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DETECTION OF FFA AND PV VALUES USING FTIR FOR QUALITY MEASUREMENT IN PALM OIL FRYING ACTIVITIES

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ABSTRACT

Fourier Transform Infra-red Spectroscopy (FTIR) instrument has been used for rapid analysis in several investigations. The analysis of frying oil quality was carried out using FTIR. FTIR profiles of commercial palm oil during short-term fish frying were performed on free fatty acid (FFA) and peroxide values (PV) correlation using multivariate statistics to confirm the oil quality. Two batches of commercial palm oil were repeatedly used for nine times to fry catfish. Each frying was done at 180°C for 15 min. Samples were analysed for FFA and PV according to standard AOAC methods. FTIR spectra were collected in wavenumber 400-4000 cm^{-1} *by using 32 scans at resolution of 1.9 cm*$^{-1}$*. The correlation between the conventional analytical results and those obtained by FTIR was performed using Ordinary Least Square (OLS) multivariate analysis with XLSTAT 2011 software from Microsoft Excel Series. The analytical results showed that the worse oil quality had higher FFA and PV. FTIR spectra observed in this study mentioned the main peaks of oil functional groups such as alkyl, ester, single and double bonds of carbon atoms. The absorbance values of the peaks exhibited a significant correlation,* $R^2 = 0.955$ *at* P_{value} *0.042 for FFA values, and* $R^2 = 0.963$ *with* P_{value} *0.030 for PV. This significant correlation gives an insight that FTIR spectroscopy can be used for a rapid analysis of palm oil quality.*

Keywords: FFA value, FTIR spectroscopy, multivariate analysis, palm oil, peroxide value.

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INTRODUCTION

Palm oil is widely used as frying oil in Indonesia and other South-east Asian countries. Compared to other oils, palm oil has superior qualitiy such as higher oxidative stability due to its relatively high percentage (almost 40%) of saturated fatty acid (Basiron, 2005). To analyse the oil quality, some parameters such as free fatty acid (FFA) value and peroxide value (PV) are frequently used. Recently, a spectroscopic method, referred to as FTIR (Fourier Transform Infra-red) Spectroscopy, has become popular in the study of edible oils and fats (Guillen and Cabo, 2002). Some researchers have used this method to detect the adulteration in food, such as in butter (Safar *et al*., 1994), vegetable oils (Lai *et al*., 1995), cow and animal fats (Jaswir *et al*., 2003), chocolate products (Che Man *et al*., 2003), virgin olive oil (Vlachos *et al*., 2006), virgin coconut oil (Rohman and Che Man, 2010), and meat balls (Rohman *et al*., 2010). Other researchers have used this instrument to determine the amount of certain chemical compounds such as short chain fatty acids in Swiss cheese (Koca *et al*., 2007), marine fatty acids in lard (Flatten *et al*., 2004), FFA in palm oil (Che Man and Setyowati, 1998), and PV in canola

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oil (Li *et al.,* 2000). Some researchers have used this technique to monitor the change of chemical compound composition during processing, such as the emergence of *trans* isomer fatty acids in oils and fats during thermal oxidation process (Moreno *et al.,* 1999), and the change of FFA, viscosity, and total polar compounds during frying in palm and soyabean oils (Al- Degs *et al.,* 2011).

FTIR spectroscopy can identify the specific functional groups in samples (Bendini *et al.,* 2007). They are identified by certain absorption bands at certain wavenumbers (cm^{-1}). The magnitude of the absorption could be varied between samples. The magnitude could be correlated to the sample quality. The oil quality was correlated to its corresponding FFA and PV values using multivariate analysis. Researchers have used the multivariate analysis such as partial least square-ordinary least square (PLS-OLS) for FTIR data correlated to those obtained from conventional analytical results such as oil viscosity, total polar compound, FFA value, and PV to assess the quality of frying oil. This analysis has been applied to various food samples during frying, including french fries (Kalogianni *et al.,* 2011), falafel (Al-Degs *et al.,* 2011), and *Catla catla* fish (Manral *et al.,* 2008). Analysis using FTIR also ensures rapid measurement which takes less than 5 min per sample (Flatten *et al.,* 2004). The objective of this research was to examine the quality of palm oil during catfish (*Clarias gariepinus*) frying by FTIR.

