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Near infrared reflectance spectroscopy: classification and rapid prediction of patchouli oil content

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Abstract. Patchouli is a fragrant oil that produces essential oils known as patchouli oil. The main content of patchouli oil is Patchouli alcohol (PA), generally to determine the PA content of patchouli oil uses chemicals and the waste is not good for the environment. Therefore, there is another alternative method to address these problem, one of the alternative technologies to determine the content of patchouli oil is to use Near Infrared Spectroscopy (NIRS). NIRS can be used to analyze samples rapidly, easily, non-destructive and does not require any chemicals. In this study, the PA content and patchouli oil refractive index were obtained through the acquisition of NIRS spectrum in patchouli oil samples from several different regions with different fractions. Patchouli oil classification is carried out by the Principal Component Analysis (PCA) method. The content of PA content and refractive index is carried out by verifying the factual results of laboratory tests using the Principal Component Regression (PCR) method. Patchouli alcohol spectrum data is found in the wavelength range of 1350-1450 nm and 1950-2100 nm. Predicted results with PCR for PA content and refractive index show values $R^2 = 0.95$ and 0.99 respectively with RPD values of 5.07 and 0.0011.

1. Introduction

The Patchouli plant (*Pogotemon cablin* Benth) is a fragrant herb with fine leaves and rectangular trunk. The dried leaves of this plant are distilled to obtain oil (patchouli oil) which is widely used in various industrial activities. The main components contained by patchouli oil are patchouli alcohol (PA, $C_{15}H_{26}O$) which serves as a binding raw material (fixative) and as controller ingredient (ether) for perfume so that its fragrance lasts longer [1].

Aceh is one of the regions producing patchouli, in addition to North Sumatra (Tapanuli region), Riau, Pekanbaru and Several regions in Sulawesi. According to ISO 3757:2002, Patchouli oil quality is controlled with patchouli alcohol (PA) level indicator at the standard limit of 27%-35%. Based on Agricultural R&D data [2], patchouli of Aceh best meets these requirements with a PA content of >30%. So far, the patchouli alcohol (PA) analysis process of patchouli oil used with GC-MS model, Samples injected into Gas Chromatography will be converted into steam phase and flowed through capillary column with the help of carrier gas. The separation of mixed compounds into single compounds occurs



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based on differences in chemical properties and the time required is specific to each compound. Detection takes place in Mass Spectroscopy with the mechanism of firing compounds by electrons into ionized molecules and recording fragmentation patterns formed compared to standard compound fragmentation patterns indicated by the percentage of Similarity Index (SI) [3]. This analysis process costs a lot and takes a long time. The availability of working tools and instruments and chemicals such as solvents is also limited.

Therefore, another alternative method is needed to obtain a better ester product, one of the alternative technologies to Rapid and efficient quality prediction can be realized through the development of Near Infrared Reflectance Spectroscopy (NIRS) technology. NIRS has become one of the most promising non-destructive methods and can be used for various material analysis such as organic and metal. The advantages that can be achieved are simple samples preparation, rapid prediction process, non-destructively, and environmental friendly because no require any chemical. In addition, NIRS also does not cause pollution, such as the use of preparations and the like. Cen and He [4] say that through the development of computer science and chemometric, NIRS engineering application capabilities become more popar and attract a lot of attention from researchers in the field of food. Components with a concentration percentage of 0.1% can be detected and evaluated using NIRS.

The record of NIRS region of the electromagnetic spectrum involves the response of the molecular bonds O–H, C–H, C=O and N–H. These bonds are subject to vibrational energy changes when irradiated by NIR frequencies, and two vibration patterns exist in these bonds including stretch vibration and bent vibration. The energy absorption of organic molecules in NIRS region occurs when molecules vibrate or is translated into an absorption spectrum within the NIRS spectrometer [4]. Figure 1 shows the distribution of overtone and overtone combination of organic bonds in electromagnetic wave region.

