

# Feasibility study of detecting palm oil adulteration with recycled cooking oil using handheld Near-infrared spectrometer

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**Abstract.** Adulterated foods are causing health-related issues since there is no rapid method available for the detection of food fraud. The main objective of this study is to determine the feasibility of near-infrared spectroscopy in differentiating pure palm oil from palm oil adulterated with recycled cooking oil in varying concentrations. The deep-frying process was carried out for chicken nuggets and French fries as fried items using palm oil as a frying medium to produce used frying oil. The refining and bleaching process was applied to used frying oil to generate recycled cooking oil. Adulterated samples were developed with different concentrations of recycled cooking oil in pure palm oil (1, 2, 3, 5, 10, 15, 20, 30, 40, and 50%). Total polar compounds increased with the number of frying cycles and the duration of frying time. The increase in acid values and anisidine values occurred with the increase in the concentration of adulteration (1% to 50%). The chemical properties of pure palm oil such as acid value, anisidine value, and peroxide value were found to be  $0.765 \pm 0.03$ ,  $2.175 \pm 0.39$  and  $1.33 \pm 0.11$ , respectively. The spectrum of pure palm oil in the near-infrared region showed the main absorption peaks of C-H second overtone and O-H first overtone at the wavelength of 1200 nm and 1450 nm, respectively. The principal component analysis was applied to analyze the distribution of pure and adulterated palm oil samples. The classification accuracy of NIR (near infrared) spectra data in distinguishing adulterated samples of all concentrations was found to be low (below 0.5). Adulterated palm oil samples of RCO concentration (15% and above) were found to show classification accuracy of 1 when detected through handheld NIR spectrometer. The SWOT analysis of palm oil was performed to explore the potential of the palm oil industry in Malaysia in order to consider the need for the NIR-based detection approach.

## 1. Introduction

Adulteration in food commodities is a frequently faced problem in the current food business. Nearly all food commodities are subjected to quality problems, especially those having high commercial values [1]. Food fraud may relate to compositional adulterations, i.e. removal, addition or substitution of certain constituents. Common examples include nitrogen replacement in milk powders using melamine to claim high protein content, cheap oil substitution in olive oil, etc. [2]. The substitution of expensive materials with cheap ingredients can be very profitable for a producer or trader of raw ingredients since vending the product as if it remained pure permits profit values to be highly amplified [3].

Edible oils are defined as a food commodity of any kind of origin, source or structure that is manufactured for human intake entirely or in portion from a fat or oil other than dairy products [4]. To prepare delicious food, edible oils are often used due to its texture and exceptional flavour attributes. For many years, people utilized oil to make the flavour of the food better [5]. It is one of the indispensable nutrition components in our daily diet [6]. Palm oil is extracted from the rose-colored pulp of the African palm (*Elaeis guineensis*) [7]. Oil palm is considered as the supreme profitable source of vegetable oil production globally, and it is planted and harvested in several countries, which includes Indonesia, Malaysia and other western nations [8].

Edible oil adulteration can be in different forms, such as the substitution of cheap oil to high price oils, for instance mixing of recycled oils to pure olive oil, or the substitution of high-value olive oils with low ones [9]. It has been reported that adulteration of edible oils resulted in severe health issues in some cases like Spanish toxic oil syndrome or Spanish olive oil disease due to trading of waste rapeseed oil as edible rapeseed oil and even as olive oil [10]. Further health problems may also arise if an adulterant used belongs to a specific type of allergens such as peanut oil, as certain buyers may be sensitive to the peanut proteins in the adulterant [11]. Thus, the detection of food adulteration should be considered an important step because of its negative impact on the health conditions of the public [12].

An edible oil categorization is purely a substantial approach, which has been usually carried out by various chemical techniques to determine peroxide value, acidity, iodine value, etc., or combined with chromatographic methods to identify the percentage of different minor components, acyl groups, etc. [13]. A lot of instrumental recognition techniques have been established and applied to evaluate the fraud in oils and fats on the basis of chemical constituents or physical properties of fats and oils [14]. Recently, the Near-Infrared (NIR) spectroscopy combined with the chemometrics analysis (multivariate analysis) has gained great significance in the food business as a rapid instrument of analysis, having accurate and low-cost measurements [11, 15].

People are facing health issues while consuming adulterated foods because they cannot differentiate between real and adulterated foods with their naked eyes [16]. Conventional methods for detecting adulteration require sample preparation which makes them time-consuming, high materials and operating costs and are labor-intensive [17, 18, 19]. Thus far, there is no study conducted on the detection of adulteration in palm oil with recycled cooking oil using Near-Infrared (NIR) spectroscopy. Therefore, the utmost objective of the present research study is to distinguish between pure palm oil and adulterated palm oil using Near-Infrared (NIR) spectroscopy. Also, chemical tests will be conducted to observe the changes in pure palm oil and adulterated palm oil samples. Hence, this study aims to develop NIR spectroscopy, particularly using handheld NIR spectrometer, as a rapid and non-destructive technique to differentiate palm oil from adulterated palm oil.

## **2. Materials and methods**

### *2.1. Materials*

#### *2.1.1. Chemicals and reagents*

All chemicals used were of analytical grade. Distilled water is used for dilutions and preparation of standard solutions. Wij's iodine solution, p-anisidine, sodium thiosulphate (0.1 N), chloroform, potassium hydroxide (0.1 N), glacial acetic acid and sodium methoxide (C.P. Grade) were purchased from Ever Gainful Enterprise. Potassium iodide (CHEMSOLN), isooctane (99.5 %, CHEMSOLN), phenolphthalein (0.1 % alcoholic, CHEMSOLN), and ethanol (95 %, CHEMSOLN) are purchased from Synertec Enterprise. Phosphoric acid (85 %) and bleaching earth are purchased from LGC Scientific. All other chemicals used are of high-grade quality and available commercially.

### *2.1.2. Sample collection and preparation*

Commercially available palm oil (Agro brand) was purchased from the local market. Three packet oil (PO) samples (three repetitions for each) were purchased randomly from the local market in Cheras, Malaysia. Frozen chicken nuggets (Farm best brand) and frozen French fries (Farm Frites brand) were procured from a near supermarket in Cheras, Selangor, Malaysia. The manual deep-frying process was carried out in the food science laboratory to prepare recycled cooking oil (RCO), followed by the process of refining and bleaching. All processes are carried out under controlled conditions.

## *2.2. Methodology*

### *2.2.1. Preparation of recycled cooking oil*

#### *2.2.1.1. Frying process*

Chicken nuggets and French fries were equally weighed into the batches of 150 g. Frying pot of capacity 5 L was filled with 3.5 L palm oil (represented as a pure sample or pure palm oil in this thesis) and heated to a temperature of 180 °C in 15 minutes. After oil reached the desired temperature, frying items were fried one by one in different cycles such that the chicken nuggets fried first followed by French fries. The average frying times for the former and latter are 3 minutes and 5 minutes respectively. Frying time varies with the temperature of the oil and it depends on the fried items until they reached a golden brown color. After each frying cycle, there is an interval time of 30 minutes before the frying of other fried items.

