

# Determination of carotene content of crude palm oil using Raman spectroscopy

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**Abstract.** As one of the world's largest palm oil producers, Indonesia regulates its production through Indonesian National Standards (SNI) to ensure the quality of palm oil available on the market. The main parameters in the SNI standard include color, odor, free fatty acids (FFA), iodine value, water content, and DOBI (Deterioration of Bleachability Index). Beta-carotene, a type of carotenoid, functions as a provitamin A that can be converted into active vitamin A after metabolism in the body. The beta-carotene content in palm oil affects its color and clarity. This research analyzed crude palm oil using two heating stages. Two methods were used to determine the carotene content: the conventional method and Raman spectroscopy combined with Partial Least Squares (PLS) as a modern alternative. Raman spectroscopy uses a light source with a wavelength of 785 nm directed at the sample. The results show that the data obtained from Raman spectroscopy closely matches the data from the conventional method, indicating that Raman spectroscopy can be used as a supporting tool to determine oil quality based on its carotene content.

## 1 Introduction

Indonesia is one of the world's largest producers of crude palm oil. According to data from the Central Statistics Agency (BPS), Indonesia produces 59% of the world's total palm oil production, or 45.5 million tons per year [1]. In accordance with Indonesian National Standard (SNI) 2901:2021 [2], crude palm oil distributed in Indonesia must meet several requirements aimed at protecting consumers, including Free Fatty Acid (FFA) content, water content, Iodine Value, Deodorization and Bleaching Index (DOBI), odor, and color. Although SNI specifies a reddish-orange color as the quality criterion for CPO, this assessment remains subjective because it can only be evaluated visually. Considering that carotenoid pigments, especially  $\beta$ -carotene, are the main factors responsible for this coloring, determining the carotene content offers a more objective and quantitative alternative for evaluating color quality.

Carotene is a type of natural pigment, specifically a carotenoid that gives certain fruits and vegetables their orange, red, or yellow color [3]. It also acts as an antioxidant and a precursor to vitamin A, meaning your body can convert it into vitamin A, which is essential for eye health, immune system function, and skin health [4]. Crude palm oil (CPO) is rich in

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carotene, especially beta-carotene, which gives the oil its reddish-orange color. This is one of the main indicators of quality in crude palm oil [5].

UV-Vis (Ultraviolet-Visible) spectroscopy is a conventional method commonly used to identify and measure carotene content in palm oil. However, this method has several weaknesses, such as the use of hazardous chemicals (hexane is highly flammable) and the long time required for data collection. Raman spectroscopy has several advantages that make it worth considering for palm oil quality testing. This technique can provide detailed information about the molecular structure and chemical bonds in palm oil samples without requiring complicated sample preparation. The purpose of this research is to test the use of conventional and Raman spectroscopy methods in determining the quality of palm oil circulating in Indonesia based on its carotene content.

Moreover, the data obtained from Raman spectroscopy can be further analyzed using statistical methods to obtain additional information. Partial Least Squares (PLS) is a versatile technique for modeling complex, high-dimensional data. PLS can model the relationship between the chemical composition of palm oil and its quality characteristics, such as carotene content [6]. Since CPO usually undergoes additional processes, such as processing to produce other products, PLS can analyze the impact of these processes on palm oil quality and even suggest optimal conditions for obtaining the best quality. Therefore, PLS is a great method to evaluate palm oil due to its ability to handle multicollinearity, reduce dimensionality, and possess the power to predict [7].