14286	12500	1111	10000	9090	8333	7692	7143	6666	6250	5882	5556	5263	5000	4762	4545	4348	4168	4000	cm ⁻¹
700	800	900	1000	1100	1200	1300	1400	1500	1600	1700	1800	1900	2000	2100	2200	2300	2400	2500	nm
																			Bonds
4		3					2					C							
..								H ₂ O
..							ROH
					NH ₂
													CONH ₂
													CONHR
5	4		3				2		2					C					
..		ArCH
...					CH ₃
...					CH ₂
...					CH
														Grease	Acid				CH ₂
									Others
							Acid			SH	Acid	C=O		CHO	Fat				

Figure 1. The distribution of overtone and overtone combination of main organic bonds in electromagnetic wave. “¼”: absorption position of bonds, “C”: tone combination, “2C”: overtone combination, “ArCH”: bond CH on the aromatic groups, “2, 3, 4, 5”: first, second, third and fourth overtone.

Thus, it should be developed a method of measuring the quality of patchouli oil quickly and accurately using NIRS. The purpose of this study is to examine in detail the capabilities of NIRS technology as a new method to predict quality of patchouli oil which includes Patchouli alcohol (PA) content and refractive index.

2. Materials and methods

2.1. Materials

The patchouli oil used comes from the areas of Aceh Selatan, Aceh Barat and Aceh Jaya. Patchouli oil is obtained from distillation result with three different fractions, namely without fractions, light fractions and heavy fractions. The equipment used is NIRSFlex N-500 Spectrometer (fiber optic solids) with wavelength range between 1000-2500 nm, a set of optical tools equipped with infrared wave transmitter and detector at wavelength 1506 nm.

2.2 Methods

2.2.1 Infrared spectrum acquisition of patchouli oil. The infrared spectrum (near-infrared) for patchouli oil samples is recognized using infrared spectroscopy instrument (FT-NIR Thermo Nicolet Antaris TM II, with workflow configuration built using Integrated Thermo Operation® software. In this present study, the instrument is set with 32 scans, so that it can acquire the diffuse reflectance spectrum of the sample [4-6]. Then the tool averaged the results and saved the scan results in two different file formats. i.e. *.SPA dan *.CSV. Sampling patchouli oil is done by placing a sample (25ml) inside the bottle, placed near the sensor for the scanning process.

2.2.2 Actual patchouli alcohol and refractive index. Measurement. Patchouli oil after spectra collection was complete, all patchouli oil samples were taken immediately to measure patchouli alcohol and refractive index in Laboratory following Standar Indonesian National (SNI) NO. 06-2385-2006 [5] which includes refractive index and patchouli alcohol content (PA, $C_{15}H_{26}O$).

2.2.3 Patchouli oil quality prediction model. Patchouli alcohol content and the refractive index of patchouli oil are predicted based on the NIRS spectrum generated through the model calibration process. The prediction model was constructed by regressing between the NIRS spectrum (variable X) with patchouli alcohol content and the refractive index (variable Y) of the measurement results in the laboratory. The regression methods used are the Principal Component Analysis (PCA) and Principal Component Regression (PCR) [7, 8].

3. Results and discussion

3.1 Raw spectrum

When a beam derived from a light source falls on a biological object, there will be an interaction between the object and the light where it will respond in the form of reflection (*reflectance*), absorption (*absorbance*) and canal (*transmittance*) [8-10]. Figure 2 shows typical diffuse reflectance spectra of NIRS for each patchouli oil sample. Based on Figure 2 it can be seen that the typical spectrum look the same where each peak and valley on the spectrum indicates the presence of certain substances. The main content of patchouli oil is Patchouli alcohol ($C_{15}H_{26}O$), it's mean the chemical bonds of patchouli oil are composed of elements C, H and O. This substances experiences vibration in the range of near infrared wavelengths in the form of *overtone*, *bending*, and *stretching*. So, it is possible for the NIRS method to predict the patchouli oil quality rapidly and simultaneously.