MATERIALS AND METHODS

Materials

Two batches of commercial refined palm oil with iodine value 59 g/100 g from the same palm oil producer and the same batch of the oil production were used as the samples**.** Catfish was purchased from local market in Bogor, West Java, Indonesia. All the reagents used were of analytical grade.

Frying Experiments

To prepare the samples for frying, the catfish was marinated for 30 min in a spice mix (10% w/w of fresh catfish) consisting of 25% spice blend (mainly ginger, curcuma and coriander) from local market, 25% salt, and 50% vinegar with 7.5% of concentration (v/v). For each frying, 1 kg of catfish and 9 kg of palm oil were used. The frying was carried out in a deep fryer. The cat fish frying was repeated for nine times without any topping up or disposal of the frying oil after each batch of frying. The temperature was controlled at 180°C with a temperature controller and checked by a thermometer at the same temperature. The frying time was 15 min. The interval between fryings was 15 min. This is to mimic the practical procedure in household. The oils from first, third, fifth, seventh, and ninth fryings were then taken as samples. The fresh oil before frying was used as a oil standard quality. Samples were then analysed titrimetically to determine their FFA and PV values. Samples for FTIR analysis were centrifuged at 3000 rpm for 20 min to precipitate the residues prior to analysis. The residues were then discarded. The FFA value analysis was performed using AOCS Ca 5a-40 method, while the PV analysis was performed using AOCS Cd 8-53 method. Composition of fatty acids composition of palm oil before and after frying experiment was determined by GC-FID (Shimadzu, Japan) according to AOAC Method using BF3-methanol for sample derivatisation and margaric acid as an internal standard for fatty acid quantification.

FTIR Spectroscopy Analysis

The FTIR instrument used was IR-Prestige 21 from Shimadzu Corporation, Japan. This instrument was equipped with high sensitivity DLAGTS detector, KBr disk, and IR solution software for data interpretation.

The disk oil samples were prepared as follows: before starting this measurement, the background spectrum callibration was always performed (*Figure 1*). The oil sample was dropped on the KBr disk and then closed with another KBr disk to form a KBr disk sandwich. The sample was then measured at wavenumber between 400-4000 cm^{-1} at resolution 1.9. Before the measurement, the KBr disk was cleaned and rinsed with pure n-hexane and then dried with lens tissue to ensure that no residues remained on the disk (Al Degs *et al.,* 2011).

Multivariate Statistical Analysis

Prior to the multivariate analysis calculation, the data of absorbance intensity obtained from FTIR measurement was converted to percentage of absorbance intensity with this equation:

$$\%\ IA_x = \frac{IAx}{IA\ total}$$

where: %IAx = percentage of absorbance intensity at certain wavenumber.
IAx = absorbance intensity at certain wavenumber.
IA total = total of absorbance intensity.

This analysis was done with XLSTAT 2011 software from Microsoft Excel Series. The tools used was OLS (Ordinary Least Square) analysis.

