Combination of near infrared spectroscopy and chemometrics for authentication of taro flour from wheat and sago flour

Rachmawati¹, E Rohaeti* and M Rafi¹,2

¹Department of Chemistry, Faculty of Mathematics and Natural Sciences, Bogor Agricultural University, Kampus IPB Darmaga Indonesia
²Halal Science Centre, Bogor Agricultural University, Kampus IPB Baranangsiang Jalan Raya Pajajaran Bogor 16151, Indonesia

E-mail : etirohaeti@ipb.ac.id

Abstract. Taro flour on the market is usually sold at higher price than wheat and sago flour. This situation could be a cause for adulteration of taro flour from wheat and sago flour. For this reason, we will need an identification and authentication. Combination of near infrared (NIR) spectrum with multivariate analysis was used in this study to identify and authenticate taro flour from wheat and sago flour. The authentication model of taro flour was developed by using a mixture of 5%, 25%, and 50% of adulterated taro flour from wheat and sago flour. Before subjected to multivariate analysis, an initial preprocessing signal was used namely normalization and standard normal variate to the NIR spectrum. We used principal component analysis followed by discriminant analysis to make an identification and authentication model of taro flour. From the result obtained, about 90.48% of the taro flour mixed with wheat flour and 85% of taro flour mixed with sago flour were successfully classified into their groups. So the combination of NIR spectrum with chemometrics could be used for identification and authentication of taro flour from wheat and sago flour.

1. Introduction

Taro (Colocasia esculenta (L.) Schott) or locally known as talas in Indonesia is famous and widely used for food. Many types of food such as cake in traditional or modern form were derived from taro flour. In the market, taro flour is usually sold at higher prices compare to the other kind of flour such as wheat and sago flour. The price of taro flour is about Rp25 000/kg while for wheat and sago flour is about Rp10,000-15,000/kg. With this situation, taro flour could be adulterated with wheat and sago flour with a purpose to reduce the production costs and also because of the availability of wheat and sago flour more available than taro flour. The substitution and mixing with other similar material could decrease the quality of the end product of taro flour. To prevent this thing we must have an authentication method for quality control of taro flour to guarantee the authenticity of taro flour.

Authentication of food raw materials is a part in the quality control of final food product will require an adequate analytical method. In the previous studies, some researcher has been demonstrated the used of near infrared spectroscopy (NIR) combined with chemometrics in the development of authentication methods for quality control purposes. NIR technique could be utilized in the

* Corresponding author
development of authentication method because able to detect small differences in the chemical composition present in some material. Also, this technique is relatively fast, requires no sample preparation, didn’t use chemicals, non-destructive measurement and relatively cheaper [1,2]. NIR spectrum is a complex pattern, so direct and visual interpretation become not easy if we would like to use to differentiate a similar or mixed material to its constituent. Chemometrics could be used to facilitate the interpretation of data by using multivariate analysis i.e. principal component or discriminant analysis for the purposes [3,4]. This combination technique has been used for rapid estimation of the quality of taro [5], evaluation ability of NIR spectroscopy to authenticate green asparagus as a function of the growth [6] and roasting degree of coffee [7]. In this study, we have been developed an authentication method of taro flour from wheat and sago flour by NIR and chemometrics analysis.

2. Materials and Methods

2.1. Material
Samples of taro were collected from several regions, two from Bogor (Cibereum and Ciapus), one from Cianjur, Bandung, Kuningan, Cimahi, Sumedang and one commercial taro flour. Five wheat and four sago flour were purchased from local market in Bogor.

2.2. Sample preparation
Taro from several regions is prepared to powdered form according to the method described by Miranda et al. [8]. Firstly, we cleaned the taro from the soil and then washed with water. After the taro was drained, we peeled and washed again, and then sliced about 0.2 cm thickness. The sliced taro dried at 65°C for 5 hours in the oven. Dried sample then pulverized with a grinding machine and sieved to obtain a 100 mesh particle size.

