

Rancidity Prediction of Soybean Oil by Using Near-Infrared Spectroscopy Techniques

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Abstract

Purpose: This study evaluated the feasibility of a near-infrared spectroscopy technique for the rancidity prediction of soybean oil. **Methods:** A near-infrared spectroscopy technique was used to evaluate the rancidity of soybean oils which were artificially deteriorated. A soybean oil sample was collected, and the acid values were measured using titrimetric analysis. In addition, the transmission spectra of the samples were obtained for whole test periods. The prediction model for the acid value was constructed by using a partial least-squares regression (PLSR) technique and the appropriate spectrum preprocessing methods. Furthermore, optimal wavelength selection methods such as variable importance in projection (VIP) and bootstrap of beta coefficients were applied to select the most appropriate variables from the preprocessed spectra. **Results:** There were significantly different increases in the acid values from the sixth days onwards during the 14-day test period. In addition, it was observed that the NIR spectra that exhibited intense absorption at 1,195 nm and 1,410 nm could indicate the degradation of soybean oil. The PLSR model developed using the Savitzky-Golay 2nd order derivative method for preprocessing exhibited the highest performance in predicting the acid value of soybean oil samples. **conclusions:** The study helped establish the feasibility of predicting the rancidity of the soybean oil (using its acid value) by means of a NIR spectroscopy together with optimal variable selection methods successfully. The experimental results suggested that the wavelengths of 1,150 nm and 1,450 nm, which were highly correlated with the largest absorption by the second and first overtone of the C-H, O-H stretch vibrational transition, were caused by the deterioration of soybean oil.

Keywords: Near-infrared spectroscopy, Optimal wavelength selection, Partial least-squares regression, Rancidity, Soybean oil

Introduction

It is well established that edible oils have a critical effect on the taste and mouthfeel of foods, whilst enhancing the nutritive value of the food. Edible oils, as one among the three major nutrients, is a high-energy source that contributes to the human body by composing

the cell membranes as the fat-soluble carriers and protecting the hypodermic tissues and organs (May et al., 1983; Lee et al., 1997); however, being used in most homes, restaurant business, and food processing industry, it undergoes changes in smell and taste owing to the chemical or microbial effects during its storage and processing. These changes reduce the nutritive components of the edible oils, lower the quality of the processed foods, and produce toxic agents in humans occasionally. This phenomenon of deterioration of edible oils is known as

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rancidity (Chung and Choe, 2003; Summerfield and Tappel, 1984).

Especially, an oxidation process, which is a representative cause of rancidity, can destroy the essential fatty acids and produce toxic agents and oxidized polymers; therefore, oxidation of edible oils is considered to be critical in terms of palatability, nutritional quality, and toxicity (Choe and Min, 2006). In particular, soybean oil, which is commonly used for frying, exhibits a low oxidative resistance when heated for a long time or repetitively because it contains a lot of unsaturated fatty acids (Park et al., 2003).

Recently, there have been several studies on spectroscopy techniques to estimate the properties of agricultural products or foods (Ben-Dor et al., 1999; Choe et al., 2008). Particularly, a study based on near-infrared spectroscopy (NIRS) has been widely recognized because the NIR region, by virtue of its wavelength (780-2,500 nm), possesses specific information of the test object (ASTM international, 2000). It is known that each functional group (O-H, C-H, and N-H) in a molecule has characteristic absorption frequency bands in the NIR spectrum owing to its vibrational characteristics. Therefore, the exact wavelength at which a vibration happens can be observed by the strengths of the bonds included. Moreover, a near-infrared spectroscopy technique has some advantages in terms of its relative high energy signal compared to far-infrared signal as well as its simple configuration of detector and light source which induce its easy implementation. In addition, the composition of NIRs such as fiber-optic cable, light source, and detector are known to be easy to operate compared to mid-infrared or far-infrared spectroscopic systems.

