

Non-Destructive Prediction of Moisture Content in Cascara Using NIR Spectroscopy with PLS and PCR

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Article Info	Abstract
<p><i>Submitted: 3 May 2025</i> <i>Revised: 4 June 2025</i> <i>Accepted: 10 June 2025</i> <i>Available online: 19 June 2025</i> <i>Published: June 2025</i></p> <p>Keywords: Cascara, Moisture Content, NIR Spectroscopy, PCR, PLS</p> <p>How to cite: Safmi, N. A., Andasuryani., Fahmy, k. (2025). Non-Destructive Prediction of Moisture Content in Cascara Using NIR Spectroscopy with PLS and PCR. Jurnal Keteknikan Pertanian, 13(2): 249-264 https://doi.org/10.19028/jtep.013.2.249-264.</p>	<p>Coffee cherry pulp is a by-product of coffee processing that has not been optimally utilized. Coffee cherry pulp can be dried to produce a herbal tea product, known as cascara. As an herbal tea product, moisture content is one of the most important quality parameters for assessing the quality of cascara. Therefore, a method is required to measure the moisture content of cascara. One of the methods developed is NIR spectroscopy, which is non-destructive, fast, and does not require chemicals. The purpose of this research is to explore the application of NIR spectroscopy in predicting cascara moisture content using partial least squares (PLS) and principal component regression (PCR) methods and to evaluate the performance of each method in building an optimal calibration model. Pretreatment of the spectrum data was carried out with standard normal variate (SNV), gap-segment 2nd derivative (dg2), and a combination of SNV+dg2. The results showed that the best prediction of cascara moisture content used the PLS calibration technique with dg2 pretreatment and five factors 5. The values obtained were $R_c^2 = 0.96$, RMSEC = 0.87 %, SEC = 0.87 %, $R_p^2 = 0.90$, RMSEP = 1.22 %, SEP = 1.16 %, and RPD = 3.44. Meanwhile, the PCR method produced good predictions using SNV pretreatment, with a factor of 8. The prediction results were $R_c^2 = 0.89$, RMSEC = 1.40 %, $R_p^2 = 0.90$, RMSEP = 1.33 %, and RPD = 3.15. NIR spectroscopy can predict the moisture content of cascara nondestructively and rapidly.</p>

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1. Introduction

Coffee is a plantation commodity with a huge opportunity to compete in international markets. Indonesia is one of the largest coffee-exporting countries in the world, ranking fourth, after Brazil, Vietnam, and Colombia (ICO, 2023). Coffee from Indonesia is advantageous in terms of its quality, variety, and diverse flavors. This has increased the demand for coffee in Indonesia, especially in various regions of Europe, America, and Asia (Maulani & Wahyuningsih, 2021).

Processing coffee fruit produces 65% of coffee beans from the total harvest, and the remaining 35% consists of coffee by-products such as pulp and husk (Azzahra & Meilianti, 2021). In the process of processing coffee fruit, only the seeds will be taken to be used as coffee drinks, while the remaining pulp and husk become organic waste. The amount of coffee pulp waste generated during processing is quite substantial and has not been optimally utilized. As a result, it has the potential to cause environmental pollution if not properly managed. In general, coffee processing by-products, particularly pulp waste, have been utilized on a small scale as raw materials for composting (Ningrum et al., 2023), bioethanol production (Azzahra & Meilianti, 2021), and as additives in animal feed formulations (Aswanto et al., 2023). Coffee pulp waste can also serve as a source of nutritional compounds, as it contains bioactive components such as antioxidants that are beneficial to health (Iriondo-Dehond et al., 2020), including in products like cascara. Cascara is an herbal tea from dried coffee pulp that can be used as a raw material for making refreshing drinks (Heeger et al., 2017). The drying process is carried out such that the moisture content in the coffee pulp is reduced, resulting in cascara, which has a crispy texture and is classified as a dry herbal tea.

One of the quality parameters of a cascara is its moisture content. High moisture content causes changes in taste and aroma, triggers the growth of bacteria and microbes (Andasuryani & Ifmalinda, 2024), and causes damage during product storage (Sari et al., 2019). Based on SNI 01-3836-2013, the maximum moisture content when storing packaged dry tea products is 8%. Tea moisture content is usually determined destructively using the gravimetric method according to ISO 1573 (Prawira-Atmaja et al., 2021). This method is time consuming, requires chemicals, and is expensive.

Near-infrared (NIR) spectroscopy is a non-destructive method that can be used to quickly determine the moisture content of a product. NIR Spectroscopy responds to the characteristics of the H, OH, and N-H atoms (Shen et al., 2022). This method uses near-infrared signals from the electromagnetic spectrum at wavelengths of 750-2500 nm. The information obtained from the NIR spectrum cannot directly determine the content of the analyzed material; therefore, chemometric methods, such as partial least square regression (PLS) and principal component regression (PCR), are needed.