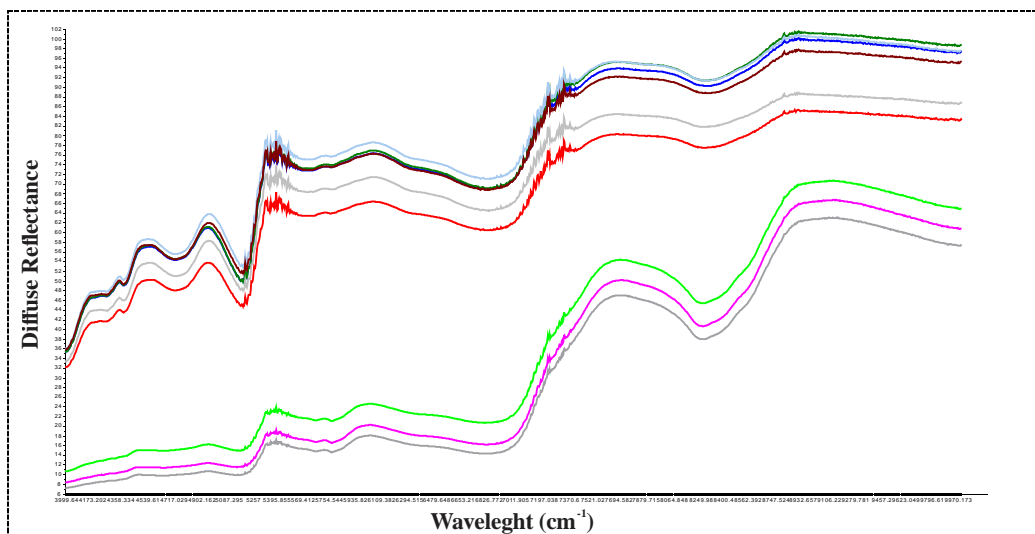


Figure 2. Fatures a diffuse reflectance spectrum of patchouli oil in the infrared wavelength range.

Based on Figure 3 it shows that two peaks and valleys were found. Both peaks show the presence of R-OH molecules identified as patchouli alcohol (PA). The first peak is in the wavelength range of 4763-5125 cm^{-1} (1350-1450 nm) and the second peak for the wavelength range of 6892-7405 cm^{-1} (1950-2100 nm), it is corresponds to Ce and He research results in the arrangement of distribution tables and the combination of organic bonds in electromagnetic wavelengths [4, 11].