2.3. NIR spectrum measurement
Into the petri dish, we added the sample until reach ¾ height of the cup. All of the samples (taro flour 100%, taro flour 95% + wheat flour 5%, taro flour 75% + wheat flour 25%, taro flour 50% + wheat flour 50% and wheat flour 100%) were weighed and the same composition also used for sago flour. NIR spectrum was acquired with NIRFlex Solids Petri N-500 spectrophotometer (Buchi, Flawil, Switzerland) with attenuated total reflection mode the near infrared region (10000–4000 cm⁻¹) with 32 scans/min and resolution of 4 cm⁻¹.

2.4. Chemometrics analysis
Preprocessing of spectral data is an important step before subjecting the NIR spectra data for multivariate analysis. We used normalization and standard normal variate (SNV) for signal processing of the NIR spectra. Principal component analysis (PCA) and discriminant analysis (DA) were used to build an authentication model using XLSTAT software version 2012 (Addinsoft, New York, USA).

3. Result and Discussions

3.1. NIR Spectrum
NIR spectrum of all taro, wheat and sago flour samples used in this work give same profile and only differ in the absorbance value under the same measurement conditions (Figure 1). This result is indicating that the chemical compounds present in the samples are same and only differ in the concentration. From the NIR spectrum of taro flour, we found eight characteristic peaks. Peak appear in the 1450 and 1540 nm correspond to stretching vibration of O─H in the first overtone. The peak at 1930 nm denotes stretching vibration of O─H or a combination of deformation H-O-H, while the peak at 1960 nm assigned as a vibration of O─H or a combination of bending vibration. A peak at 2100 nm indicates bending vibration of O─H or a combination of stretching vibration of C─O. A peak at 2280
and 2322-2330 nm defined as stretching vibration of C─H or deformation of CH2, while for peak appear in 2500 nm denotes stretching vibration of C─H or C─C or C─O [1].

![NIR spectra graph](image)

**Figure 1.** Representation of NIR spectra of taro flour from Kuningan (▬), Bogor Ciapus (▬) and Sumedang (▬)

Based on the results of taro flour NIR spectra, we selected three samples from Bogor-Ciapus, Sumedang, and Kuningan that show high, medium and low absorption to build an authentication method of taro flour. The three samples were mixed with wheat and sago flour as counterfeit material to taro flour are. Figure 2 showed the NIR spectra of the mixture samples (5% wheat and sago flour added to taro flour) give same pattern and only differ in the intensity.

![NIR spectra graph](image)

**Figure 2.** Representation of NIR spectra of taro flour (▬), wheat flour (5%) in the taro flour (▬), sago flour (5%) in the taro flour (▬)

### 3.2. Identification and authentication taro flour from wheat and sago flour

Distinguishing the three flour samples is crucial to identify and authenticate the taro flour. The combination of NIR spectra and chemometrics could be used as the potential choice for such purposes. Some preprocessing signal on the NIR spectra of the sample should be conducted to obtain a good
predictive authentication model of the samples used. Preprocessing signal can avoid some problems due to baseline drift and reduce random noise in the initial spectrum. This process would lead to the distinctive character of the spectrum becomes more quantized, so the identifier factors become increasingly precise.

3.2.1. Principal Component Analysis. PCA is one multivariate analysis widely used for a purpose to reduce data and extract information to find a combination of variables describing major trends in a data set. Each new variable knows as a principal component (PC) that is generated as a linear combination of the original variables measurement. In PCA we will have first principal components (PC1) which have the greatest variance and second principal component (PC2) which has the next largest variance in the data set [4]. Figure 3 showed the PCA plot by using the first two PCs with explaining 96.62% of the total variance (PC-1 = 87.83%, PC-2 = 8.79%). Based on the PCA plot, we found that the three flour samples were separated according to the type of flour. So, PCA was able to distinguish the taro, wheat and sago flour.

