Proceedings of AESAP 2016

The 1st International Conference on the Role of Agricultural Engineering for Sustainable Agriculture Production

AESAP 2016
Agricultural Engineering for Sustainable Agriculture Production

Department of Mechanical and Biosystem Engineering
Bogor Agricultural University 2016

ISBN: 978-602-61580-0-0

Editors:
Leopold Oscar Nelwan
Usman Ahmad
Rokhani Hasbullah
I Wayan Astika

PERTETA

INSTITUT PERTANIAN BOGOR

YARI - IPB INDONESIA

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Publisher:
DepartemenTeknik Mesin dan Biosistem IPB (Department of Mechanical and Biosystem Engineering IPB)

Kampus IPB Darmaga PO Box 220 Bogor 16602
Telp/Fax: (0251)8623026

Replicated in Indonesia.

PREFACE

Proceedings of AESAP 2016 contains papers presented in technical session of the 1st International Conference on the Role of Agricultural Engineering for Sustainable Agriculture Production, held at Bogor Agricultural University (IPB) in December 2016.

As many as 28 papers have been presented in the technical session of the conference. The papers covered a broad range of areas in Agricultural Engineering. The papers discuss the topics in postharvest and food engineering, energy and agricultural machinery, land water resources engineering, agricultural structures and environmental engineering and system and management in agriculture production.

We would like to thank all authors for their efforts in preparing their papers. A great appreciation is also given to the members of the proceedings and technical paper committee for their assistance in reviewing the manuscripts. Special thanks to Mr. M. Hafiz and Mr. Lilis Sucahyo for their assistance in formatting the layout of the proceedings.

Bogor, April 2017

Leopold Oscar Nelwan
Usman Ahmad
Rokhani Hasbullah
I Wayan Astika

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A Method to Compensate the Influence of Different Particle Size of Coffee Powder in NIR Calibration Model Performance

Diding Suhandy[*,1] Meinilwita Yulia[2]
[1]Department of Agricultural Engineering, The University of Lampung, Indonesia
[2]Department of Agricultural Technology, Lampung State Polytechnic, Indonesia
Email: diding.sughandy@fp.unila.ac.id[*]

Abstract

In this research, a method to compensate the influence of different particle size of coffee powder in NIR calibration model performance was evaluated. A number of 220 coffee powder samples with two different types of coffee (civet and non-civet) and two different particle sizes (212 and 500 µm) were prepared. Spectral data was acquired using NIR spectrometer equipped with an integrating sphere for diffuse reflectance measurement. A discrimination method based on PLS-DA was conducted and the influence of different particle size on the performance of PLS-DA was investigated. Using the best local calibration models at 221µm and 500µm, prediction results for a prediction sample set at 221 and 500 µm were excellent, with low RMSEP values. However, SEP was significantly increased when samples from different particles size were used in these models; higher RMSEPs resulted. On the other hand, global calibration model based on combinations of different particle size gave better prediction results, with lower RMSEP values for all prediction samples at 221 and 500µm.

Keywords: *Compensation method, global model, local model, particle size, PLS-DA.*

1. INTRODUCTION

Civet coffee or kopi luwak is one the Indonesian specialty coffee and it is regarded as one of the rarest and the most expensive coffee in the world. With very few productions, civet coffee has been a target of adulteration by mixing civet coffee with other cheaper non-civet coffee. Therefore, in order to protect the sustainability of civet coffee, it is important to develop a method for civet coffee authentication. Several reports have been published on the use of UV-visible spectroscopy for rapid method of civet coffee authentication (Suhandy *et al.,* 2016a; Suhandy *et al.,* 2016b; Yulia *et al.,* 2016; Yulia and Suhandy, 2016). However, such method has several limitations. For example, UV-Visible spectroscopy involved the use of extraction process (time consuming) and therefore it is difficult to apply an on-line system of authentication using UV-visible spectroscopy.

NIR spectroscopy has been widely used as a nondestructive method for quality evaluation tool in food and agriculture sectors. For coffee, several research articles have been reported on the use of NIR spectroscopy for evaluation of coffee roasting degree (Esteban-Díez *et al.,* 2004), authentication of coffee types (origin of coffees) (Wang *et al.,* 2009), chlorogenic acid (CGA) content determination (Shan *et al.,* 2014) and etc.