The total numbers of 60 frying cycles were performed assuming that no food restaurant used the same oil for more than these frying cycles since the oil color becomes dark. The value of total polar compounds (TPC) was calculated after 10 min of each frying cycle according to the method describes in 2.3.3.1. The frying process was carefully carried out to make sure that the value of TPC should not exceed 24 % since 25% is the maximum limit set by the Malaysian Palm Oil Board (MPOB) after which the oil cannot be used for cooking or frying purpose. After this, used frying oil was allowed to reach normal temperature and then initially filtered by using a stainless steel strainer to remove the residues of nuggets and fries. The final filtration process of used frying oil was carried out to remove small black burning residues by using a vacuum filtration process with a 90 mm Whatman filter paper.

#### *2.2.1.2. Recycling process of used frying oil*

Used cooking oil collected after frying and subjected to multiple filtration processes was then subjected to a series of cleaning processes, including washing, degumming, and bleaching [20]. Deodorization of oil was not performed due to the unavailability of facilities.

##### *2.2.1.2.1. Washing*

Washing of used frying oil was carried out according to the method described by [20]. For washing of 1 L frying oil, 12.5 mL of distilled H<sub>2</sub>O was added in oil and stirred at 400 rpm for 15 min using a magnetic stirrer. It was then allowed to stand in a separatory funnel for around 30 minutes until clear separated aqueous and oil layers were formed. The water portion was then removed and the similar washing process was again made on the remaining quantity of oil. Heating of washed oil was performed at 105 °C for 20 min to ensure total removal of moisture.

##### *2.2.1.2.2. Degumming*

Degumming is the initial step in the refining of edible oils [21]. Degumming of oil was carried out according to the method described by [22] with slight alterations. Oil was initially heated at temperatures of 80-85 °C and then phosphoric acid (0.05% w/w of the oil volume obtained after washing and heating process) was added in the oil. The oil was then allowed to stand for a retention time of 20 min.

#### 2.2.1.2.3. Bleaching

Degummed oil was then subjected to the bleaching process as mentioned by [23] with slight modifications. For the bleaching of oil, 1.5 % w/w of bleaching earth was added in the oil at 120 - 140 °C followed by filtration with filter paper using Büchner flask while the oil was hot. For removing remaining residues of bleaching earth, the oil was leftover at room temperature overnight to allow the settling of bleaching earth at the bottom. Final filtration of oil was carried out to remove the residues of bleaching earth.

#### 2.2.2. Adulterated palm oil samples

Palm oil and recycled cooking oil were already prepared in the previous steps. For adulterated palm oil samples, palm oil samples are mixed with a concentration of recycled cooking oil varying from 1% to 50 % (v/v). The concentrations of adulterations were set as 1, 2, 3, 5, 10, 15, 20, 30, 40 and 50%. To ensure complete homogenization, the solutions were mixed for 15 minutes using a magnetic stirrer [20]. Figure 1 represents the pictorial view of the pure palm oil, 100 % RCO and palm oil samples adulterated with recycled cooking oil in the proportion of 50% to 1% (from left to right).



**Figure 1.** Pictorial view of pure, 100% RCO and RCO (50% to 1%) adulterated palm oil samples.

#### 2.2.3. Determination of physical and chemical properties

##### 2.2.3.1. Total polar compounds

Total polar compounds (TPC) were determined by using a prompt handheld device (Testo 270 deep-frying oil tester, Testo Inc., Germany) as mentioned by [24]. The instrument manual was followed for taking measurements. TPC of the oil used for frying was measured after 10 minutes of each frying cycle [20]. To measure total polar compounds, the optimum temperature of the oil should be between 40 °C and 200 °C [25]. Each TPC value took ten seconds to become stable and it was noted along with the temperature.

##### 2.2.3.2. Peroxide Value

The peroxide value was determined according to AOAC Official Method 965.33 [26]. Each oil sample is weighed 5 g ( $\pm$  50 mg) in a 250 mL conical flask. A mixture of acetic acid and chloroform solvent is prepared in the ratio of 3:2 and then 30 mL of it is mixed to each oil sample and allowed to dissolve. Then, 0.5 mL of freshly prepared saturated potassium iodide solution is added and the solution is kept

for 1 min in dark place with infrequent shaking followed by the addition of 30 mL distilled water. The burette is filled with 0.01 N sodium thiosulphate solution to slowly titrate the liberated iodine with continuous shaking until the yellow color is disappeared. Then, 0.5 mL of the starch solution is added and the titration is continued with continuous shaking to release all iodine from the CH<sub>3</sub>Cl layer until the blue color disappeared. Peroxide value expressed as milliequivalent of peroxide oxygen per kg sample (meq/kg) is estimated using the following equation:

Equation 1:

$$\text{Peroxide Value} = \frac{\text{Volume of titre} \times N \times 1000}{\text{Weight of sample (g)}}$$

Where,

N = Normality of sodium thiosulphate

#### 2.2.3.3. Acid Value

All oil samples are weighed 10 g each in 250 mL conical flask and 50 mL of freshly neutralized hot ethyl alcohol is added. One mL of phenolphthalein indicator is mixed with each sample and the resulting mixture is subjected to boiling for 5 minutes. While the solution is hot, it is titrated with 0.1 N potassium hydroxide solution with continuous shaking until light pink color appeared permanently. The acid value is estimated using the following equation [27]:

Equation 2:

$$\text{Acid Value} = \frac{56.1 \times \text{Volume of titre} \times N}{\text{Weight of sample (g)}}$$

Where,

N = Normality of potassium hydroxide

#### 2.2.3.4. Iodine Value

Oil sample (10 g) is weighed in 250 mL conical flask, followed by 10 mL of chloroform along with 25 mL of Wij's solution is added in the oil sample and the solution is well mixed. Then, the mixture is allowed to stand for 30 min in dark followed by the addition of 15 mL of freshly prepared potassium iodide solution. After this, 50 mL of distilled water is added and the resulting mixture is titrated with 0.1 N sodium thiosulphate solution until a pale straw color is observed. Starch indicator (1 mL) is added and the titration process is continued again with vigorous shaking until the blue color of the solution in the flask turns colorless and the volume of sodium thiosulphate used is noted. A similar process is repeated for a blank sample without the presence of oil. Iodine number of oil is calculated using the following equation [26]:

Equation 3:

$$\text{Iodine Value} = \frac{12.69 (B - S) \times N}{\text{Weight of sample (g)}}$$

Where,

B = Titre of blank

S = Titre of sodium thiosulphate

N = Normality of sodium thiosulphate

#### 2.2.3.5. Anisidine value

To find out the anisidine value of oils, approximately 0.5 g of an oil sample is weighed in 25 mL measuring cylinder and the volume is made up with iso-octane. Then, it is mixed in a way to dissolve the sample completely. Absorbance is measured at 350 (nm) against pure iso-octane followed by the addition of 1 mL p-anisidine reagent. The measuring cylinder is then covered with cork and kept in dark for 10 minutes. Finally, absorbance is again measured at 350 (nm) wavelength of a sample solution containing anisidine reagent. Anisidine value is determined using the following equation [28]:

Equation 4:

$$p - \text{anisidine Value} = 37.5 \times \frac{(\text{Absorbance with anisidine} - \text{Absorbance without anisidine})}{\text{Weight of sample (g)}}$$

#### 2.2.3.6. Statistical analysis

Each sample was analyzed in terms of chemical properties in triplicate (n=3) and the values obtained are represented as mean values  $\pm$  standard deviation. All data collected were analyzed by one-way analysis of variance (ANOVA) Tukey test. The statistical software package Minitab® 19 for Windows (Minitab LLC., State College, PA, USA, ver. 19.2) was used. Statistical significance was set at  $p < 0.05$ .