Despite the advantages described above, the NIR spectra may contain complex and overlapped frequency bands that, accordingly, necessitate additional signal processing computations to analyze the measured spectra. Since the 1990s, a multivariate analysis based on Chemometric method was used in the analysis of spectral features which related to the properties of object (Kim et al., 2008; Kawano et al., 1992). The most representative methods are multiple linear regression (MLR), principal component regression (PCR), and partial least-squares regression (PLSR) (Dull et al., 1992). Among these chemometric methods, PLSR is known to be more robust to reach minimum error with fewer variables than MLR and PCR (Geladi and Kowalski, 1986; Helland, 1988).

However, as the spectra measurements are prone to noise from instruments and their environment, suitable preprocessing methods are being developed for removal of noise (Porep et al., 2015; Rinnan et al., 2009). Herein, we also discuss the optimal variable selection methods that are critical to build a stable and practical model because the developed model includes core subsets of optimal spectra frequencies for better prediction (Sarathjith et al., 2016; Zhang et al., 2017). Previous studies showed that the removal of redundant and noisy wavelengths can improve the prediction performance of a model and reduce its complexity (Kamruzzaman et al., 2013; Kandpal et al., 2016); therefore, we employed four kinds of variable selection method and demonstrated the performance enhancement of the developed prediction models (Lee et al., 1998; Mehmood et al., 2012).

The objective of this study is to develop a non-destructive rancidity evaluation technique of a soybean oil using the near-infrared spectroscopy techniques by using partial least squares regression and optimal wavelength selection methods. Soybean oil samples were deteriorated artificially by boiling them, and titrimetric analysis was used to obtain the reference acid value of each sample. PLSR multivariate analysis with several preprocessing techniques has been employed to quantitatively predict the acid value of soybean oil. In addition, four kinds of optimal variable selection method were used to reveal the optimal wavelength range through model development, and the model performance was also evaluated in conformity with the variable selection methods.

Materials and Methods

Sample materials and treatment

Soybean oils (CJ CheilJedang Corp., Korea) were obtained from a local market and were boiled using an oil bath (WHB-11, DAIHAN Scientific, Korea) to prepare artificially deteriorated test samples. Twelve soybean oil samples, which were kept in tempered glass bottles, were heated at 180°C for 14 days for 4 h every day using the oil bath, and a sample of soybean oil (10 g) was collected ten times every day for titrimetric analysis test and transmission spectra measurements.

Measurements of acid value

The acid value (AV) of the soybean oil samples was

determined using the method of the American Oil Chemists' Society (AOCS, 1977) with suitable modifications. 1 g of the sample was placed in a flask and dissolved in 20 ml of an ether:ethanol solution (2: 1, v/v) while shaking. 1% of phenolphthalein solution was added as an indicator, and titrated with 0.1 N KOH solution until the red color was maintained for 30 s. The formula for calculating the acid value is shown in equation (1).

$$AV = \frac{5.611 \times A \times F}{W} \quad (1)$$

where AV is the acid value, A denotes the volume of KOH (mL), 5.611 is a constant value with equivalence of mass of 0.1 N KOH, W is the weight of sample, and F is the normality of the standard KOH solution.

Measurement of near-infrared transmission spectrum

The soybean oil samples were placed in a plastic cuvette, and the near infrared spectra were measured in a wavelength band of 900-1,700 nm using a near infrared spectrometer (CDI-NIR128, Control Development Inc., USA). The light source was a tungsten halogen light source (LS-1, Ocean optics, USA). A schematic diagram of the measurement setup is shown in Figure 1.

A dataset comprising 140 sets of readings of NIR spectra was obtained by acquiring 10 samples every day for 14 days, whereby daily sampling and measurement were performed in triplicate. The measurement exposure time of the instrument was set to 50 ms, and the measured spectrum was calibrated using equation (2) to convert the measured signal to transmittance. The white reference signal was measured on a plastic cuvette

containing no sample, and the dark reference signal was measured in a dark room without light.

$$\text{transmittance}(\%) = \frac{S_i - D}{R - D} \times 100, \quad (2)$$

where S_i is the raw transmittance of the samples, and D and R represent the raw transmittance of the dark and white referenced spectra, respectively.