It is well known however, that NIR spectra coming from spectral data acquisition system contains both chemical information of samples as well as physical information of the samples, such as particle size and bulk density (Barnes *et al.*, 1989; Pasikatan *et al.,* 2001). It is common to use various spectral transformations to suppress the physical information in NIR spectra because it obscures the chemical information (Barnes *et al.*, 1989). However, a body of literature has also emerged detailing research focused on quantifying this information. Physical properties that have been correlated to NIR diffuse reflectance spectra include sample temperature, particle size, particle size distribution and compact density (Gupta *et al.*, 2005; Yulia *et al.*, 2014; Barajas *et al.*, 2007).

Several spectral data preprocessing have been used to remove the influence of physical properties and to improve the quality of the calibration model. SNV-DT (standard normal variate-detrending) was developed by Barnes *et al.* (1989) to remove multiplicative interferences of scatter and particle size and to account for the variation in baseline shift and curvilinearity in diffuse reflectance spectra. Multiplicative scatter correction (MSC), also known as multiplicative signal correction, is another pre-processing technique for baseline correction in spectra. It assumes that the wavelength-dependent scatter effects on the spectrum can be separated from the chemical information. The MSC was used to correct for additive and multiplicative effects of NIR spectra and observed that pre-processing with MSC resulted in models that were better than models without MSC (Gomez *et al.,* 2006).

For handling the influence of temperature variation of the samples, several methods have been established for developing calibration models that can compensate for sample temperature variations (Swierenga *et al.*, 2000). One common approach is to include temperature variation in the calibration model both explicitly and implicitly. In implicit approach, a global calibration model is developed in combination with calibration samples from a broad range of temperatures (Peirs *et al.,* 2003). By doing so, it is possible to include all possible wavelengths for optimal determination of the target. However, this method requires a large data set.

In this study, a method to compensate the influence of different particle size of coffee powder in NIR calibration model performance was evaluated using implicit method.

2. MATERIAL AND METHODS

2.1 Coffee Samples

A number of 2 kg ground roasted civet and non-civet coffee samples were collected directly from coffee farmers at Liwa, Lampung, Indonesia. All samples were grinded using home-coffee-grinder (Sayota). Particle sizes were not uniform in the ground coffee powder. In order to check the effect of particle sizes on NIR spectra, coffee powder from ground roasted civet and non-civet coffee was separated into two different particle sizes (212 and 500 μm) by sieving through a nest of U. S. standard sieves on a Meinzer II sieve shaker (CSC Scientific Company, Inc. USA) for 10 minutes. The sieving conditions were the same for every sample class. These experiments were performed at room temperature (around 27-29°C). In this study, a number of 220 coffee powder samples with two different types of coffee (civet and non-civet) and two different particle sizes (212 and 500 μm) were prepared for samples.

2.2. NIR Spectral Data Acquisition

The NIR spectra were collected using a V-670 UV-Visible-NIR Spectrophotometer (JASCO Co., Japan). The spectrometer was equipped with an integrating sphere beside detector (ISN-723, JASCO Co.) which is connected to the spectrometer by an optical fiber. The V-670 double-beam spectrometer utilizes a unique, single monochromator design covering a wavelength range from 190 to 2500 nm. This spectrometer has two detectors: A PMT detector is provided for the UV/VIS region and a Peltier-cooled PbS detector is employed for the NIR region. For light source, this spectrometer utilizes two types of lamps: Halogen lamp and Deuterium lamp.

The NIR instrument was controlled by a compatible Windows XP system, and Spectra Manager (V-670, JASCO Co.) was used to acquire spectra data. A 4 cm by 4 cm square sample holder was used; with care taken to ensure every sample (weight of approx. 0.2 g) was placed into the sample holder and the sample surface kept flat. Each spectrum was measured from 1300 to 2500 nm, with a bandwidth of 20.0 nm, and a scan speed of 1000 nm/min. All experiments were conducted at room temperature (around 20°C).

2.3. Chemometrics Methods

In order to evaluate the possibility of using implicit method for particle size compensation, two types of calibration models were developed namely: local and global PLS-DA model.