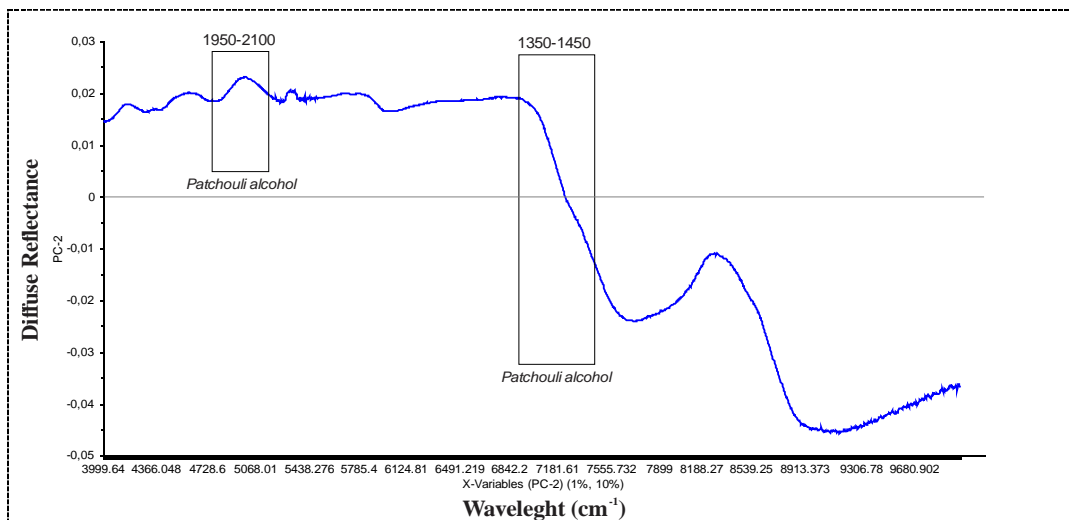
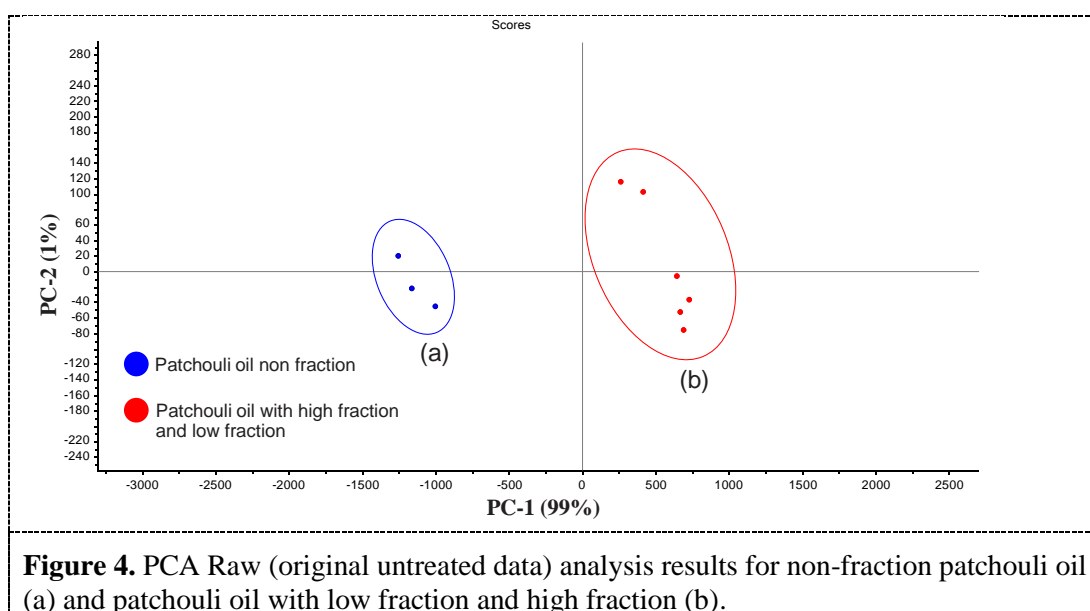


Figure 3. NIRS spectrum for patchouli oil.

3.2 Patchouli oil classification using PCA

Raw data classification (original untreated data) generate data result that can be analyzed with total cumulative explained variance using latent variable 1 and latent variable 2 which is 100%, meaning samples data from patchouli oil can be classified well with a 100% success percentage. PCA analysis of raw data shows that there was a difference latent variable position between patchouli oil without fraction and fractionated as shown in Figure 4.



The location of variables that are separated between patchouli oil without fraction and patchouli oil that has gone through the fractionation process shows that NIRS method is able to classify data well.