PLSR modeling

Spectral preprocessing methods were used to correct the error factors such as noise and light scattering during the measurement of spectra. In this study, two types of scatter correction methods such as multiplicative scatter correction (MSC) and standard normal variate (SNV), three kinds of normalization methods including maximum, range and mean normalization, and Savitzky-Golay 1st and 2nd order derivative methods were employed and evaluated. The prediction model for the acid value of the soybean oil samples was constructed using PLSR which is defined in equation (3). Algorithms for preprocessing and regression modeling were developed using commercial software (Matlab ver. R2016, MathWorks, Natick, MA, USA).

$$\begin{aligned} X &= TP^T + E \\ Y &= TQ^T + F \end{aligned}, \quad (3)$$

where X is the n by m matrix of predictors, Y is the n by p matrix of responses, T is the n by 1 matrix of the score matrix, E and F are Gaussian error terms, and P and Q are the m by 1 and p by 1 loading matrices, respectively.

The selection process of latent variables number, a new variable in the PLSR model, is an important process in PLSR modeling. In this study, we randomly sampled the data 100 times from a cross-validation set of 100 samples to 7:3 (calibration set:validation set) and selected the optimal number of latent variables through cross validation. The model was constructed from the cross-validation sets by applying the optimal number of latent variables, and the accuracy of the model was evaluated using the test set of the remaining 40 samples. The performance of the constructed model was evaluated by root mean square error (RMSE) calculated as described in equation (4).

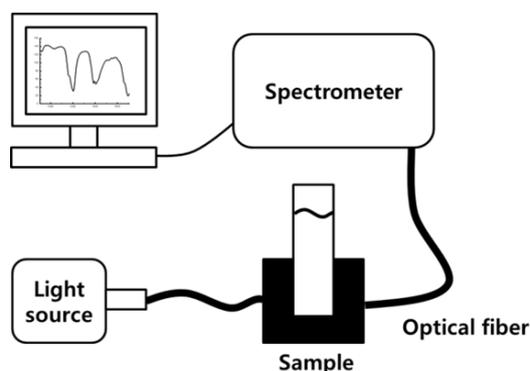


Figure 1. Schematic of near-infrared transmission spectrum measurement.

$$RMSE = \sqrt{\frac{\sum_{i=1}^m (y_{obs} - y_{pre})^2}{m}} \quad (4)$$

where $RMSE$ represents the sample standard deviation of the differences between predicted values and observed values. y_{obs} and y_{pre} are the observed and predicted values, respectively. m is the number of data.

Optimal variable selection

PLS-Beta-Bootstrap

Bootstrap was invented to obtain new datasets through random resampling of a sample population and establishing confidence intervals by taking statistical values from the sampled datasets (Bickel and Freedman, 1981). The PLS-bootstrap method resamples multiple times for a measured spectral dataset and constructs each PLSR model for the resampled datasets. From the distribution of the regression coefficient (beta coefficient) for each PLSR model, the confidence interval of the beta coefficient of each variable is calculated, and if there is a 0 in the confidence interval, the corresponding variable is removed. The confidence interval in this process is calculated as equation (5) (Lazraq et al., 2003).

$$I_k = \bar{b}_k \pm c s_k, \quad (5)$$

where I_k represents the confidence interval, \bar{b}_k is the mean of the beta coefficients at the k th variable, c is a constant that determines the confidence interval, and S_k is the standard deviation of the beta coefficients at the k th variable.

In equation (5), the constant c is explicitly determined regarding the level of significance. A higher significance level could select fewer wavelength bands. In this study, the number of resampling is set to 1,000, and the model accuracy with the selected wavelength is evaluated by changing the c values (Lazraq et al., 2003). In our study, the wavelength was selected by setting the number of resampling times to 1000 and c to 1.