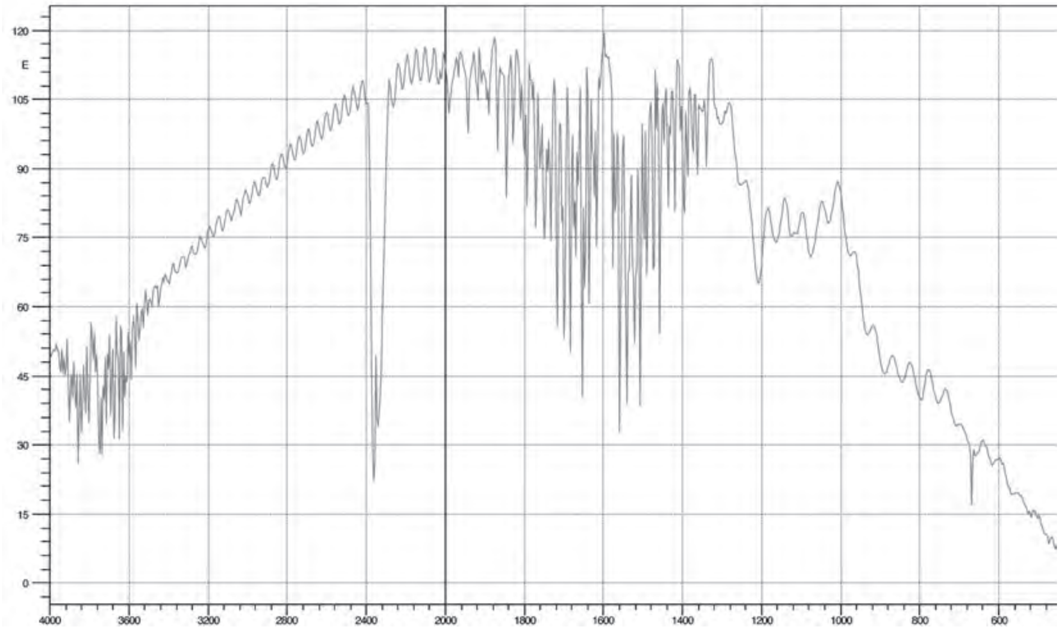

Figure 1. Baseline spectra of KBr disk for calibration used in Fourier Transform Infra-red Spectroscopy (FTIR) measurement of refined palm oil quality after fish frying activites.

RESULTS AND DISCUSSION

FTIR Profile

The FTIR absorbance spectrum was measured for 24 samples within the wavenumbers between 400-4000 cm^{-1}. This region is similar to the region chosen by other researchers (Vlachos *et al.,* 2006; Al Degs *et al.,* 2011). The FTIR absorbance spectrum is depicted in *Figure 2* which shows that certain wavenumbers contribute to specific functional groups. Referring to Guillen dan Cabo (1997), Al-Degs *et al.* (2011), Proctor *et al.* (1996), Lerma-Garcia *et al.* (2011), Guillen and Cabo (2002), and Navarra *et al.* (2010), the FTIR peaks and wavenumbers which are shown here indicate the functional groups of alkene double bond (-C=C-) both *cis* and *trans*, C = O ester, C-H methyl, and secondary oxidation compounds. Some functional groups identified from their spectra are commonly found for vegetable oils such as ester, carboxylic acid, saturated and unsaturated hydrocarbon functional groups (*Appendix 1*).

Alkene (-C=C-) is a common functional group found on vegetable oil which has a high percentage of unsaturated fatty acids. The correponding wavenumbers for the functional group are 722 cm^{-1}, 872 cm^{-1}, 912.5 cm^{-1}, 966 cm^{-1}, 1654 cm^{-1}, 1418 cm^{-1}, 1402 cm^{-1} and 3005.54 cm^{-1} (Guillen and Cabo, 1997; Lerma-Garcia *et al.*, 2011). Consequently, Moreno *et al.* (1999) have used the wavenumber around 3006 cm^{-1} to determine the saturation level on vegetable oil.

Furthermore, the occurrence of wavenumber 966 cm^{-1} shows the existence of *trans* fatty acid in oils. This can be caused by the heating process during frying. The frying process can turn the *cis* unsaturated fatty acid into *trans* unsaturated fatty acid. It was proven that the increase in *trans* fatty acid was in accordance with the decrease in *cis* fatty acid (oleic fatty acid). The mechanism could be explained as follows: the oxidation of oleic fatty acid (C18:1 *cis*) will turn it into *trans* elaidic fatty acid, while the oxidation of linoleic fatty acid (C18:2) will produce a mixture conjugation between 9- and 13-hydroperoxyde diene which undergoes geometric isomerisation to form its isomeric *trans,* which is linoleidic *trans* fatty acid (C18:2 *trans*). The ester bond is mostly found in monoglycerides, diglycerides, and triglycerides. Palm oil mainly consists of triglycerides, approximately 95%, with a small percentage of mono- and diglycerides (Sundram, 2004; Basiron, 2005). A range of 7%-10% of their total triglycerides are saturated triglycerides, dominated by palmitic fatty acid. Generally, the sn-2 position of triglycerides is filled with unsaturated fatty acids. At this condition, more than 85% of unsaturated fatty acids form ester bond with glycerol. The wavenumbers 1032 cm^{-1}, 1091 cm^{-1}, 1130 cm^{-1}, and 1729 cm^{-1} contribute to the interaction of C – O in ester (Guillen and Cabo, 1997; Lerma-Garcia *et al.,* 2011; and Proctor *et al.,* 1996). These wavenumbers (*Figure 1*) are then used to determine the ester bond on the palm oil samples. The ester bond indicates that the fatty acid is still linked with glycerol.