## 2 Materials and Methods

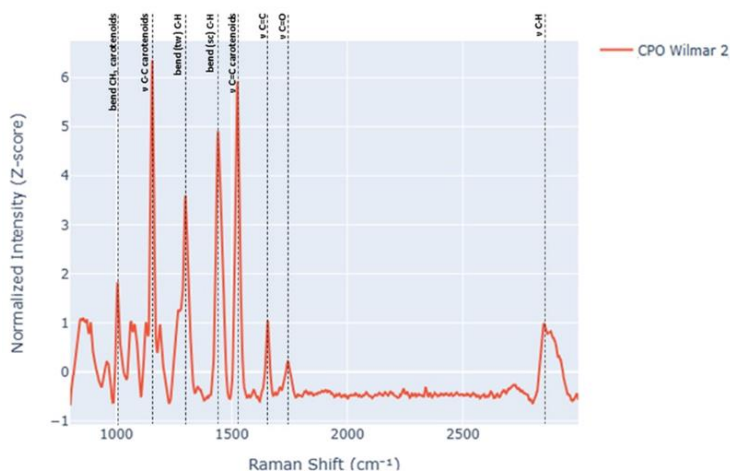
In this research, we analyzed 15 types of Crude Palm Oil (CPO) from four different companies: Cikasungka, Musim Mas, Kertajaya, and Wilmar, with each sample consisting of 10 Raman spectra due to sample limitations in evaluating quality based on carotene content. Each sample was heated using an oven at a temperature of 200°C, producing three types of data: samples before heating, samples from the first heating stage (200°C for 1 hour), and samples from the second heating stage (200°C for 2 hours). This method is based on research by Sampaio et al., which showed that carotenoids in palm oil undergo significant degradation during heating at temperatures of 170–230 °C with heating times of up to ±140 minutes [8]. The carotene content in each sample was obtained based on ISO 17932:2011 [9]. Test samples were weighed with a precision of 0.1 g according to standard procedures and placed in small tubes. Next, 8 g of isoctane solvent was added, and the mixture was stirred thoroughly to ensure homogeneity. After thorough mixing, the absorbance of the test sample was measured in the range of 400 to 500 nm using an Evolution 220 UV-Visible Spectrophotometer (Thermo Scientific). The carotene content can be calculated using equation 1, where  $A$  is the absorbance value at 446 nm and  $W$  is the sample weight in grams.

$$\text{Carotene content (ppm)} = \frac{383 \times A \times 25}{W \times 100} \quad (1)$$

MacroRAM Raman Spectroscopy (Horiba) with an excitation wavelength of 785 nm and power beam of 90 mW was used in this research to record the intensity in the Raman shift range from 800 to 3500  $\text{cm}^{-1}$ . Then, the spectra were pre-processed by doing baseline, smoothing, and cropping. Next, we used Partial Least Square (PLS) to analyze oil quality based on carotene content. For PLS, we defined variables as predictors and responses, then created a PLS model using Raman spectrum data and a latent variable number of 3. We calculated the intensity in the Raman shift range from 800 to 3000  $\text{cm}^{-1}$  for the PLS model of carotene content. Here, we evaluate the model fit using R-squared and RMSE values and visualize the results in graphs.

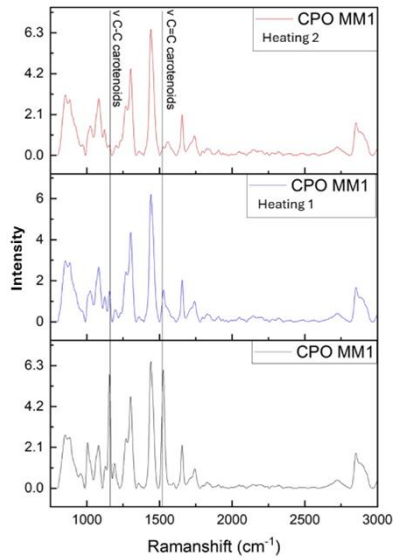
### 3 Results and Discussions

Due to the highly sensitive nature of Raman spectroscopy to any changes in molecular symmetry, particularly for symmetric vibrations and non-polar bonds, we observed the Raman spectra of Crude Palm Oil (CPO) as shown in Figure 1. Based on the results, we found that the Raman spectra show peaks such as the CH<sub>2</sub> bending (scissoring) in the fatty acid chains at 1442 cm<sup>-1</sup> or the bending vibration of C-H (twisting) typically in the saturated fatty acids at around 1310 cm<sup>-1</sup>. It also presents the existence of the C=C (alkene) bond in unsaturated fatty acids at around 1657 cm<sup>-1</sup> and the stretching of the carbonyl group (C=O). Furthermore, we noticed the notable peaks at around 1150-1200 cm<sup>-1</sup> and at 1500 cm<sup>-1</sup>, associating with the stretching vibrations of the conjugated double bonds in the polyene chain in the carotenoid. Carotenoids are pigments in plants that give vibrant colors thus giving the reddish color of CPO. Compared to other palm oil, CPO has a broader range of fatty acids. Thus, CPO contains abundant amounts of palmitic acid (C16:0), oleic acid (C18:1), and linoleic acid (C18:2).