#### 2.2.4. NIR spectra measurement

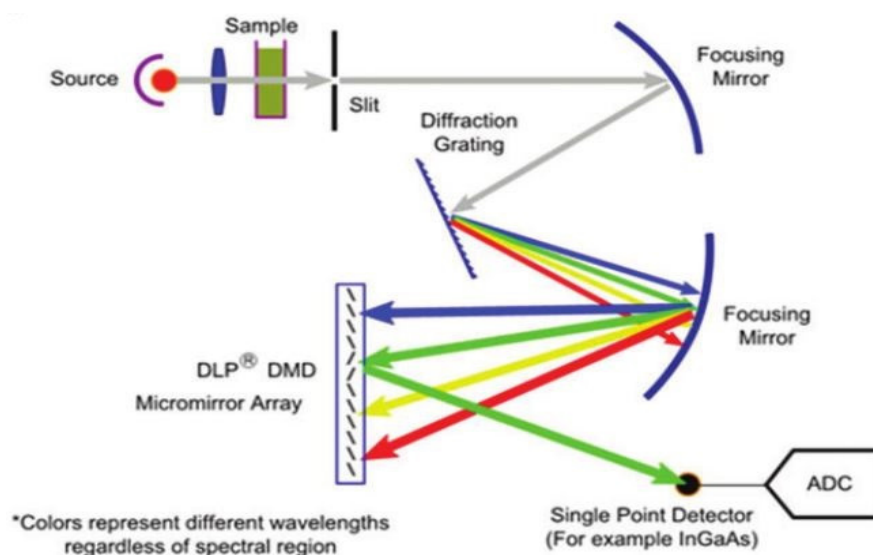
Palm oil and recycled cooking oil were prepared without any adulterant. The sets of ten each adulterated palm oil sample sets were made with varying concentrations of RCO (1 to 50 % v/v). NIR spectral acquisition was executed according to the process described by [29-31] with few amendments. The spectra of all oil samples were measured using handheld NIR spectrometer with the engine developed by Texas Instruments (Figure 2) in wavelength range of 900 to 1700 nm. Oil samples were pipetted with a glass pipette into a 20 mL glass cuvette of size 50 mm and each sample was scanned 32 times an average at three different locations. The spectra of all samples were taken against the same background to prevent variations in observations.



**Figure 2.** Pictorial view of handheld NIR spectrometer used.

The handheld spectrometer (NIRscan™ Nano by Texas Instrument) works by illuminating two lamps held at an angle at the side of the collimating lens. The lens collects the reflected light from the sample and sends the light onto a diffraction grating, where the light diffracts and only the wavelengths corresponding to near infra-red are sent to the detector by the second diffraction at the wavelength

selector array. The detector analyses the received light signal and calculates the absorbance value from the reflectance value. The working principle of the NIRscan™ Nano is shown in Figure 3.



**Figure 3.** Handheld spectrometer (NIRscan™ Nano-Texas Instruments) working diagram.

After carrying out sample measurements, chemometrics techniques such as Principle Component Analysis (PCA) and logistic regression were used. These techniques were performed using the “Orange 3.23.1” software. The spectral areas where the dissimilarities found between palm oil and recycled cooking oil were considered for classification purposes. Spectral processing including Savitzky-Golay (S-Golay) derivatives, baseline correction as well as Gaussian smoothing was considered and applied [11].

The principal component analysis was implemented on the NIR spectra of oil samples. PCA is a multivariate unsubstantiated technique used in a tentative analysis of data. PCA decreases the dimensionality of the data set by substituting a set of coordinates: principal components, PCs and it has the tendency to analyse the data on both observation and variable levels. The principal components are linear groupings of original variables, orthogonal to each other, with each one consecutively accounting for the extreme inconsistency of the data sample [32].

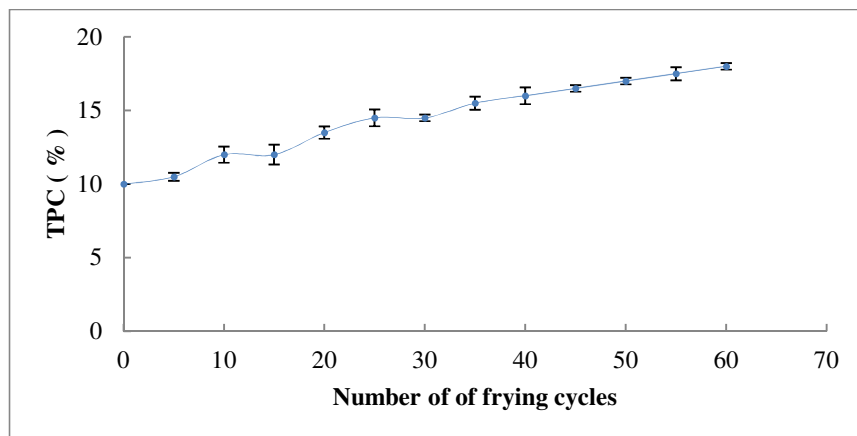
### 3. RESULTS AND DISCUSSION

#### 3.1. Chemical evaluation of oil samples

##### 3.1.1. Total polar compounds

In terms of oil quality, acid value and total polar compounds are widely considered as principal measures [33, 34]. It was found that after 60 cycles of the deep-frying process, TPC of the oil did not exceed a 25% limit as set by the Malaysian government. Figure 4 represents the graph showing an increase in total polar compounds with an increase in the number of frying cycles performed. It was clearly observed that there was variability in change in TPC (0 to 1.5 %) from 0 to 35 frying cycles. This observation is

in agreement with the study conducted by [24], which stated that the duration of heating and nature of oil affects the total polar compounds when measuring through Testo 270.



**Figure 4.** Increase in TPC with respect to the number of frying cycles.

### 3.1.2. Peroxide Value, Acid Value, Anisidine value and Iodine value

The peroxide values of pure, used and palm oil samples adulterated with recycled cooking oil were determined by acid-base titration technique. PV measures a transient (temporary) product of oxidation and the low peroxide values may represent early or advanced oxidation; which can be distinguished with time [35]. Peroxide value is a significant biomarker that points out the preliminary phases of oxidation [36]. Even the exposure of the oil samples to sunlight or ultraviolet rays for a short period of time from fluorescent lighting will result in increasing of peroxide values and decrease flavor grading [37]. Table 1 shows the peroxide value of pure oil, used frying oil and recycled cooking oil.

**Table 1.** Peroxide values of pure, used and recycled cooking palm oil samples

Oil Samples	Peroxide Value (meq/kg)	Acid Value (mg KOH / g oil)	Anisidine Value (mEq/kg)	Iodine Value (gI <sub>2</sub> /100g)
Pure palm oil	1.33 ± 0.11 <sup>a</sup>	0.76 ± 0.03 <sup>a</sup>	2.17 ± 0.39 <sup>a</sup>	53.99 ± 0.23 <sup>b</sup>
Used frying oil	13.06 ± 0.46 <sup>b</sup>	3.15 ± 0.31 <sup>b</sup>	68.11 ± 0.27 <sup>c</sup>	51.60 ± 0.38 <sup>a</sup>
Recycled cooking oil	1.46 ± 0.11 <sup>a</sup>	4.10 ± 0.20 <sup>c</sup>	59.20 ± 0.56 <sup>b</sup>	50.92 ± 0.83 <sup>a</sup>

<sup>a-c</sup> Means ± standard deviation (triplicate determination) followed by different superscripts within the same column represented a significant difference at p<0.05 according to one way ANOVA Tukey's test.