![Figure 3. PCA plot of samples: taro flour (♦), wheat flour (▲), and sago flour (■)](image)

We also used PCA to build an authentication model of taro flour when mixed with wheat or sago flour. PCA generates a first two PCs explain 95.86% of the total variance (PC-1 = 86.28%, PC-2 = 9.58%) for the sample mixed with wheat flour (5, 25 and 50%) and 95.58% of the total variance (PC-1 = 87.57%, PC-2 = 8.01%) for the sample is mixed with sago flour (5, 25 and 50%) (Figure 4 and 5). Based on the result obtained, separation of the pure taro flour and mixed with wheat or sago flour was achieved but didn’t give clear separation. To have better separation, we used another multivariate analysis namely discriminant analysis.

3.2.2. Discriminant Analysis. DA is one of the supervised pattern recognition techniques and work to find a discriminant factor (DF) for each group by finding a linear combination of data provide separation of two or more groups of observations [9]. In this work, we used the first two PC obtained from PCA to build a predictive model for authentication of taro flour. The use of the first two PC because the PCA plot of this two PCs could distinguish well the three samples. From the DA we obtained two DF with total variance greater than 99% (DF-1 = 98.79% and DF-2 = 1.21%). Separation of the three samples by DA is much better because the distance is much longer than the PCA. Evaluation of the predictive ability of the model to distinguish the three sample by cross-validation also gives a satisfactory result, about 100% of the samples are identified according to its type.
Figure 4. PCA plot of samples: taro flour (♦), wheat flour (▲), 5% (●), 25% (∗), 50% (+) wheat flour in taro flour

Figure 5. PCA plot of samples: taro flour (♦), sago flour (■), 5% (●), 25% (∗), 50% (+) sago flour in taro flour

Figure 6. DA plot of samples: taro flour (♦), wheat flour (▲), sago flour (■)

The first two PC is also used to create an authentication model using DA for mixed taro flour with wheat and sago flour as the counterfeiters. In the plot of DA from taro flour mixed with wheat flour and sago flour we found the total variance of the two DF were 100%. From the DA plot (Figure 7 and 8) the separation of pure taro flour with the mixed taro with wheat and sago flour (5, 25 and 50%) is much clearer compare with PCA. The evaluation of the predictive ability of the model by cross-validation give an adequate accuracy with 90.48% and 85% of the samples are mixed with wheat and sago flour respectively were successfully classified into their group. Based on the results of DA, we could authenticate the taro flour by using the NIR spectra follow with DA.
4. Conclusions
The combination of NIR spectra and chemometrics could be used for authentication of taro flour from wheat and sago flour. PCA and DA could be utilized for the authentication of taro flour from wheat and sago flour. DA give clearer separation of pure taro flour (100%) and taro flour mixed with 5, 25 and 50% of wheat and sago flour.

References
[1] Burns D and Ciurczak E 2008 Handbook of Near-Infrared Analysis Third Edition (New York: CRC Press)
[2] Reynertson AK and Mahmood K 2015 Botanicals Methods and Techniques for Quality and Authenticity (New Jersey: CRC Press)
[3] Njintang NY, Mbofung CMF and Kesteloot R 2007 J. Food Eng. 81 250-6
[4] Miller JN and Miller JC 2010 Statistics and Chemometrics for Analytical Chemistry 6th Edition (Harlow: Pearson Education)
[5] Lebot V, Malapa R, and Bourrieau M 2011 J. Agric. Food Chem. 59 9327-9334
[6] Sanchez MT, Varo AG, Guerrero JE, and Marin DP 2013 Postharvest Biol. Technol. 85 116-23.
[7] Bertone E, Venturello A, Giraudo A, Pellegrino G, and Geobaldo F 2016. Food Control 59 683-9.
[8] Miranda J R, Lopez I R, Lara E H, Sanchez M, Licon E D and Vera V 2011 LWT-Food Sci Tech. 44 673-80
[9] Gad H A, El-Ahmady S H, Abou-Shoer M I and Al-Azizi M M 2012 Phytochem. Anal. 24 1-24