PLS-VIP (Variable Importance in Projection)

Variable importance in projection (VIP) method is a technique that considers the contribution of each variable reflected in weight, which is a PLSR parameter. The VIP value is calculated as written in equation (6).

$$v_j = \sqrt{\frac{p \sum_{a=1}^A [SS_a (w_{ak} / \|w_a\|^2)]}{\sum_{a=1}^A (SS_a)}}, \quad (6)$$

where v_j represents the VIP value, p is the number of variables, SS_a is the sum of squares explained by the a^{th} component, w_a is the weight of the a^{th} component, and w_{ak} is the weight of the a^{th} component at the k^{th} variable.

Hence, the v_j is a measure of the contribution of each variable according to the variance explained by each PLS component where $(w_{ak} / \|w_a\|^2)$ represents the importance of the k^{th} variable. The wavelength selection using the VIP method is performed by setting a threshold value and selecting a variable having a VIP value equal to or higher than a threshold value. In general, a threshold value of 1 is used (Mehmood et al., 2012). In this study, the model was constructed by setting the threshold value to 1.

Results and Discussion

Titration analysis of soybean oil

The rancidity extents of the artificially deteriorated soybean oil samples were estimated as the acid values through titration test, and the results are summarized in Figure 2. The soybean oil samples were boiled at 180°C for 4 h every day during fourteen days and the relative humidity (RH) of the laboratory was controlled at 20% RH. For statistical analysis of the acid values, all the treatment groups were compared using one-way

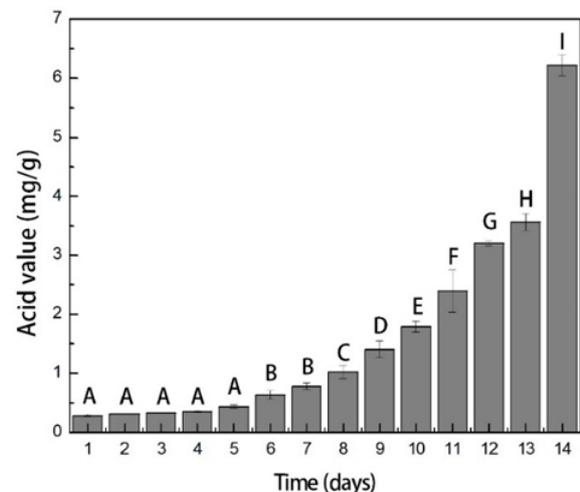


Figure 2. Acid values of soybean oil samples for time periods.

analysis of variance, with a *post hoc* Student-Newman-Keuls (SNK) multiple comparison test. Analysis of variance with a *post hoc* SNK test for multiple comparisons was performed using the commercial statistics software SPSS (ver. 18, IBM, Armonk, NY, USA) to verify the significance of differences between the acid values of each treatment group. The differences between means were assumed to be significant if the *p* value was less than 0.05. As shown in Figure 2, there were significant different rises in the AVs from six days during whole test periods. The result of a *post hoc* SNK multiple comparison is shown in Figure 2, and averaged acid value was considered to be significantly different if the SNK test yielded a *p* value of less than 0.05. Different letters in each column bar of Figure 2 indicate significant differences ($p < 0.05$). The acid value of soybean oil samples which artificially treated for six days was 0.6408 ± 0.0787 mg/g, and the acid value of soybean oil samples treated for whole test period (14 days) was 6.2147 ± 0.1804 mg/g.

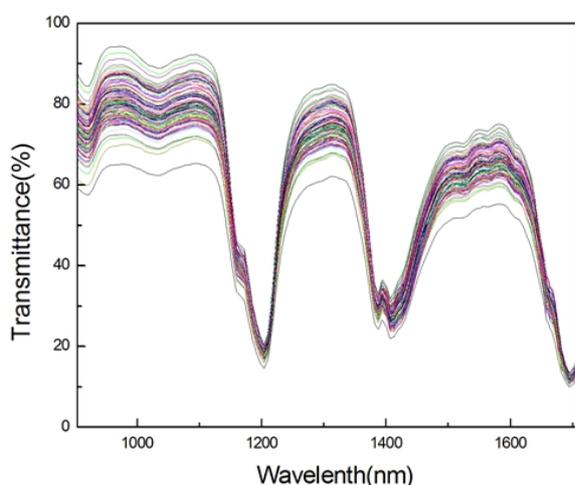


Figure 3. Near-infrared transmission spectra of soybean oil.