The wavenumber 2974 cm^{-1} contributes to the C-H bond (Al-Degs *et al.,* 2011) which is mostly found in hydrocarbons. The wavenumber 3474.91

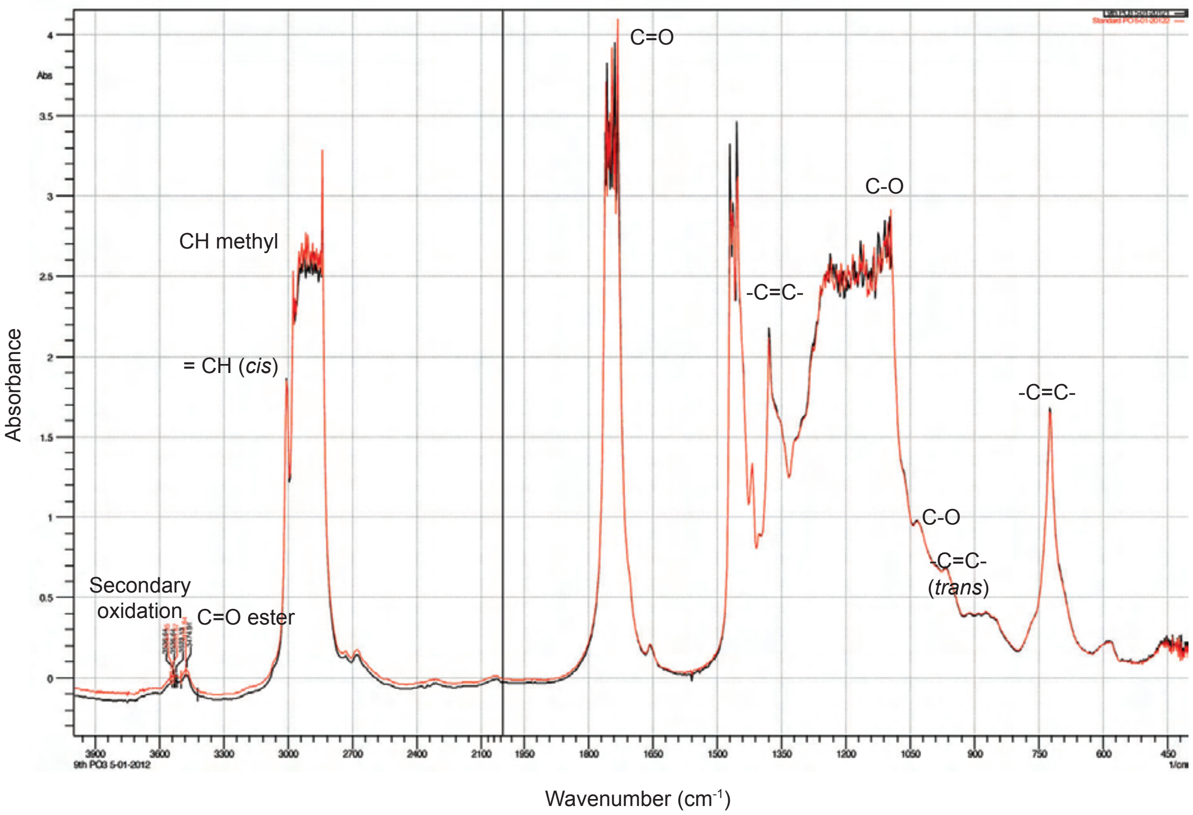

Figure 2. Fourier Transform Infra-red Spectroscopy (FTIR) spectra profile of refined palm oil before (black line) and after (red line) fish frying experiment (in duplicate) at 15 min frying time and 15 min duration between frying using fryer with a temperature controller set at 180°C.

cm^{-1} shows the interaction of C = O ester (Guillen and Cabo, 1997). This ester indicates the presence of triglycerides. Lastly, the detection of wavenumber 3536 cm^{-1} indicates the presence of secondary oxidation compounds such as alcohol, aldehyde, and ketone (Guillen and Cabo, 2002; Navarra *et al.*, 2010).

FFA Value Profile and Correlation with FTIR Profile

The FFA profile obtained during the short-term frying process showed a decreasing trend up to third frying times (*Figure 3*). This result is in contrast to other results reported by earlier researchers. Abdulkarim *et al.* (2007) and Tyagi and Vasishta (1996) have found that the FFA value increased with the repeated use of the frying oil. According to Lalas (2009), the FFA value tended to increase in accordance with the increasing frying time. The existing water in the food sample will react with triglyceride in the oil and will cause the hydrolytic reaction. This reaction will break the ester bond on the triglyceride and produce FFA and glycerol. However, sometimes the decrease of FFA value may occur during frying as shown in this study, perhaps due to short time frying.