**Fig. 1.** Raman spectrum of one sample of Crude Palm Oil (CPO).

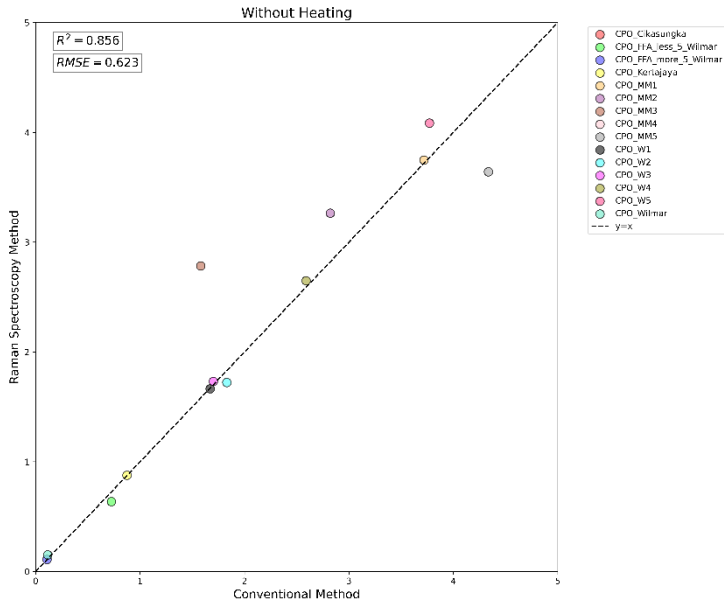
As part of refining process, CPO is often heated to break down certain compounds such as free fatty acids and make it easier to process. Here, we investigated the effect of heating CPO by comparing the Raman spectra before and after heating as shown in Figure 2. The obvious change is the absence of peaks associated with carotenoids in Raman spectra of heated sample, causing the CPO visually changes to less reddish. The peaks associated with carotenoids appear in the range of 1150–1200 cm<sup>-1</sup> and around 1500 cm<sup>-1</sup>, which gradually diminish as the heating process progresses.



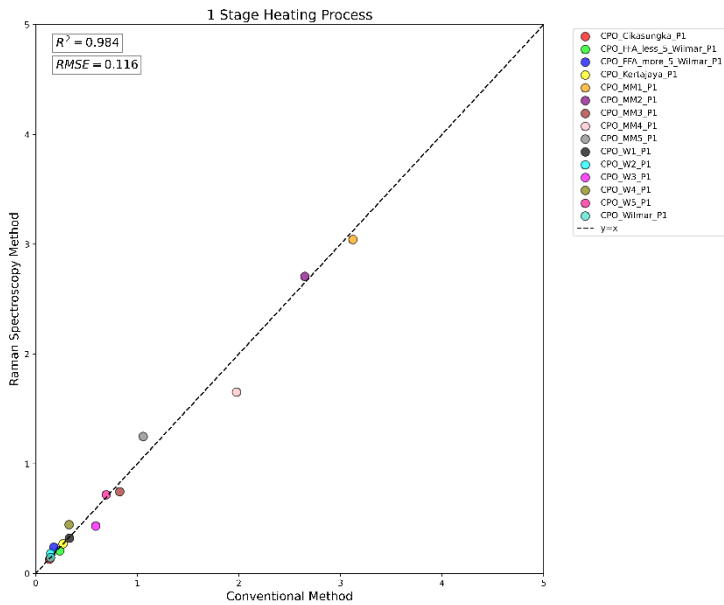
**Fig. 2.** Raman spectrum of one sample of Crude Palm Oil (CPO) before and after the heating process.