Near-infrared transmission spectra of soybean oil

Figure 3 shows the transmission spectra of soybean oil samples before preprocessing applied. As shown in Figure 3, the transmission spectra decrease with increasing test periods as well as high absorptions at around the wavelengths of 1,200 and 1,400 nm. A degradation in a vegetable oil can cause the increase of $-ROOH$, $-OH$, $=O$, and $-CHO$ groups by the formation of hydroperoxide and carbonyl compounds, and also the NIR spectra are absorbed at 1,195 nm and 1,410 nm by the second and first overtone of the C-H and O-H stretch vibrational transitions, respectively; therefore, the sharp decrease of transmission spectra at and around 1,200 nm and 1,400 nm might be demonstrated on the basis of previous study described above.

Estimation of PLSR models for preprocessing methods

Seven kinds of preprocessing methods were employed to enhance the performance of the prediction model, and Table 1 shows the results of the PLSR models for each method. The performance of the developed model was evaluated by the coefficient of determination (R^2) and RMSE, between the titration test results and the prediction values. The test results of the regression models showed that Savitzky-Golay 2nd order derivative method showed the highest performance (R_t^2 : 0.8711, $RMSE_t$: 0.6680), and followed by the regression model with mean normalization preprocessing (R_t^2 : 0.8650, $RMSE_t$: 0.6835). Figure 4 shows the prediction result of two most highly correlated PLSR models for AV estimation.

Table 1. Performance comparison of PLSR models for different preprocessing methods

Preprocessing methods	LV	Cross-validation		Test		
		R^2	RMSE	R^2	RMSE	
raw data	9	0.8807	0.5005	0.8650	0.6837	
MSC	9	0.8826	0.4965	0.8385	0.7477	
SNV	9	0.8872	0.4868	0.8366	0.7521	
Savitzky-Golay 1 st order derivative	7	0.8619	0.5384	0.8114	0.8079	
Savitzky-Golay 2 nd order derivative	7	0.9088	0.4375	0.8711	0.6680	
	maximum	10	0.8864	0.4885	0.8510	0.7182
normalization	mean	9	0.8801	0.5018	0.8650	0.6835
	range	9	0.8823	0.4973	0.8416	0.7406

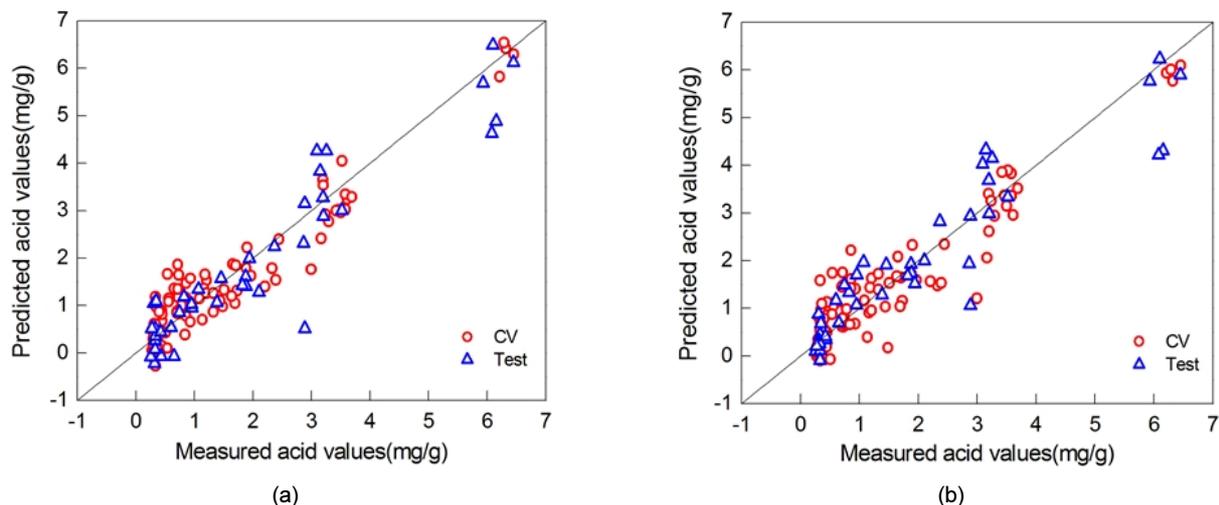


Figure 4. PLSR results for (a) SG 2nd, and (b) mean normalization preprocessing methods.