3.3 Patchouli alcohol and refractive index prediction

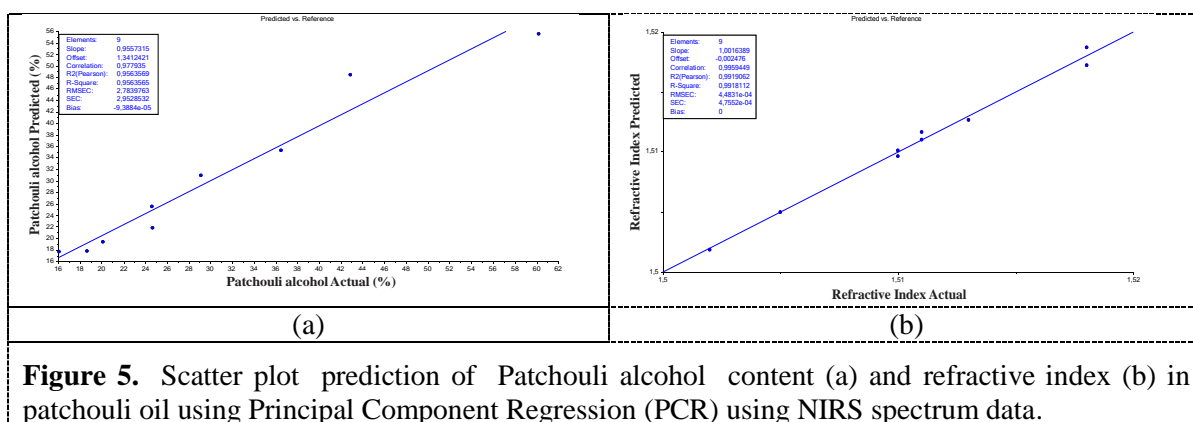
Patchouli alcohol and refractive index prediction models were established using Principal Component Regression (PCR). Then the spectrum data results are compared to factual tests using the GC-MS test. The results analysis of GC-MS method and NIRS prediction for patchouli alcohol and refractive index of patchouli oil are shown in Table 1.

Table 1. GC-MS test results and PCR method prediction of each patchouli oil sample.

Sample	Y Reference (GC-MS)	Y Predicted (PCR)	Y Reference (GC-MS)	Y Predicted (PCR)
	<i>Patchouli alcohol</i>	<i>Patchouli alcohol</i>	Refractive index	Refractive index
MH1	42.889	48.482	1.518	1.517
MH2	36.509	35.362	1.505	1.505
MH3	60.159	55.625	1.518	1.518
ML1	18.620	17.845	1.510	1.510
ML2	16.040	17.716	1.510	1.509
ML3	20.090	19.347	1.511	1.511
MN1	24.600	25.544	1.513	1.512
MN2	29.120	30.981	1.502	1.501
MN3	24.670	21.791	1.511	1.511
Description:	M : Patchouli Oil 1 : Aceh Selatan H : High Fraction 2 : Aceh Barat L : Low Fraction 3 : Aceh Jaya N : None Fraction			

Patchouli alcohol content in patchouli oil can be predicted well using Principal Component Regression (PCR). The predicted results indicate that the regression coefficient (R^2) produced for the prediction of Patchouli alcohol content is 0.95 (Figure 5a) with a Standard Deviation of 14.13. PCR Raw (original untreated data) regression method with regression coefficient of 0.95 produces Residual

Predictive deviation (RPD) index, RPD = 5.07. Based on literature, it is classified in excellent model performance [7].



Principal Component Regression (PCR) result for the refractive index shows a regression coefficient value (R^2) of 0.99 (Fig. 5b) with an RPD index of 0.0011. A small standard deviation value in the patchouli oil sample of refractive index causes the RPD value to be small. The limitation of detecting components that have very small values is one of the drawbacks of NIRS spectroscopy method [8], therefore the use of large and varied samples can improve for the better performance of NIRS.

4. Conclusion

Patchouli oil from South Aceh, West Aceh and Aceh Jaya has the same typical spectrum which means every spectra has the same content. Patchouli alcohol content was found in the wavelength range of $4763\text{--}5125\text{ cm}^{-1}$ (1350–1450 nm) and $6892\text{--}7405\text{ cm}^{-1}$ (1950–2100 nm). PCA is able to classify patchouli oil from each different location and fraction with a 100% success rate with a wavelength range of 999–2500 nm. Generally NIRS technology can be used to classify patchouli alcohol and refractive index of patchouli oil. Principal Component Regression (PCR) Analysis shows the R^2 values for Patchouli alcohol and refractive index are 0.95 and 0.99 respectively with RPD index of 5.07 and 0.0011, respectively. Based on this obtained result, it may conclude that NIRS technology can be an alternative method to predict the content of Patchouli alcohol and the refractive index in particular and patchouli oil quality in general.

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