NIR spectroscopy has been widely applied to analyze the moisture content of various products. The moisture content of Sumatran coffee beans (Andasuryani et al., 2024), moisture content of Sumatran coffee beans (Andasuryani et al., 2024), moisture content of black tea (Zou et al., 2022), and grain moisture content (Sari et al., 2019). Several studies have shown that the PLS method is superior to PCR for moisture content analysis. For example, Budiastira et al. (2023) used PLS in determining the moisture content of vanilla fruit, Hayati et al. (2024) in predicting the moisture content of cocoa beans and Lozano et al. (2025) in predicting the moisture content of coffee beans. However, there is still little information related to the determination of water content in cascara using these two chemometric methods. Therefore, this study aimed to explore the application of NIR spectroscopy in predicting

cascara moisture content using PLS and PCR methods and evaluate the performance of each method in building a good calibration model.

2. Materials and Methods

2.1 Sample Preparation

The sample used was arabica coffee pulp from the coffee processing industry in Solok Regency, West Sumatra. The coffee beans were processed using a full wash. The resulting coffee pulp was dried using a rack-type dryer at 60°C under sunlight until it had a crispy texture. Dried cascara was stored at room temperature using LDPE plastic packaging until data collection. A total of 120 samples were used in this study, each weighing 20g. In general, there are no rules regarding the determination of the number of samples required for the development of calibration models. However, Martín (2022) stated that at least 100 samples are used to build calibration models, although accurate models can still be built with less data.

2.2 NIR Spectra Acquisition

NIR spectral data were collected using a Buchi NIRFlex N-500 solid-state spectrophotometer (Buchi AG, Switzerland) at wavelength of 1000-2500 nm with an interval of 4 nm. The sample was evenly placed in a petri dish and scanned three times. Illuminating the sample with near-infrared light causes some of the energy reflected by the sample to be detected as reflectance (R) data. The reflectance data were converted into absorbance data (A) using Equation 1 (Lozano et al., 2025).

$$A \log (1/R) \tag{1}$$

2.3 Pretreatment of The Spectrum

The absorbance spectra were processed using the Unscramble X 10.4 software. Before predicting the water content of the cascara, the spectral data must first undergo a pretreatment stage to improve the quality of the results. Pretreatment is used to reduce the noise in the spectra (Rivaldi et al., 2019) caused by the equipment and sample characteristics to produce a good model (Soares et al., 2024).

The selection of various pre-treatment methods before chemometric analysis aims to reduce the additive or multiplicative effects caused by light scattering, the presence of a spectrum baseline, or noise in the spectrum (Zhu et al., 2021). The NIR spectra are influenced by the material characteristics. The size and shape of the cascara cause voids between the particles, which affect the transmission of light through the sample and reflectance. The pretreatments used in this study were standard normal variate (SNV), gap-segment 2nd derivative (dg2), and SNV+dg2. SNV pre-treatment reduces interference from noise and eliminates the effect of scattering on the spectrum to produce a better and clearer spectrum (Nurhasanah et al., 2019). Meanwhile, dg2 pretreatment serves to separate the

overlapping spectra from the original spectrum so that the resulting absorbance value is minimized to clarify each peak in the spectrum (Mardiantono et al., 2019).

2.4 Chemical Analysis

Chemical analysis was used as reference data to create a prediction model for determining the moisture content of cascara correlated with the NIR spectral data. The moisture content analysis was based on the ISO 1573 standard. A total of 500 mg of the sample was weighed and placed in an aluminum cup with a known constant weight. The samples were then dried in an oven at 105°C for 6 h. After heating, the samples were weighed again until they reached a constant weight. Subsequently, the wet-basis moisture content (bb) was calculated using Equation. 2 (Prawira-Atmaja et al. 2021).

$$KA (\% \text{ bb}) = \frac{W1 - W2 (\text{gram})}{W1 - W0 (\text{gram})} \times 100\% \quad (1)$$

Where, KA is moisture content (% bb), W0 is the weight of the empty cup (grams), W1 is the weight of the container with the sample before drying (grams), and W2 is the weight of the container with the sample after drying (grams).

2.5 NIR Data Processing and Analysis

The obtained NIR and chemical spectrum data were divided into calibration datasets of 80 samples (2/3) and validation datasets of 40 samples (1/3). The division of the number of samples was performed by considering the similarity of the mean value and standard deviation of the reference moisture content so that the distribution of data in both groups remained balanced. The range of moisture content values in the calibration dataset was wider than that in the validation dataset; therefore, the validation data range remained within the calibration set (Kurniasari et al., 2017). The amount of data used in the calibration dataset before processing must be checked for outliers. Sample outliers are data obtained that deviate too far from other observations. This causes the calibration performance to be poor.