Comparison of PLSR models for variable selection methods

In this study, two kinds of variable selection methods were employed to select the optimal wavelength bands from whole wavelength dataset (900-1,700 nm). NIR dataset which treated by Savitzky-Golay 2nd order derivative method was used in the works of optimal variable selection because the PLSR model which developed by the preprocessing method of Savitzky-Golay 2nd order derivative method showed the highest performance in the acid value of soybean oil samples. Then, the selected spectra data set was used as a variable of the PLSR model. Figure 5 shows the first and second weight vectors with respect to equivalent latent variables of the PLSR model which obtained from the entire

wavelength's spectra. The weight vector can imply the correlation between the measured spectrum and calculated latent variables through PLSR analysis. It revealed how much the measured spectrum contributed to the formation of corresponding latent variables. As shown in Figures 5 (a) and (b), the highest peaks are observed around the wavelengths of 1,150 nm and 1,450 nm, which are highly correlated with the largest absorption by the second and first overtone of the C-H and O-H stretch vibrational transition, respectively, caused by the deterioration of soybean oil.

Figure 6 shows the optimal wavelength bands determined by two different variable selection methods. By setting the significant level constant c as 1, the extracted wavelength bands were relatively evenly distributed

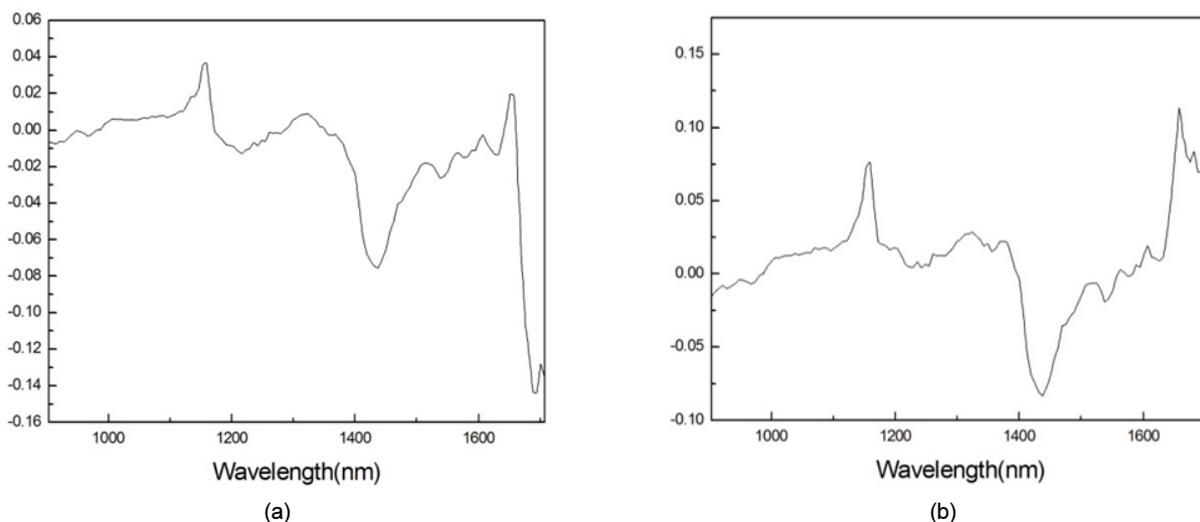


Figure 5. Two weight vectors with respect to equivalent latent variables of the PLSR model: (a) First weight vector and (b) Second weight vector.

over the entire range, as shown in Figure 6(a). VIP method was implemented with threshold values v set to 1.0, and the extracted wavelength bands were relatively evenly distributed over the entire range, as shown in Figure 6(b).

