|                     | SNV               | 2      | 0.66 | 0.4287  | 0.4126  | 8.72   | 1.32   | 103             |
|                     | Dg2               | 2      | 0.67 | 0.4134  | 0.3899  | 8.13   | 1.27   | 106             |
| Kubelka Munk        | original          | 2      | 0.66 | 0.4282  | 0.4339  | 8.71   | 1.33   | 98              |
|                     | SNV               | 2      | 0.66 | 0.4242  | 0.4139  | 8.62   | 1.33   | 102             |
|                     | Dg2               | 3      | 0.84 | 0.3053  | 0.4031  | 6.12   | 1.86   | 76              |

Beside that model, the original spectral data and pretreated spectra data using SNV in the data transformed by Kubelka-Munk (K/S) are also found good accuracy for the prediction of moisture content of Liberica coffee as the result of r (0.83), RPD (1.8), consistency (109%) and CV (6.57%) and r (0.86), RPD (1.96), consistency (80%) and CV (6.05%) respectively. Puringsih et al. [5] stated that Kubelka-Munk spectral data based on absorption and scattering could reduce the scattering effects that exist in the original reflectance data. In the Absorbant (Log 1/R) data transformation, the original spectral data and pretreated spectral data using SNV also get good result as the value of r (0.86), RPD (1.97), consistency (81%) and CV (6.01%) and r (0.85), RPD (1.88), consistency (80%) and CV (6.3%) respectively. According to Pizarro et al. [7], the standard normal variance (SNV) data pretreatment can get good accuracy model for prediction by normalizing the spectrum when the wavelength varies between samples. However, the spectral data pretreated with dg2 in Absorbant (Log 1/R) data transformation was attained poor accuracy prediction model for moisture content in table 3.

Table 3. Result of Calibration and Validation for moisture content by PLS (150 spectral data)

| Data Transformation | Data Pretreatment | Factor | r    | SEC (%) | SEP (%) | CV (%) | RPD     | Consistency (%) |
|---------------------|-------------------|--------|------|---------|---------|--------|---------|-----------------|
| Absorbant           | original          | 10     | 0.86 | 0.2963  | 0.3648  | 6.02   | 1.97   | 81              |
|                     | SNV               | 8      | 0.85 | 0.3102  | 0.3855  | 6.30   | 1.88   | 80              |
|                     | Dg2               | 3      | 0.75 | 0.3823  | 0.4291  | 7.79   | 1.52   | 89              |


### Table 1: Summary of Data Transformation Results

| Data Transformation | Pretreatment | Factor | r     | SEC (%) | SEP (%) | CV (%) | RPD | Consistency (%) |
|---------------------|--------------|--------|-------|---------|---------|--------|-----|-----------------|
| Kubelka Munk        | original     | 10     | 0.83  | 0.3233  | 0.2956  | 6.57   | 1.80 | 109             |
|                     | SNV          | 10     | 0.86  | 0.2977  | 0.3722  | 6.05   | 1.96 | 80              |
|                     | Dg2          | 4      | 0.87  | 0.2829  | 0.2836  | 5.76   | 2.05 | 99              |

Among these results in both tables 2 and 3, the good calibration can be found in the spectra of Dg2 pretreated and data transformed by the Kubelka-Munk (K/S) method. Besides, all spectral data that is transformed by the Kubelka-Munk model in the use of 150 spectra show good results to predict the moisture content of Liberica coffee. Purningsih et al. also [5] concluded that spectral data transformed by the Kubelka-Munk method combined with Dg2 is better than absorbance data (Log 1/R) in the prediction of caffeine, trigonelline and chlorogenic acid in coffee green bean powder. According to Mouazen et al.[12], RPD value must be higher than 1.5 so that the calibration model can be used. Therefore, all of the results of spectral data in table 3 can be used as the calibration model.

Figures 2 and 3 show the scatter plot of reference and prediction values of moisture content with 50 spectral data and 150 spectral data using the PLS method. Both of these figures show a correlation and RPD between moisture content predicted using NIRS and moisture reference using the gravimetric method, indicated by R-value > 0.85 and RPD > 1.5. This result shows that the obtained calibration model using the PLS method could predict moisture content in Liberica green coffee beans.

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![Figure 2](image_url)  
**Figure 2.** Plots of moisture content referenced vs. predicted using the PLS method in 50 spectral data
Figure 3. Plots of moisture content referenced vs. predicted using the PLS method in 150 spectral data

4. Conclusion
The best calibration model for good prediction of moisture content in Liberica coffee green bean was obtained by the original absorbance spectral which is data transformed by Kubelka-Munk (K/S) method combined with second derivative (dg2) data pretreatment and the number of PLS factor 4 with correlation coefficient $r$ (0.87), RPD (2.05), consistency (99%) and CV (5.76 %). The accuracy of the PLS model transformed by Kubelka-Munk (K/S) is better than Absorbance (Log 1/R) in the prediction of moisture content in Liberica coffee.

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