The calibration method was carried out because the obtained spectral data cannot directly provide information on the chemical content of the material. PLS and PCR were used as calibration methods. After the calibration, a validation stage was performed to test the accuracy of the predictions. Validation was performed internally with the calibration dataset (cross-validation) and externally with other data. Cross-validation uses samples that are not used in the calibration process but are still in the same dataset to evaluate the calibration results obtained, so that they can be used to predict samples in different datasets (Zhu et al., 2021).

The statistical parameters used to evaluate the calibration and validation results were the coefficient of determination for calibration (R_c^2), coefficient of determination for cross-validation (R_{cv}^2), coefficient of determination for prediction (R_p^2), root mean squared error of calibration (RMSEC), root mean squared error of cross-validation (RMSECV), root mean squared error of

prediction (RMSEP), standard error of calibration (SEC), standard error of prediction (SEP), and residual predictive deviation (RPD). If the resulting prediction has an R^2 value > 0.91 , the resulting prediction is very good (Mouazen et al., 2005), and predictions that obtain a high R^2 value with a low RMSE value are good (Huang et al., 2021). Standard error values closer to zero indicate that the predictions are accurate. Meanwhile, if the prediction has an RPD value > 3 , the prediction is very good; an RPD value of 2.5 - 2.9 gets a good prediction; an RPD value of 2.0 - 2.4 explains a fairly good prediction; an RPD value of 1.5 - 1.9, which explains that the prediction is rough; and if the RPD value < 1.5 , the prediction cannot be used (Mouazen et al., 2005). These parameters were calculated using Equations 3, 4, and 5 (Malvandi et al. 2022).

$$R^2 = 1 - \frac{\sum_{i=1}^n (y_i - \hat{y}_i)^2}{\sum_{i=1}^n (\bar{y}_i - \hat{y}_i)^2} \quad (3)$$

$$RMSE = \sqrt{\frac{\sum_{i=1}^n (\hat{y}_i - y_i)^2}{n}} \quad (4)$$

$$RPD = \frac{SD}{RMSEP} \quad (5)$$

Where, y_i , \hat{y}_i , and \bar{y}_i describes the reference, predicted and average moisture content values, respectively, and n is the number of samples for calibration or prediction.

3. Results and Discussion

3.1 Spectra NIR

The NIR absorbance spectra of cascara showed absorption peaks in the wavelength range of 1000-2500 nm (Figure 1).

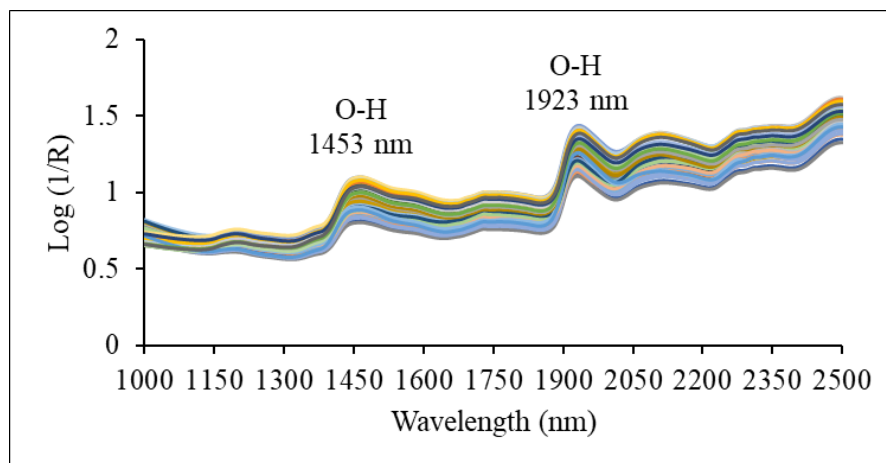


Figure 1. The original spectrum of cascara.

The absorption of NIR spectra occurs because of the presence of near infrared rays received as energy in the sample, which causes vibration and stretching of atomic bonds of organic compounds in O-H, C-H, C-O, and N-H (Cen & He, 2007). Characteristic spectra at wavelengths of 1400-1440 nm and 1900-1950 nm cause vibrations and strains in O-H bonds, indicating the presence of water content (Lengkey et al., 2013). In addition, Cen and He (2007) reported that O-H bonds were observed at wavelengths of 1400, 1450, 1690, and 1900 nm. In this study, O-H absorption peaks were observed at wavelengths of 1453 and 1923 nm (close to 1450 and 1900 nm).