From Table 1, it was observed that peroxide values of pure and recycled cooking oil are nearly equal, whereas; the used frying oil showed relatively high peroxide value which is in confirmation with the study conducted by [25] on heated oil samples. Similarly, [36] mentioned that the peroxide value of oil after 100 frying cycles increased from 7.08 to 15.48 (meq/kg). A product is in moderate oxidation state if it has peroxide value between 5 and 10 meq/g and high oxidation occurs when peroxide value exceeds



10 meq/g [38]. The peroxide value of pure palm oil was found to be in good agreement with the Malaysian standards [39] and also has similar values as mentioned by [40].

The acid value is considered as an indicator of triglyceride hydrolysis, which takes place as a result of oil decomposition during oil exposure to heat [25]. Both acid value and free fatty acid are used analytically to identify the amount of unesterified fatty acids in an oil sample to represent its excellence [35]. The acid values of pure, used and recycled cooking oil were shown in Table 1. Used frying oil showed a high acid value than pure palm oil. It is because when the number of frying cycles increases, there is usually a rise in the acid value irrespective of the oil type [21].

On the other hand, the acid value of recycled cooking oil was significantly higher as compared to used and pure palm oil. Since the bleaching process was not carried out under vacuum, this results in the high acid value of recycled cooking oil [41]. Similar results were reported by [42], who concluded that an increase in acid value is because of the addition of phosphoric acid during the degumming process as well as hydrolysis of triglyceride during the heating process [37]. As mentioned before, TPC of the oil after 60 frying cycles was found to be under permissible limits, but the acid value of used frying oil exceeds the maximum range. This is in agreement with the study conducted by [43] in which authors found high acid values in the samples but relatively lesser samples showed high TPC values. The allowable limit of acid value should be less than 0.6 mg KOH / g according to the recommendation by FAO / WHO for all edible vegetable oils [44].

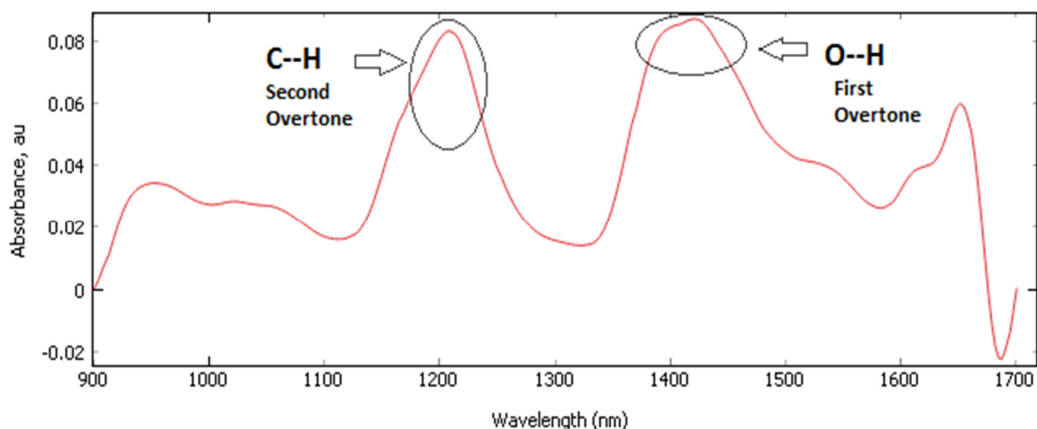
Usually, anisidine values start to increase with bleaching temperatures above 110 °C, specifying destruction to oxidative stability [37]. The results showed the accurate values of the anisidine for pure palm oil according to the Malaysian standard [39]. On the other hand, anisidine values of used and recycled cooking oil were found extraordinary high (Table 1). The high values of the anisidine in the used frying oil are because of the formation of secondary oxidation products due to prolonged exposure to high-temperature treatment [45, 46]. The anisidine value of oil used for five consecutive days of frying in this study was found to be 68.11 mEq/kg, which is quite similar to the results in the study on the quality of discarded oil conducted by [47].

The iodine value is also one of the significant parameters for checking oil quality and it represents the degree of unsaturation of oils and fats [48]. The iodine values of pure palm oil used frying oil, and the recycled cooking oil was represented in Table 1. It was observed that the iodine values of used and recycled cooking oil were lower than the pure palm oil. The main reason for the lower iodine value of used frying oil is the decomposition of double bonds by polymerization and oxidation reactions due to frying temperatures [49]. It is also in agreement with the study in which the iodine value of oil used for frying fast food was found to be lower than the pure palm oil [50].

### 3.2. NIR spectral data

#### 3.2.1. Spectrum of pure palm oil

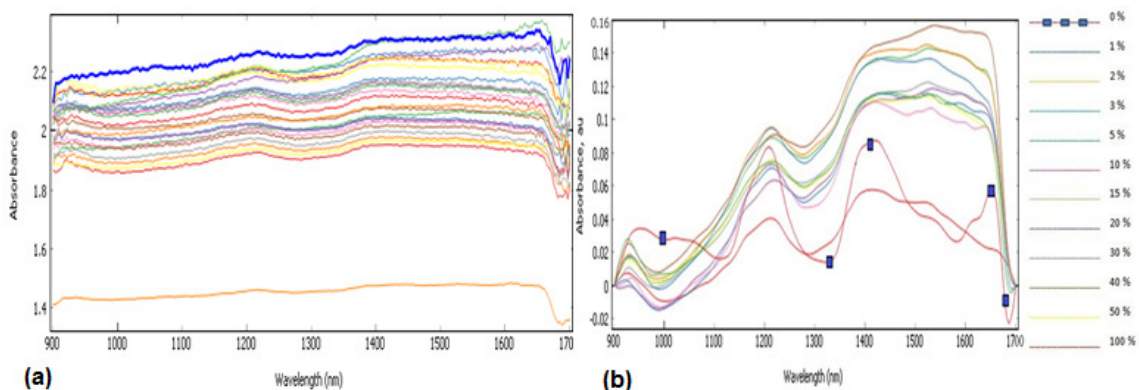
Figure 5 represents the absorbance spectra of pure palm oil. There were two significant peaks that observed as shown in the figure. The first peak observed at 1200 nm is associated with carbon and hydrogen bonding in fats and oils and is classified as C-H second overtone, whereas; the other vibrational mode i.e. O-H stretch first overtone occurs around 1450 nm [51]. The overlapping absorptions correspond to a wide range of overtone bands which build NIR an authentic tool for computation of organic and biological systems [17].