2.3.1. Developing local and global calibration models for civet and non-civet coffee discrimination using PLS-DA

For each particle size (212 and 500 μm), we separated the samples into two groups; a calibration and cross-validation sample set (CalValset), and a prediction sample set (Predset). The calibration and cross-validation sample set consisted of 80 samples and 60 samples for particle size 212 and 500 μm, respectively. This sample set was used for developing the calibration model and performing the full cross-validation test for the local PLS-DA model. An uncombined prediction sample set, consisting of 40 samples at each particle size, was used for prediction purposes.

For the combined calibration sample set, the calibration sample sets at each particle size were combined; resulting in 140 samples for calibration sample set (the particle size combinations of 212 and 500 µm). This set was used for developing a calibration model and performing the full cross-validation test and is referred to as the global PLS-DA model.

PLS-DA is working based on PLS regression algorithm (in this study using PLS1 algorithm with only one dependent Y variable), which searches for latent variables (LVs) with a maximum covariance with the Y-variables. PLS-DA attempts to build models that can maximize the separation among classes of objects. In PLS-DA, each sample in the calibration set is assigned a dummy variable as a reference value (variable y), which is an arbitrary number designating whether the sample belongs to a particular class or not (1 = civet coffee, 2 = non-civet coffee).

2.3.2. Calibration model and prediction evaluation

The calibration model was evaluated based on the following parameters: number of PLS factors, coefficient of determination (R^2_{cal}), the RMSECV, and the standard deviation ratio (SDR) of calibration SDR_{cv}) (Golic and Walsh, 2006).

The prediction results were evaluated using several parameters: the coefficient of determination in prediction (R^2_{pred}), the root mean square error of prediction (RMSEP), bias between the actual and predicted value, the bias-corrected standard error of prediction (SEP) and the standard deviation ratio (SDR) of prediction (SDR_{pred}).

3. RESULTS AND DISCUSSION

3.1. Typical Spectra of Civet Coffee with Different Particle Sizes

Figure 1 shows the typical spectra of civet coffee with different particle size (212 and 500 µm). It can be said that the coarser the particles, the greater the penetration of light and the greater the absorbance. This result is in line with the previous reported study (Shan *et al.*, 2014).

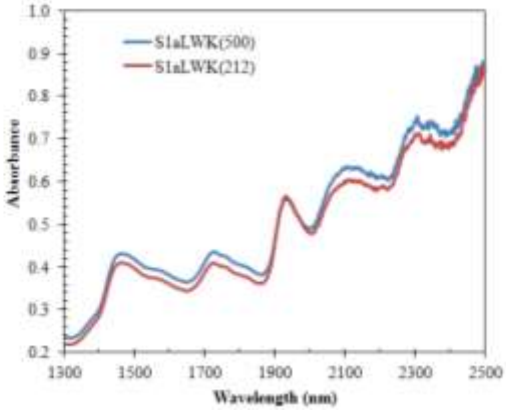

Figure 1. Typical spectra of civet coffee with different particle size.

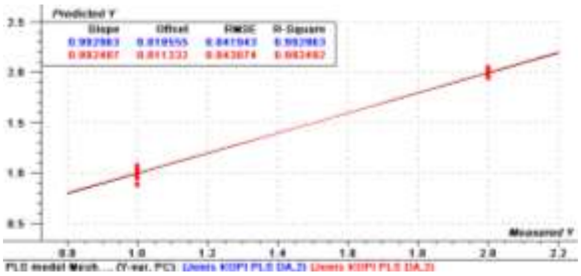

Figure 2. PLS-DA local model at 212 µm of particle size.

Figure 3. PLS-DA local model at 500 µm of particle size.

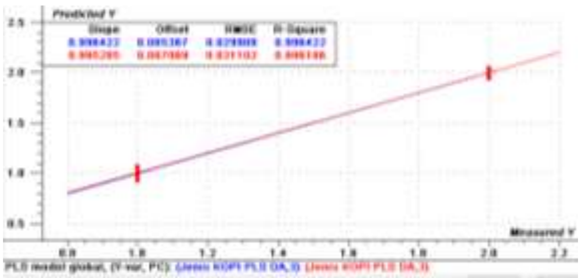

Figure 4. PLS-DA global model at combination of 212 and 500 µm of particle size.