3.2 Data Outlier

The results of outlier detection analysis using the residual sample calibration variance method showed the presence of sample outliers. Outliers were observed in samples with a spectrum peak that was too high. In this study, the 42nd sample was identified as outlier data because it had a very large Y-variance value. Removal of this sample improved the accuracy of the prediction of the moisture content of cascara (Figure 2).

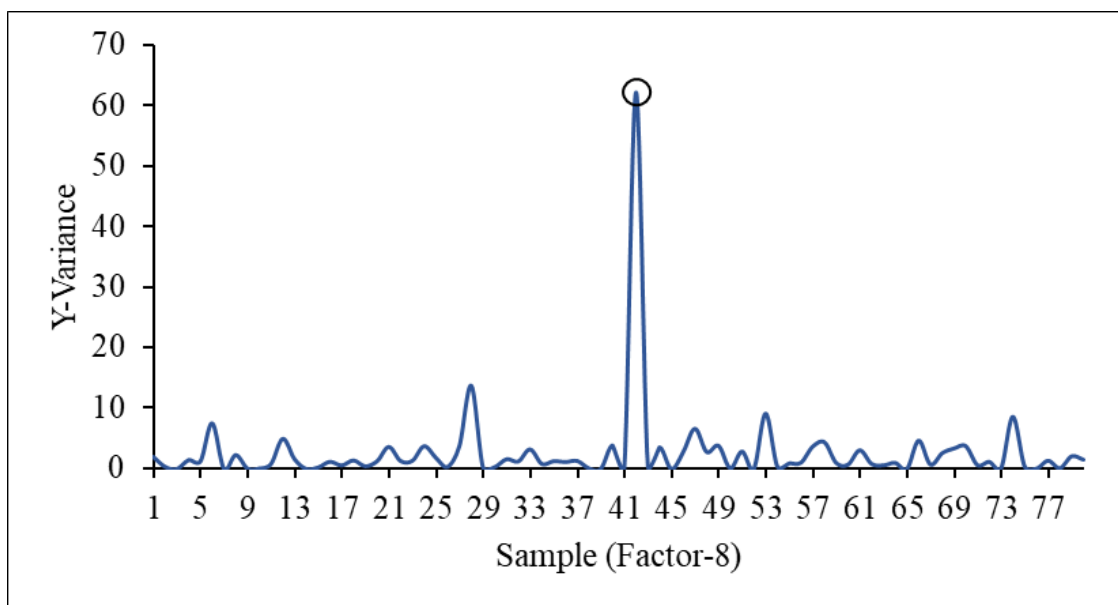


Figure 2. Residual sample calibration variance.

The number of samples removed is in accordance with Rehman and Belhaouari's (2021) statement that the amount of outlier data that should be removed should be no more than 1–5% of the total data to prevent the loss of important information. After checking the outlier data, the number of samples used for subsequent analysis was 79 and 40 for the calibration and validation sets, respectively (Table 1).

Table 1. Statistical data of cascara moisture content in calibration and validation sets.

Parameters	Data sets	Total Sample	Minimum (%)	Maximum (%)	Average (%)	Standard deviation (%)
Moisture Content (% bb)	Calibration	79	7.38	21.21	13.52	4.19
	Validation	40	7.76	20.03	12.18	3.51

3.3 Cascara Moisture Content Prediction

The calibration was built using PLS and PCR methods with all wavelengths from 1000 to 2500 nm as independent variables (1501 samples) and chemical analysis data as dependent variables. When predicting the cascara moisture content, the data were divided into calibration and validation data to prevent overfitting. The purpose of the calibration data was to train the prediction data, whereas the validation data were used to test the reliability of the prediction results obtained. When testing the prediction results, internal validation (cross-validation) and external validation were performed. The difference is that internal validation uses a cross-validation method with the calibration set data, whereas external validation uses other data that are not used in the calibration set (Andasuryani et al., 2024).

The original NIR spectral data of cascara must go through a pretreatment stage before predicting the moisture content. The pretreatments used were SNV, dg2, and a combination of SNV and dg2 to improve the quality of the NIR spectral data for predicting the moisture content of cascara (Figures 3, 4, and 5). Spectra pretreated with SNV showed smoother and denser spectra, while those pretreated with dg2 showed clearer spectrum absorption peaks. In addition to pre-treatment, the selection of the number of factors is also an important aspect in obtaining the best prediction.

The number of factors was chosen with the aim of avoiding overfitting or underfitting to maximize the prediction accuracy (Andasuryani et al., 2014). After pretreatment, the number of factors was reduced using both PLS and PCR (Mardjan & Surbakti, 2023). The calibration and validation results were evaluated using statistical descriptions, including R^2 , RMSEP, RMSEC, SEC, SEP, and RPD values.