Table 2 shows the cross validation and test results of the PLSR models developed with the optimal variables. Prediction performance of each model was compared, and the PLSR model with whole spectra of wavelength is also described. When the bootstrap was used, the PLSR model with spectra data ($c = 1.0$) corresponding to 210 numbers of wavelengths showed good performance (R_t^2 : 0.8403, $RMSE_t$: 0.7436) when compared to the full PLSR model (R_t^2 : 0.8711, $RMSE_t$: 0.6680). The VIP variable selection method presented that the prediction performance of the PLSR model with spectra dataset which corresponding to 234 numbers of wavelengths is similar (R_t^2 : 0.8081, $RMSE_t$: 0.8150) to that of the full PLSR model. Thus, the prediction performance of the PLSR models with reduced spectra owing to the variable selection methods showed similar performance with the full PLSR model, but not an enhanced performance; however, these optimal variable selection methods can be successfully used to enhance the efficiency of the model runtime and the data saving

effectiveness of the measuring device, while providing a good prediction accuracy by way of selecting the best spectra optimally. Figure 7 shows the prediction performance of the PLSR models that were developed using the optimally selected spectra. As shown in Figure 7, whole samples were fairly well predicted compared to the result of full PLSR model shown in Figure 4. Furthermore, the results confirm the reliability of the model to predict the acid value of soybean oil.

Conclusions

In this paper, we demonstrated the feasibility of predicting the rancidity of soybean oil (using its acid value) by means of a near-infrared spectroscopy technique and optimal variable selection methods. While developing the PLSR model, we successfully used suitable preprocessing methods to remove the noise properties associated with transmission spectra. We investigated seven processing methods, and we found that the Savitzky-Golay 2nd order derivative method exhibited the best performance in estimating the acid value. Furthermore, we employed the

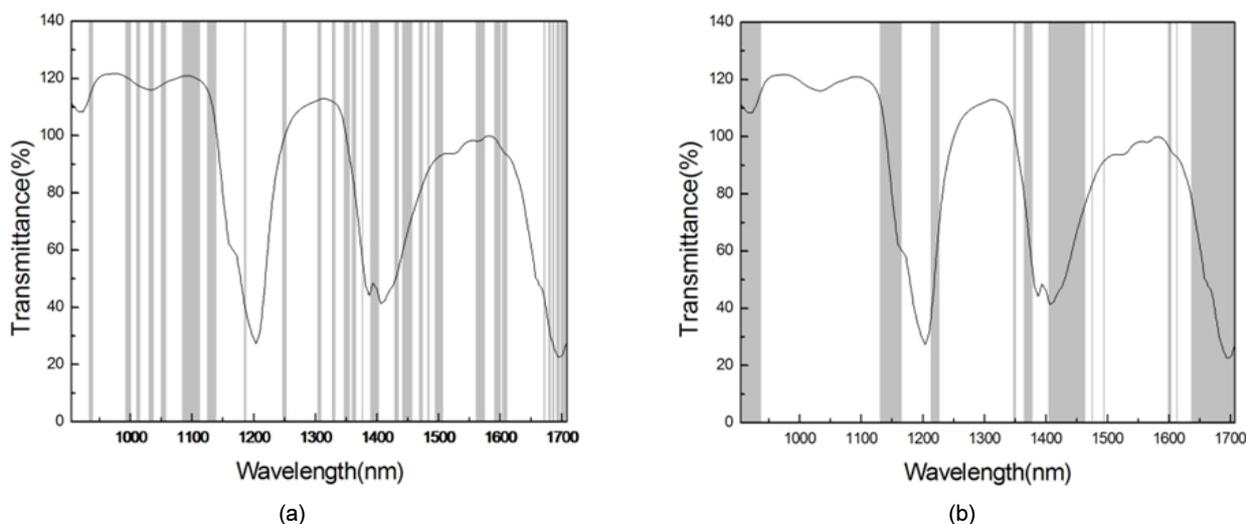


Figure 6. Selected wavelengths by (a) bootstrap ($c = 1.0$, $N = 210$) and (b) VIP ($v = 1.0$, $N = 234$) methods for transmittance spectrum.