Indeed, Raman is a powerful tool for analyzing oil quality. However, relying only on the Raman spectrum might make it difficult to determine the quality of unknown oils. Here, we propose using Partial Least Square (PLS) to analyze the condition of unknown oils. PLS can model the relationship between oil quality attributes and Raman spectra. By using PLS, we can predict the concentration of these parameters by learning several predictors such as spectra. PLS reduces the dimensions of spectral data but retains the main information from the data.

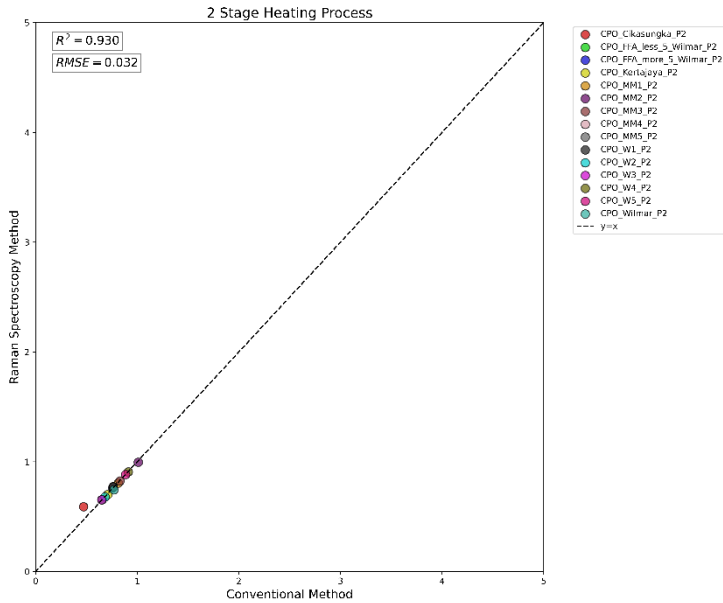
To predict carotene content, the complete spectral range from 800 to 3000  $\text{cm}^{-1}$  and a latent variable number of 3 were used, giving results for samples before heating, samples from heating stage 1, and samples from heating stage 2, as shown in Figure 3-5. The diagonal line ( $y = x$ ) represents perfect agreement between Raman predictions and conventional methods. The proximity of data points to the  $y = x$  line demonstrates the reliability of the PLS model in predicting carotene content within the expected quality range. Figure 3 shows the PLS regression results for evaluating CPO quality before the heating process based on carotene content, with an  $R^2$  value of 0.856 and an RMSE of 0.623, indicating that the Raman spectroscopy model explains 85.6% of the variance and the average prediction error of the model is within the range of 0.623 units compared to the actual value. Subsequently, Figure 4 shows the PLS regression results for CPO after the first heating stage, with an  $R^2$  value of 0.984 and an RMSE of 0.116, indicating that the Raman spectroscopy model explains 98.4% of the variance and the average prediction error of the model is within the range of 0.116 units compared to the actual value. Meanwhile, Figure 5 presents the PLS regression results for CPO after the second heating stage, with an  $R^2$  value of 0.93 and an RMSE of 0.032, indicating that the Raman spectroscopy model explains 93% of the variance and the average prediction error of the model is within the range of 0.032 units compared to the actual value. The  $R^2$  and RMSE values obtained indicate that the PLS model is capable of explaining the variation in carotene content and producing relatively low prediction errors across the entire dataset analyzed.



**Fig. 3.** PLS regression results to evaluate CPO quality before the heating process based on carotene content.



**Fig. 4.** PLS regression results for evaluate CPO quality from heating process stage 1 based on carotene content.



**Fig. 5.** PLS regression results for evaluate CPO quality from heating process stage 2 based on carotene content.

## 4 Conclusion

Based on our research, we found that Raman spectroscopy can be used as a supporting or screening tool to determine the quality of palm oil circulating in Indonesia based on its carotene content in accordance with Indonesian National Standards (SNI). In addition, Raman spectroscopy can be used to identify differences between palm oil and its derivatives. Furthermore, the results obtained through the Raman spectroscopy method in carotenoid content can be categorized as fairly close to the results of conventional methods, as indicated by the R-squared and RMSE values obtained.

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