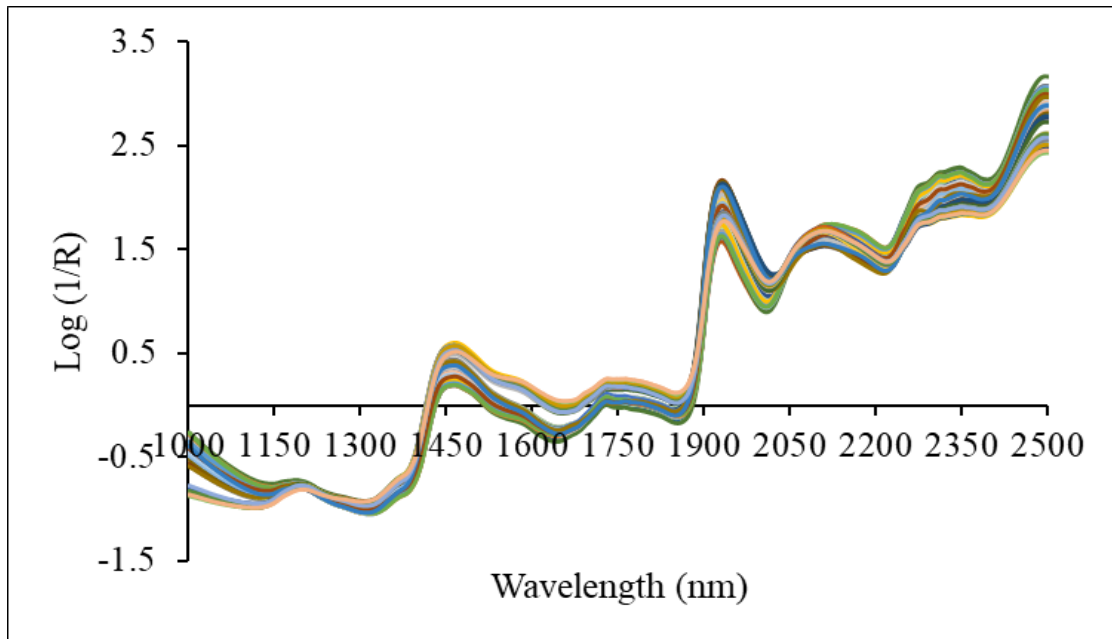


Figure 3. Spectrum pretreatment results using SNV.

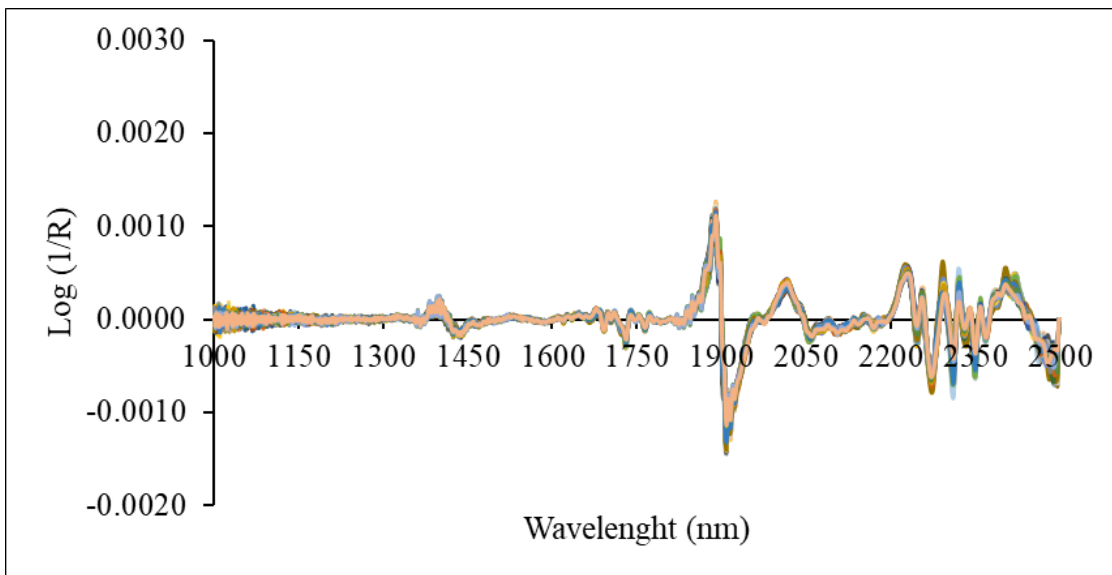


Figure 4. Spectrum pretreatment results using dg2.