Table 2. Comparison of the PLSR model results for different optimal wavelength selection methods

Wavelength selection method	No. of variables	LV	Cross-validation		Test	
			R^2	RMSE	R^2	RMSE
Full PLSR	804	9	0.9088	0.4375	0.8711	0.6680
Bootstrap	210	8	0.8657	0.5311	0.8403	0.7436
VIP	234	8	0.8672	0.5281	0.8081	0.8150

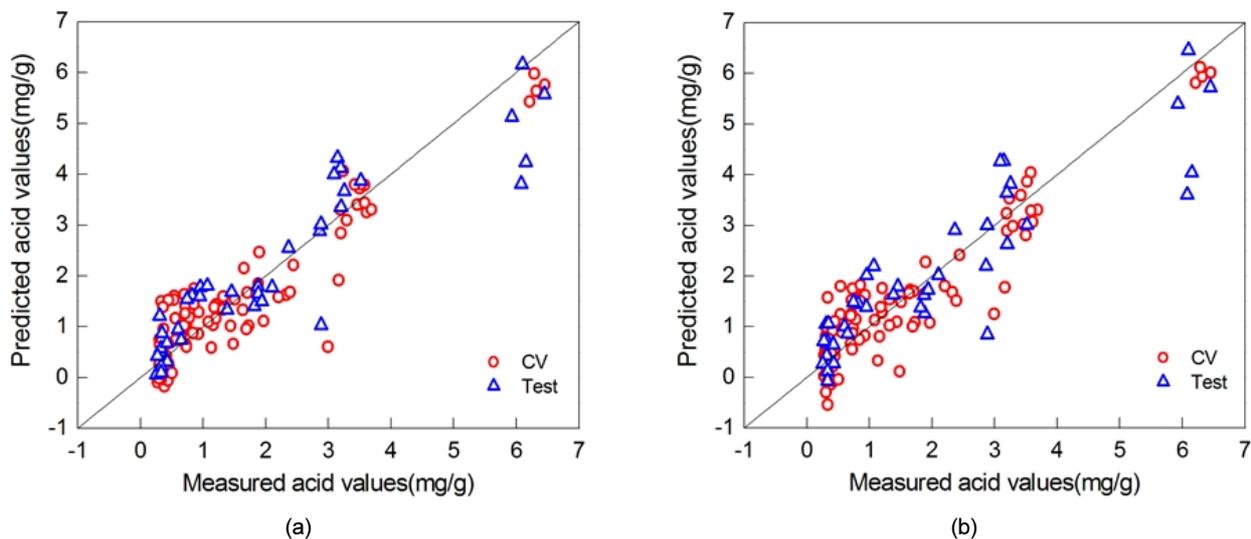


Figure 7. PLSR results for (a) bootstrap and (b) VIP optimal variable selection methods.

optimal variable selection methods because the spectra information corresponding to the entire wavelength is likely to reduce the model accuracy. Results showed that both methods of bootstrap and VIP seems to be the good variable selector to extract the optimal spectra but not enhanced in this study.

It is commonly known that NIRS techniques can be used in both qualitative and quantitative analysis of various objects owing to their mechanisms related to the vibrational energy of molecules. Moreover, this NIR method, being non-destructive and remote sensing, has a lot of advantages, especially in simplifying the activities at the work spot; however, it has a specific limitation in respect of the noise from the instrument and its environment. In order to overcome these drawbacks, multivariate analysis such as PLSR technique with optimal variable selection methods and proper preprocessing methods have been studied and developed similar to our study. We expect that the results of this NIRS study about modeling and statistical evaluations can potentially provide good opportunities for multivariable analysis of agricultural products and foods, as well as many other scientific area.

Conflict of Interest

The authors have no conflicting financial or other interests.

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