**Figure 5.** Absorbance spectra of pure palm oil.

### 3.2.2. Spectra of pure, used and adulterated palm oil samples

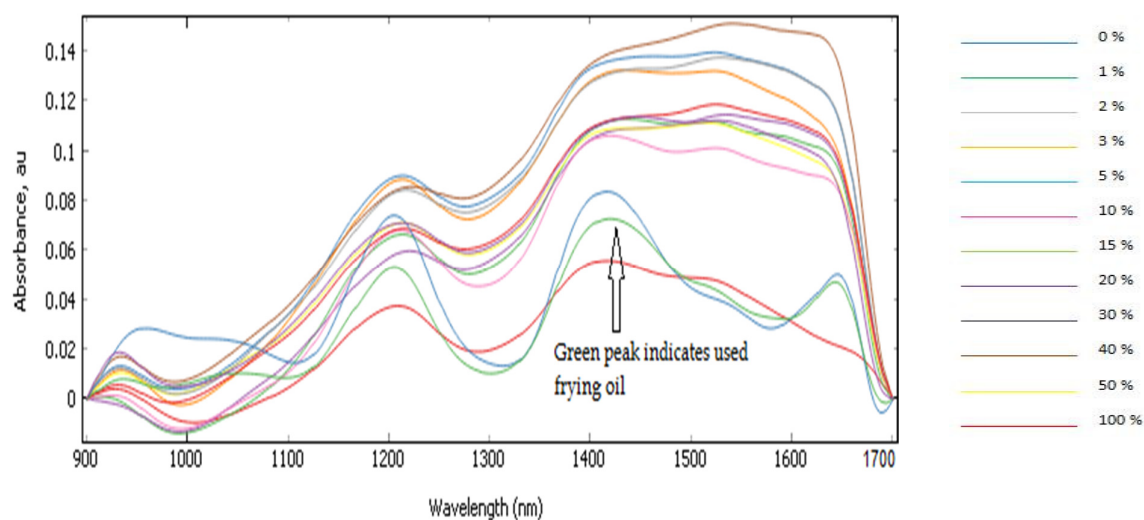
The combined original spectrum of pure, used and RCO adulterated palm oil samples between 900-1700 nm are shown in Figure 6 (a). All the spectra seem like showing the same pattern, as all contain a similar composition of fatty acids in oil [15]. It was seen that the absorbance of samples varies due to the differences in concentration of adulteration.



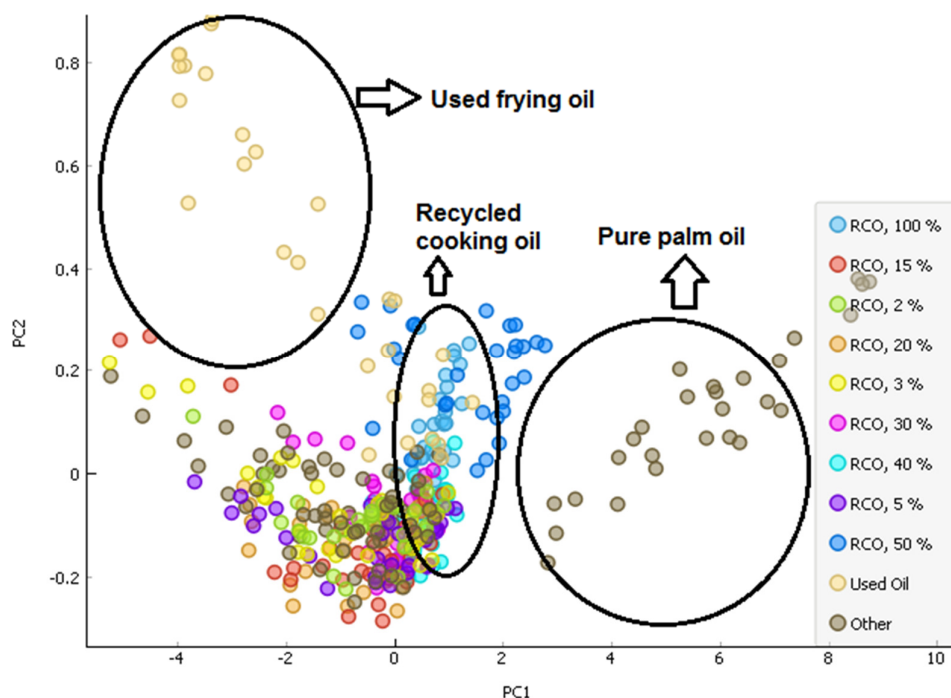
**Figure 6.** (a) NIR spectra of all samples before preprocessing (b) After preprocessing

Figure 6 (b) represents the individual spectrum (after preprocessing) of the pure sample and the adulterated samples with recycled cooking oil of concentration varying from 0 to 50 %, whereas; 0 % and 100 % sample indicated the pure palm oil and recycled cooking oil respectively. There was a clear difference observed between pure palm oil and recycled cooking oil, whereas; all other adulterated samples were showing a similar pattern. Therefore, adulterated oils could be discriminated in terms of the position of the absorption band and the absorption intensity of the same band [16]. A band at 1450 nm that corresponding to  $-OH$  first overtone stretching vibration shown to be wider and intense in recycled cooking oil as compared to pure palm oil, indicating a greater intensity of band absorbance [20].

The spectrum of used oil (indicated by green coloured peak) was found to have a similar pattern as pure oil sample but not similar to recycled cooking oil (Figure 7). This means that the recycling process which involves the bleaching step has resulted in the formation of decomposition products, such as conjugated trienes and hydroperoxides [41]. It was also observed that the height of peaks of pure palm oil was higher than that of used frying oil and recycled cooking oil in the large area of the NIR region. This is in agreement with the study in which the peak height of edible oil was found higher at 1390-1480  $\text{cm}^{-1}$  than that of used frying oil because of complex chemical reactions during the frying process that resulted in hydrogen substitution [16]. The scatter plot of all samples was shown in Figure 8 (a).



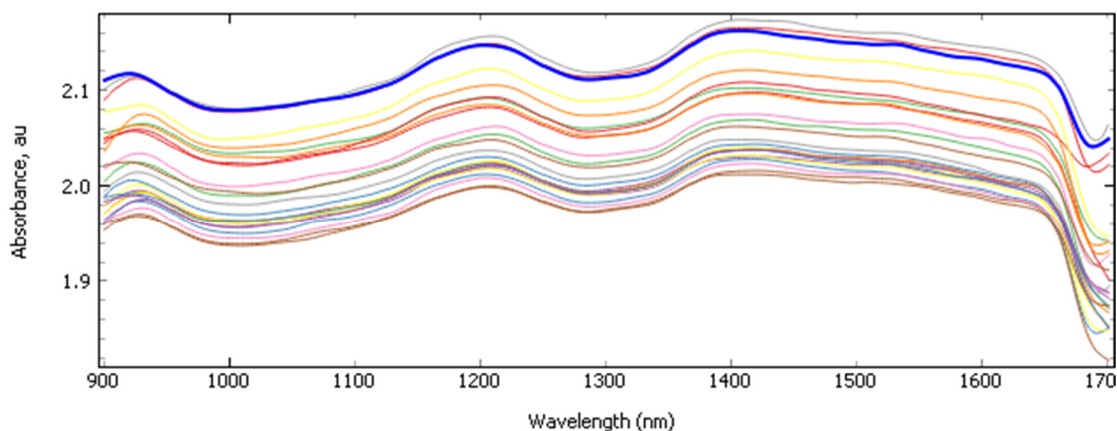
**Figure 7.** Spectra of all samples including used frying oil.