3.2. The Result of Model Development (Local and Global Model Based on PLS-DA)

Figure 2 and 3 shows the local calibration model based on PLS-DA for 212 and 500 µm of particle size. Both calibration model resulted in a very good relationship between actual and predicted value of types of coffee (R^2=0.99 for 212 µm of particle size and R^2=0.99 for 500 µm of particle size, respectively). Figure 4 shows the global model of PLS-DA using combination of 212 and 500 µm of particle size. The calibration is also very good with R^2=0.99.

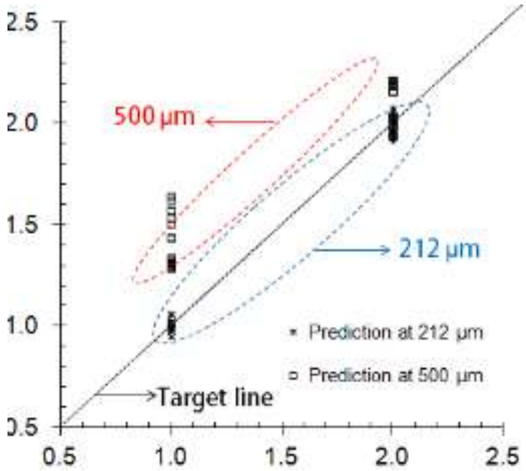

Figure 5. The result of prediction for civet coffee discrimination using local PLS-DA model at 212 µm of particle size.

3.3. Prediction Result Using Local Calibration Model 212 µm

Figure 5 shows the prediction result of civet coffee discrimination using local PLS-DA calibration model at 212 µm of particle size. It can be seen that the prediction was good only for 212 µm samples. When the local PLS-DA model at 212 µm was used to predict at different particle size (500 µm), high error of prediction was observed. This error was mainly due to an underestimation of the particle size among the samples. We guess that the prediction was worse when the particle size difference between the calibration and prediction samples increased.

3.4. Prediction Result Using Global Calibration Model at Combination of 212 and 500 µm

Figure 6 shows the prediction result of civet coffee discrimination using global PLS-DA calibration model at combination of 212 and 500 µm of particle size. It can be seen that the prediction was good for all samples at 212 and 500 µm. When the global PLS-DA model at 212 and 500 µm was used to predict at different particle size (212 and 500 µm), no longer shows biases or other errors of prediction was observed. Therefore, we can handle the influence of particle size completely by using global PLS-DA model.

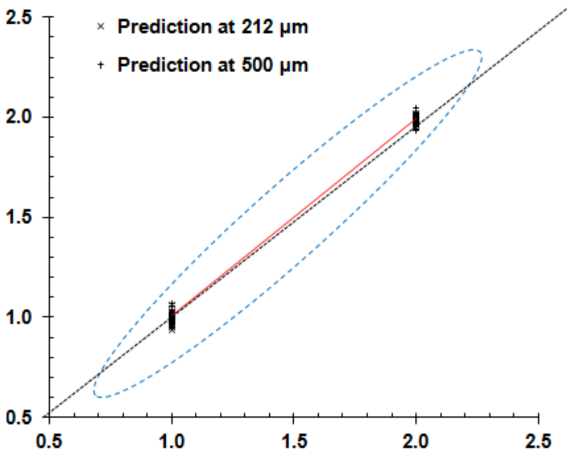

Figure 6. The result of prediction for civet coffee discrimination using global PLS-DA model at combination of 212 and 500 µm of particle size.

CONCLUSSIONS

In this study, we investigate the effect of different particle size of samples powder on model performance of PLS-DA in NIR region in two different particle sizes of 212 and 500 µm. The prediction results of using global calibration model is improved significantly resulted in low RMSEP and SEP. The global calibration model can handle completely the influence of particle size on the model prediction.

ACKNOWLEDGEMENT

We gratefully acknowledge support of the Indonesian Ministry of Research, Technology and Higher Education (KEMENRISTEKDIKTI) via Penelitian Strategis Nasional (STRANAS) 2016 (Nomor: 419/UN26/8/LPPM/2016). We thank to the Laboratory of Bio-Sensing Engineering, Kyoto University for their permission to use their facilities for NIR spectral data acquisition. All spectral data measurements were conducted at

Kyoto University during JASSO follow-up research program.

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