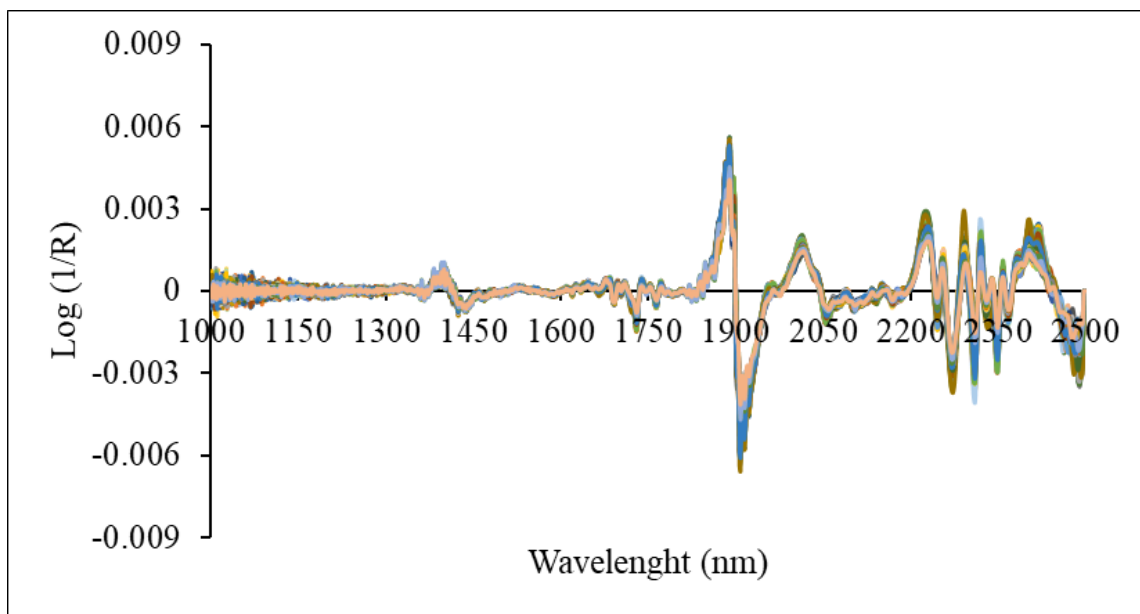


Figure 5. Spectrum pretreatment results using SNV+dg2.

The calibration results showed that the PLS method provided a better prediction performance than the PCR method in determining the moisture content of cascara (Table 2). The PLS method developed using dg2 pretreatment with a factor of five produced excellent predictions with R_c^2 values of 0.96, RMSEC 0.87%, SEC 0.87%, SEP 1.16%, and RPD 3.44. The selection of the optimal number of factors in the original spectrum is larger, that is, eight, because of the noise in the spectra before pretreatment. As more data processing is performed, the number of PLS factors used will decrease to improve prediction performance (Budiastra et al., 2020).

Table 2. Statistical results of cascara moisture content based on NIR spectra with different pretreatment methods.

<i>Pretreatment</i>	Number of Factors	Calibration set			Cross Validation		Validation Set			RPD
		Rc ²	RMSEC (%)	SEC (%)	Rcv ²	RMSECV (%)	Rp ²	RMSEP (%)	SEP (%)	
PLS										
Asli	8	0.89	1.39	1.39	0.86	1.60	0.90	1.31	1.12	2.68
SNV	6	0.89	1.40	1.41	0.86	1.59	0.90	1.32	1.12	3.18
dg2	5	0.96	0.87	0.87	0.89	1.43	0.90	1.22	1.16	3.44
SNV + dg2	3	0.92	1.21	1.22	0.87	1.56	0.89	1.31	1.19	3.20

Continue

Continue

<i>Pretreatment</i>	Number of Factors	Calibration set			Cross Validation		Validation Set			RPD
		Rc ²	RMSEC (%)	SEC (%)	Rcv ²	RMSECV (%)	Rp ²	RMSEP (%)	SEP (%)	
PCR										
Asli	10	0.88	1.46	1.47	0.85	1.66	0.91	1.25	1.09	2.81
SNV	8	0.89	1.40	1.41	0.87	0.56	0.90	1.33	1.15	3.15
dg2	7	0.87	1.53	1.54	0.84	1.69	0.85	1.46	1.38	2.87
SNV + dg2	6	0.87	1.50	1.51	0.86	1.61	0.88	1.38	1.26	3.04

The calibration results obtained have a range of values of R² 0.85 to 0.91 (R²>0.91). This value indicates that all predictions generated were able to determine the water content of the cascara well (Mouazen et al., 2005). The best Rc² value was obtained in the dg2 pretreatment, which was 0.96, indicating that 96% of the reference moisture content contributed to the variation in the NIR-predicted moisture content. In cross-validation, the highest Rcv² value was found to be 0.87, indicating that the prediction was also effective when tested with data not used in the calibration set. SEP and SEC values show that the prediction performance is good if they have low SEC, low SEP, and small differences in SEP and SEC values. The small difference between the SEP and SEC values indicates that the validation data can prove the accuracy of the calibration data used in the development of calibration results (Andasuryani et al., 2014). The dg2 pretreatment significantly improved the prediction accuracy, reducing SEC to 0.87%, which is in line with previous research on NIR-based prediction of moisture content in whole seeds of Brassica species (Prem et al., 2012). The SEC value is expected to be smaller than the SEP value to obtain a good prediction (Mardjan and Surbakti, 2023).