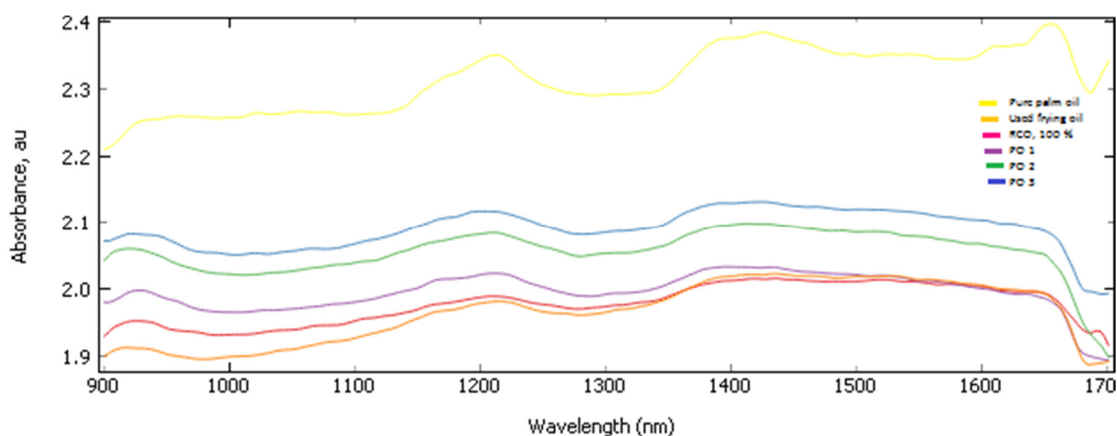
**Figure 8. (a)** The scatter plot of adulterated, 100% RCO and pure palm oil samples. “Other” includes 1% and 10 % RCO samples.

### 3.2.3. Spectra of packet oil samples

Three packet oil (PO) samples of different brands (three repetitions of each) were scanned through NIR to receive spectral data. The combined spectra of all three oil packet oil samples showing a similar pattern of peaks are shown in Figure 9(a). It was observed that the samples can be distinguished with the difference in their absorbance peaks. The pure oil sample was showing quite different and sharp peaks as compared to packet oil samples, used frying oil and RCO (Figure 9 (b)). This also suggested that PO1, PO2, and PO3 have the possibility of adulteration with RCO.



**Figure 9. (a)** NIR spectra of packet oil samples.

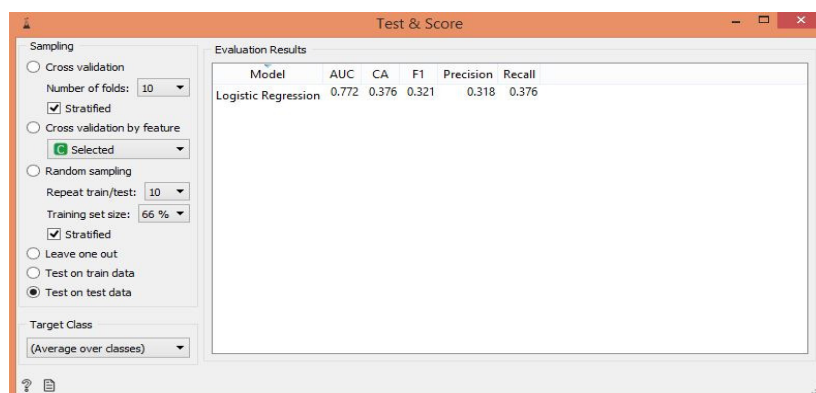


**Figure 9. (b)** NIR spectra of pure palm oil, used frying oil, RCO (100%) and three PO samples.

### 3.3. Classification accuracy and confusion matrix

#### 3.3.1. Pure, used and adulterated palm oil samples

Two principal components were used for analysis. It was observed that the classification accuracy of NIR in detecting a low concentration of adulterated samples was 37.6 % which was quite low (Figure 10). Even after applying preprocessing technique i.e. Gaussian smoothing, the classification accuracy shows minimal changes and reaches 41.9 %. This clearly showed that NIR was unable to detect adulterated samples with a low concentration of adulteration. The overall accuracy can be obtained from the confusion matrix [5]. Figure 11 (a) and (b) showed the confusion matrix representing the predictions carried out by NIR before and after preprocessing, respectively.



**Figure 10.** Classification accuracy of adulterated, 100% RCO and pure palm oil samples before preprocessing.

		Predicted													Σ
		Pure Sample	RCO, 1 %	RCO, 10 %	RCO, 100 %	RCO, 15 %	RCO, 2 %	RCO, 20 %	RCO, 3 %	RCO, 30 %	RCO, 40 %	RCO, 5 %	RCO, 50 %	Used Oil	
Actual	Pure Sample	10	0	1	0	0	0	0	0	0	0	0	0	0	11
	RCO, 1 %	0	1	2	2	0	0	1	0	1	2	0	0	0	9
	RCO, 10 %	0	1	3	1	0	0	1	1	0	1	0	0	0	8
	RCO, 100 %	0	0	0	7	0	0	0	0	0	0	0	0	0	7
	RCO, 15 %	0	0	0	0	0	0	5	2	0	3	1	0	0	11
	RCO, 2 %	0	0	1	0	0	0	4	1	4	1	0	0	0	11
	RCO, 20 %	0	1	0	1	0	0	2	0	2	2	1	0	0	9
	RCO, 3 %	0	0	2	0	0	0	1	1	0	2	0	0	2	8
	RCO, 30 %	0	0	1	1	0	0	1	0	2	2	0	0	1	8
	RCO, 40 %	0	0	0	1	0	0	0	0	0	7	0	1	0	9
	RCO, 5 %	0	0	0	0	0	0	3	2	1	4	0	0	0	10
	RCO, 50 %	0	0	0	0	0	0	0	0	0	1	0	5	3	9
	Used Oil	0	0	0	1	0	0	0	0	0	0	0	0	6	7
	Σ	10	3	10	14	0	0	18	7	10	25	2	6	12	117

**Figure 11. (a)** Confusion matrix of adulterated, 100% RCO and pure palm oil samples before preprocessing.

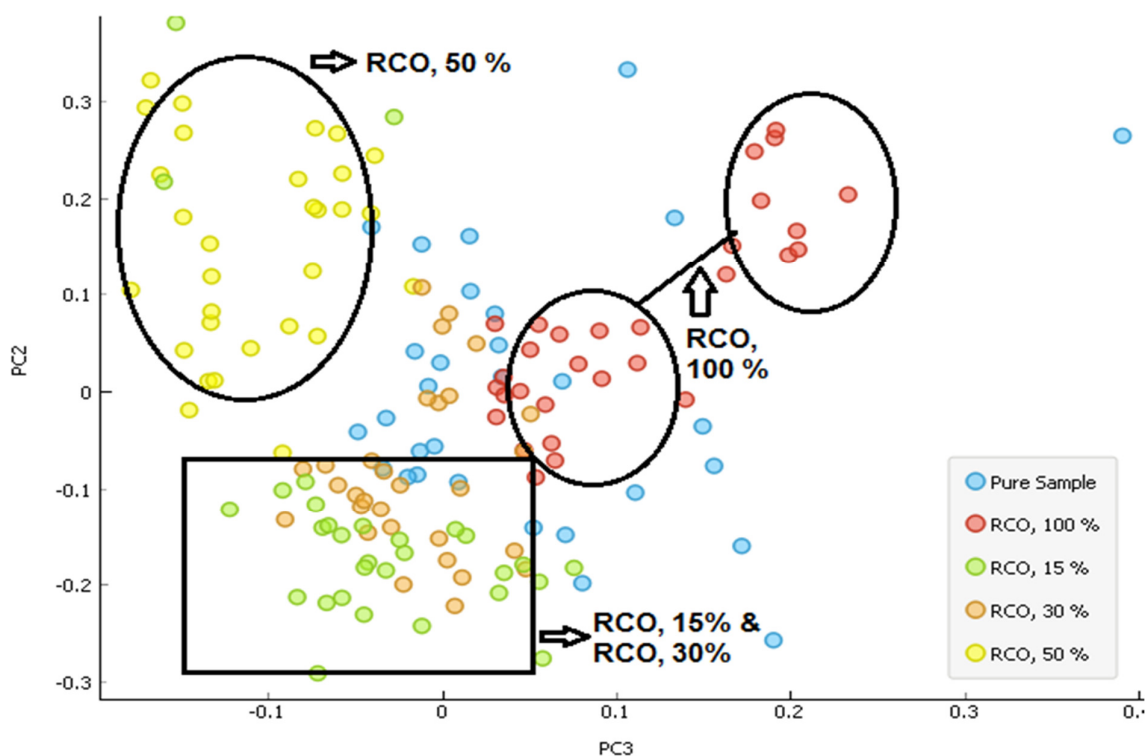
The use of a confusion matrix is preferred as the direct representation of the prediction results [5]. Also, viewing the classification result by showing a confusion matrix is quiet convenient [15]. In both above-stated figures, pure, used and 100% RCO samples were predicted correctly by NIR, whereas; prediction accuracy was also observed satisfactory for RCO 50% and RCO 40%. However, 30% of RCO has classified accurately after preprocessing as shown in Figure 11 (b). On the other hand, RCO 20% and low concentrated adulterated palm oil samples were not classified properly.

		Predicted													Σ
		Pure Sample	RCO, 1 %	RCO, 10 %	RCO, 100 %	RCO, 15 %	RCO, 2 %	RCO, 20 %	RCO, 3 %	RCO, 30 %	RCO, 40 %	RCO, 5 %	RCO, 50 %	Used Oil	
Actual	Pure Sample	7	0	1	0	0	0	0	0	0	0	0	0	0	8
	RCO, 1 %	0	0	1	0	0	0	0	0	7	0	0	0	0	8
	RCO, 10 %	0	0	5	0	0	0	0	0	2	1	0	0	0	8
	RCO, 100 %	0	0	0	8	0	0	0	0	1	0	0	1	0	10
	RCO, 15 %	0	0	0	0	0	0	4	0	6	1	0	0	1	12
	RCO, 2 %	0	0	3	0	0	0	2	0	6	1	0	0	0	12
	RCO, 20 %	0	0	1	0	0	0	4	0	3	0	0	0	0	8
	RCO, 3 %	0	0	2	0	0	0	2	1	3	1	0	0	0	9
	RCO, 30 %	0	0	0	0	0	0	1	0	4	0	0	0	0	5
	RCO, 40 %	0	0	0	0	0	0	0	0	2	5	0	1	0	8
	RCO, 5 %	0	0	2	0	0	0	2	0	4	2	0	0	0	10
	RCO, 50 %	0	0	0	0	0	0	0	0	0	0	0	7	0	7
	Used Oil	0	0	0	1	0	0	0	0	0	0	0	3	8	12
	Σ	7	0	15	9	0	0	15	1	38	11	0	12	9	117

**Figure 11. (b)** Confusion matrix of adulterated, 100% RCO and pure palm oil samples after applying Gaussian smoothing.

### 3.3.2. Pure and some selected adulterated palm oil samples

After analyzing the ability of NIR in detecting samples from low concentration to high concentration adulteration, another set of data with only high concentration samples were classified using principal component analysis. The scatter plot of these samples is shown in Figure 8 (b). It was observed that the majority of 15% RCO samples found in the same region similar to RCO 30%. However, 50 % and 100 % RCO samples were showing clear separate regions due to their typical principal components. Figure 12 (a) and (b) showed the confusion matrix of the samples before and after preprocessing, respectively. Even after reducing the sample size, NIR was unable to classify RCO 15 % accurately. Results showed that 6 samples of RCO 15 % were classified as RCO 30% (Figure 12 (a)).



**Figure 8. (b)** The scatter plot of adulterated (15%, 30%, 50%), 100% RCO and pure palm oil samples.



		Predicted					
		Pure Sample	RCO, 100 %	RCO, 15 %	RCO, 30 %	RCO, 50 %	Σ
Actual	Pure Sample	8	0	0	0	0	8
	RCO, 100 %	0	4	0	0	0	4
	RCO, 15 %	0	0	2	6	0	8
	RCO, 30 %	0	0	1	3	0	4
	RCO, 50 %	0	0	0	0	6	6
Σ		8	4	3	9	6	30

**Figure 12. (a)** Confusion matrix of adulterated (15%, 30%, 50%), 100% RCO and pure palm oil before preprocessing.

It was observed that after applying Gaussian smoothing, the classification accuracy reached 1.00 (100%), but still, there was an error found in the classification of 15% RCO and 30% RCO. However, pure palm oil was precisely classified from recycled cooking oil samples through NIR (Figure 12 (b)).

		Predicted					
		Pure Sample	RCO, 100 %	RCO, 15 %	RCO, 30 %	RCO, 50 %	Σ
Actual	Pure Sample	11	0	0	0	0	11
	RCO, 100 %	0	5	0	1	0	6
	RCO, 15 %	0	0	7	1	0	8
	RCO, 30 %	0	0	4	4	0	8
	RCO, 50 %	0	2	0	1	9	12
Σ		11	7	11	7	9	45

**Figure 12. (b)** Confusion matrix of adulterated (15%, 30%, 50%), 100% RCO and pure palm oil after applying Gaussian smoothing.

### 3.3.3. Pure, adulterated and packet oil samples

The authentication of packet oil samples (PO1, PO2, and PO3) available in the market was carried out. It was observed that all three packet oil samples were not detected as pure palm oil samples used in our study (Figure 13). It means that the packet oil samples have different fatty acid compositions as compare to pure palm oil. However, NIR was unable to differentiate among all three packet oil samples, maybe because they all have similar properties. On the other hand, it was quite interesting that 1,3 and 6 samples of RCO 15%, RCO 20%, and RCO 40% were detected as PO2 before preprocessing by NIR, respectively. This does not indicate that PO2 is adulterated because RCO produced in the laboratory might not be similar to the waste oil or recycled cooking oil produced in the food or commercial frying industries [20].