The RPD, RMSEC, RMSECV, and RMSEP values were also used to evaluate the calibration performance. RPD is the ratio between the standard deviation of the reference data and the SEP value. This value indicates the ability of the calibration to predict the components of interest. The analysis method using PLS obtained an RPD value of 3.44 using dg2 pretreatment. The obtained value exceeded the value obtained without pretreatment. This is because dg2 pretreatment can remove unnecessary basic noise and overlapping spectra. This can increase the resolution of the spectrum, clarify the peaks and valleys of the spectrum, and prevent the spectrum from overlapping (Nurhasanah et al., 2019). An RPD value above three indicates that the prediction is very good (Mouazen et al., 2005). According to Shen et al. (2022), an RPD value > 2 indicates that the calibration has excellent predictive ability, and a higher RPD value yields a more accurate and robust prediction (Malvandi et al., 2022). The PLS model with dg2 pretreatment produced the lowest RMSEP in the range of 1.22-1.46%, indicating high accuracy. The lowest RMSEC and RMSECV values obtained in

PLS were 0.87% and 1.43%, respectively, indicating minimal prediction error in the calibration data and good accuracy when tested with new data (Andasuryani et al., 2024).

The PCR method showed that SNV pretreatment gave the best results with a factor count of eight for predicting the moisture content. PCR tends to require more factors than PLS. Mardjan and Surbakti (2023) confirmed that the selection of pretreatment and chemometric analysis methods affects the prediction results. SNV pretreatment was used to correct for multiplicative interference from light scattering and particle size. The SNV reduces the influence of wave interference such that the resulting spectrum is smoother and denser (Nurhasanah et al., 2019).

Although the lowest SEP value obtained is 1.09, it shows good performance; however, this result is not as good as that obtained using the PLS method. The highest R_{cv}^2 value obtained was 0.87, which was still lower than that obtained using the PLS method. The lowest RMSECV value obtained by PCR was 0.56% and the highest RPD value was 3.15. Although the PCR method yielded good results, the PLS method provided more accurate predictions. The best predictions obtained using the PLS and PCR methods are shown in Figures 6 and 7.

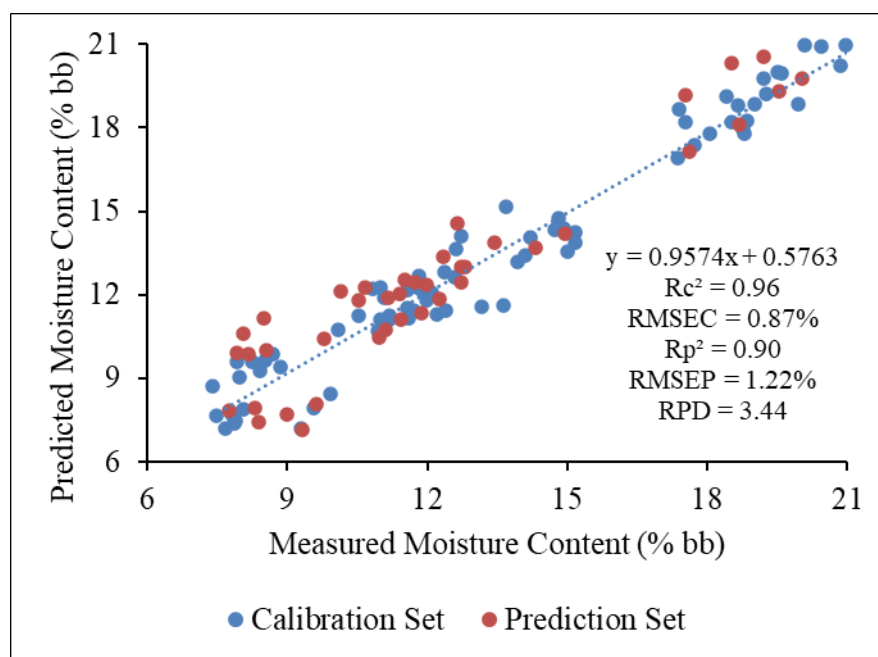


Figure 6. The plot of cascara moisture content results using PLS method with 2nd derivative gap-segment pretreatment.