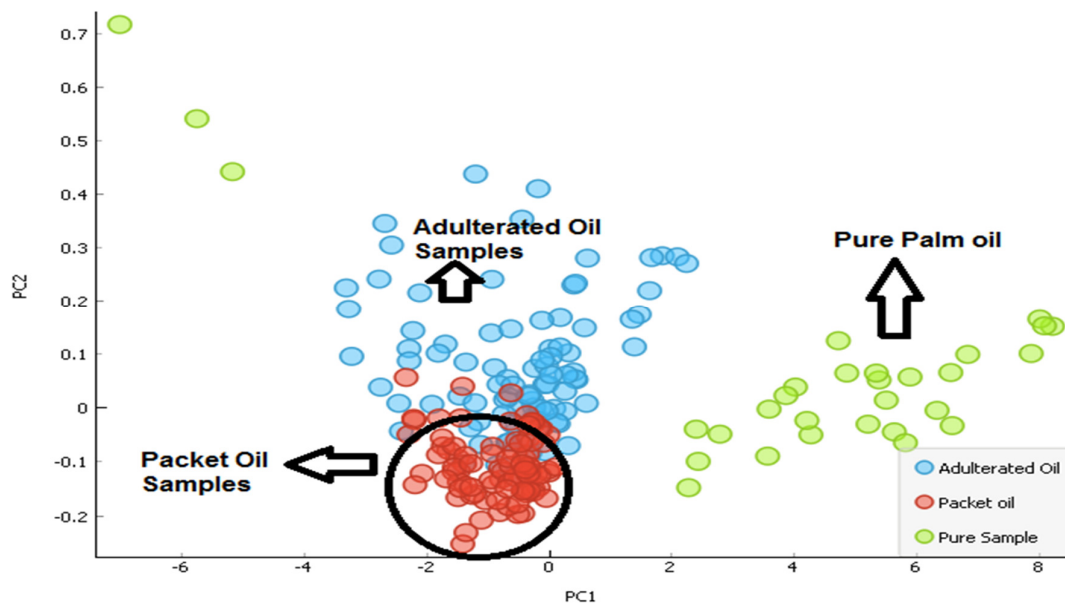


Even after the preprocessing of spectra (Figure 13), the confusion matrix was showing some negative results for PO2, where RCO samples (10%, 15%, 20%, 30%, 40%) were detected as PO2. This again does not suggest PO2 as adulterated palm oil because PO1 and PO3 were also detected as PO2. Besides, the pure palm oil used in the study belongs to one single brand, so variations in oil composition with respect to storage conditions and expiry dates might be overlooked [20]. The overall accuracy was also found to be below 50%, which is quite poor but a pure palm oil sample was accurately detected (100%).

		Predicted													
		RCO, 100 %	Used Oil	PO 1	PO 2	PO 3	Pure Sample	RCO, 10 %	RCO, 15 %	RCO, 20 %	RCO, 30 %	RCO, 40 %	RCO, 50 %	Σ	
Actual	RCO, 100 %	10	0	0	0	0	0	0	0	0	0	0	0	10	
	Used Oil	1	8	0	0	0	0	0	0	0	0	0	3	12	
	PO 1	0	0	1	7	0	0	0	0	0	0	0	0	8	
	PO 2	0	0	0	7	0	0	0	0	0	0	0	0	7	
	PO 3	0	0	0	8	0	0	0	0	0	0	0	0	8	
	Pure Sample	0	0	0	0	0	7	1	0	0	0	0	0	8	
	RCO, 10 %	0	0	0	4	0	0	3	0	0	0	0	0	7	
	RCO, 15 %	0	0	0	4	0	0	3	0	0	0	3	0	10	
	RCO, 20 %	1	0	0	5	0	0	4	0	0	0	0	0	10	
	RCO, 30 %	1	0	0	3	0	0	4	0	0	0	2	0	10	
	RCO, 40 %	0	0	0	4	0	0	1	0	0	0	4	0	9	
	RCO, 50 %	0	1	0	0	0	4	0	0	0	0	0	4	9	
Σ	13	9	1	42	0	11	16	0	0	0	0	9	7	108	

**Figure 13.** Analysis of packet oil samples along with other samples after applying Gaussian smoothing.

In order to resolve this issue, all samples are simplified in the way that RCO (10 to 50%) considered as one class and named as adulterated palm oil (Figure 8 (c)). The spectral information of used oil and RCO 100 % was not used since all three POs were clearly differentiated from these samples. The scatter plot in Figure 8 (c) represents all three classes of oil classified on the basis of their principal components. It was observed that there is not a huge difference in the distance between the region of adulterated palm oil and packet oil samples, whereas; pure palm oil samples were shown separately in a different region.



**Figure 8. (c)** The scatter plot of adulterated (15%, 30%, 50%), packet oil and pure palm oil samples.

The confusion matrix for this simplified sample data was also analysed. Figure 14 (a) and (b) represents the confusion matrix before and after preprocessing, respectively. All packet oil samples and pure samples were classified accurately before and after preprocessing stages. However, 10 and 3 samples of adulterated palm oil were again detected as packet oil samples by NIR before and after the application of Gaussian smoothing, respectively. This classification by NIR was found to be significant, with a classification accuracy of 84.1% and 93.7% before and after preprocessing, respectively. It is also suggested that NIR was able to detect fewer classes more precisely as compared to a huge range of samples.

		Predicted			$\Sigma$
		Adulterated Oil	Packet oil	Pure Sample	
Actual	Adulterated Oil	22	10	0	32
	Packet oil	0	25	0	25
	Pure Sample	0	0	6	6
$\Sigma$		22	35	6	63

**Figure 14. (a)** Confusion matrix of adulterated palm oil, packet oil, and pure palm oil before preprocessing.

		Predicted			
		Adulterated Oil	Packet oil	Pure Sample	$\Sigma$
Actual	Adulterated Oil	23	3	0	26
	Packet oil	0	30	0	30
	Pure Sample	1	0	6	7
$\Sigma$		24	33	6	63

**Figure 14. (b)** Confusion matrix of adulterated palm oil, packet oil, and pure palm oil after Gaussian smoothing.

It is concluded that the findings and models developed in this study from pure palm oil and recycled cooking oil are not well applicable to the packet oil samples found in the local market, resulting in the inconsistent classification and authentication of the packet oil samples. Thus, the pure palm oil from different brands should be used as well as the laboratory prepared RCO and commercial RCO should be taken into consideration in future studies.

#### 4. CONCLUSIONS

The research conducted above was divided into three sections: the main objective of this study was to determine the feasibility of near-infrared spectroscopy in detecting adulteration in palm oil with recycled cooking oil so as to develop a rapid approach for authentication of palm oil. The second section was about determination of the chemical properties of pure palm oil, used frying oil, recycled cooking oil and the adulterated palm oil with varying ROC concentrations were determined as peroxide value, acid value, anisidine value, and iodine value. Lastly, market feasibility such as SWOT analysis of palm oil in Malaysia and the target market for promoting the use of NIR was also studied.

The total polar compounds after 60 frying cycles were found to be 18 % (under the allowable limit). Recycled cooking oil was produced by applying refining (pre-filtration, washing, and degumming) and bleaching process followed by post-filtration. The adulterated samples were prepared with different proportions of palm oil and recycled cooking oil (0, 1, 2, 3, 5, 10, 15, 20, 30, 40, 50% (RCO)). There was an uneven change in TPC values observed after every 5 cycles of frying until 35 frying cycles range from 0 to 1.5% TPC, and then constant change in TPC value of 0.5 % from 35 to 60 frying cycles. The changes in peroxide value were found to be non-linear with respect to change in concentration of recycled cooking oil in palm oil. The acid value showed the linear graph as an increase in the concentration of adulteration resulted in high acid values. Similarly, anisidine value also found to be increased with an increase in the proportion of recycled cooking oil in palm oil.

Pure palm oil and RCO adulterated palm oil samples were analyzed through NIR at 900-1700 nm wavelength range. Principle component analysis was used to distinguish between these samples and a pre-processing technique such as Gaussian smoothing was applied to analyze the spectra more efficiently. The classification accuracy of detecting the samples with adulterations less than 15 % through NIR was found to be very low. However, NIR distinguished among pure palm oil, used frying oil as well as recycled cooking oil along with adulterated samples with concentrations of 15 % and above effectively. Moreover, packet oil samples were also analyzed for detecting adulteration, but results

showed no possible adulteration in these samples. However, these packet oil samples were found to have quite different peaks than pure palm oil.

Therefore, it was concluded that DLP® NIRscan™ NANO EVM was able to detect adulterated samples with concentrations above 10%. Different models of NIR have the potential to authenticate the market samples based on their adulteration level. However, packet oil samples in the market were not found to be adulterated based on this study.

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