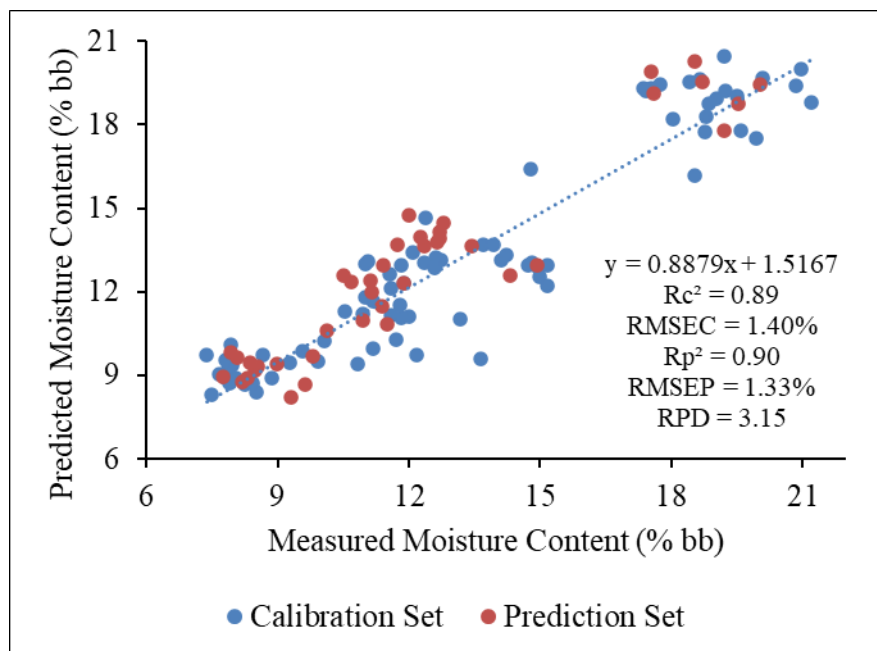


Figure 7. The plot of cascara moisture content results using PCR method with SNV pretreatment.

The results of this study show that the PLS method is superior to PCR in terms of the accuracy and predictive ability of the moisture content of cascara. PLS has a higher coefficient of determination and a lower prediction error, resulting in better predictions for analyzing the moisture content in cascara. These results are in accordance with research conducted by Kurniasari et al. (2017), who showed the superiority of PLS in predicting tannins and total undissolved solids in persimmon fruit, as well as research by Mardjan and Surbakti (2023) in determining the moisture content and piperine content of black pepper powder. The superiority of PLS in determining moisture content was also confirmed in Budiastira and Noviyanti's (2023) study on porang flour and determination of the moisture content of vanilla pods (Budiastira et al., 2023).

Choosing the right pretreatment method plays an important role in improving prediction accuracy. Calibration using the original spectra has shown good calibration results, but the R^2 value obtained is still low; therefore, pretreatment is needed to improve the prediction. SNV and dg2 pretreatments increase the value of statistical parameters, resulting in more accurate calibration (Andasuryani et al., 2014). The dg2 pretreatment provided optimal results for improving the accuracy and reducing the prediction error. The application of gap-segment derivatives can help smooth and eliminate the noise generated by the derivatives themselves (Ditcharoen et al., 2023). The combination of SNV and their derivatives can also improve the accuracy of the resulting predictions (Purningsih et al., 2018).

In the PCR method, SNV pretreatment produced better calibration, in accordance with the research of Mardiantono et al. (2019) in predicting the moisture content of white glutinous rice and research by Hayati et al. (2024) in predicting the moisture content of cocoa beans. The use of SNV pretreatment was also confirmed in the study by Andasuryani et al. (2024) for the prediction of moisture and ash content in sungkai leaf herbal tea. However, not all pretreatments improved prediction performance; for example, the use of dg2 in PCR actually decreased model performance owing to overfitting. This is also due to the loss of important information in the spectrum (Andasuryani & Ifmalinda, 2024).

4. Conclusion

This study showed that NIR spectroscopy combined with chemometrics can predict the moisture content of cascara. Different pretreatment methods for NIR spectra yield different prediction performance. The best calibration method was obtained using partial least squares regression (PLS). The application of gap-segment 2nd derivative pretreatment with a factor of five yielded the best prediction results in determining the moisture content of cascara ($R_c^2 = 0.96$, RMSEC = 0.87%, $R_p^2 = 0.90$, RMSEP = 1.22%, and RPD = 3.44). Calibration using the PCR method with SNV pretreatment and a factor of 8 resulted in good prediction ($R_c^2 = 0.89$, RMSEC = 1.40 %, $R_p^2 = 0.90$, RMSEP = 1.33 %, and RPD = 3.15). These findings demonstrate the potential of NIR spectroscopy as a fast and non-destructive tool for quality measurement in the cascara industry, allowing the real-time measurement of moisture content without the need for chemical analysis.

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