These different results could be due to the differences in total time of frying and time interval between frying of samples. Tyagi and Vasishta (1996) used 6 hr interval time for each sampling with a total frying time of 70 hr. Abdulkarim *et al.* (2007) did the sampling every 6 hr and with a total frying time of 30 hr. In contrast, in our study we used much shorter sampling and frying time in order to mimic the cooking habit in household and food vendors. Sampling interval was every 15 min with the total frying time of only 2.25 hr. Consequently, these differences in sampling interval and duration of frying produced the different trend in FFA values reported in this article. Some earlier researchers reported decreasing trends in the FFA values during frying (Kress-Rolgers *et al.*, 1990; Manral *et al.*, 2008; Kalapathy and Proctor, 2000). Kress-Rolgers *et al.* (1990) used sampling time interval of 4 min each with the total frying time of 13.5 hr and carried out the first sampling after 30 min of frying. Manral *et al.* (2008) fried the sample with a total of 14 hr, with the interval between frying time of 6 min, and took the first sample after 2 hr of frying. Kalapathy and Proctor (2000) removed their first sample after 40 min of frying. These three groups of researchers, using shorter frying time and shorter sampling interval time, have shown a decreasing trend of FFA value (*Appendix 2*).

The decrease in FFA value is caused by the oxidation reaction of the FFA. The formation of peroxide can help in the oxidation of a small number

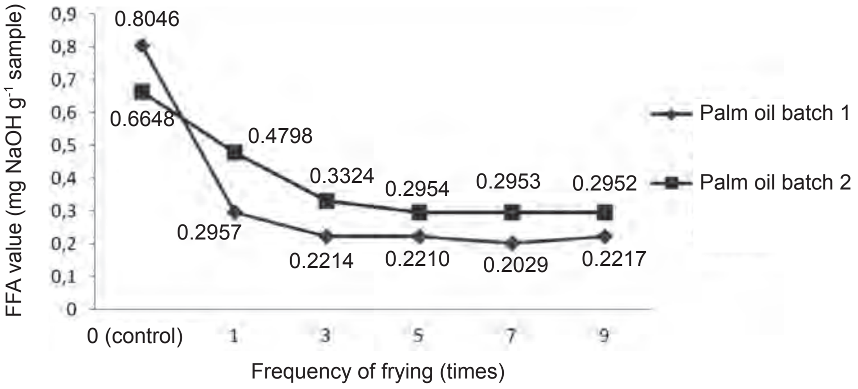

Figure 3. Free fatty acid (FFA) value profile of refined palm oil after fish frying experiment at 15 min frying time and 15 min duration between frying using fryer with a temperature controller set at 180°C.

of FFA. This reaction occurs by the interaction between FFA and oxygen which is triggered by the high temperature during frying. The oxidation reaction on this FFA is faster than the glycerol bonded fatty acid (Velasco *et al.,* 2009). During the early phase of frying, the rate of this oxidation reaction is faster than that of the hydrolytic reaction. Therefore, the FFA value shows a decreasing trend on the first stage of frying.

Palm oil contains unsaturated fatty acid such as oleic acid (C18:1) and linoleic acid (C18:2). The oleic acid composition reaches 38.7% and the linoleic acid composition reaches 10.5% of the total fatty acid (Rival, 2010). As such, as much as 49.2% of total fatty acid in palm oil consists of unsaturated fatty acid which is vulnerable to oxidation reaction. Moreover, the oxidised FFA may undergo another series of reactions. The oxidised fatty acid can make a bond with protein functional group and produce carboxyl compound. This carboxyl compound is an insoluble macromolecule which is hard to be detected (Pokorny, 1999). The composition of commercial palm oil fatty acids before and after frying used in this study is presented in *Table 1*.

The OLS method is used to find the correlation between spectrum profile and the FFA value. The selection of wavenumbers that are highly correlated with FFA values is based on the fittest model with a high degree of R^2 and the most significant *P* number (Pr>F) in the ANOVA. The 14 wavenumbers that meet these criteria are 722 cm^{-1}, 872 cm^{-1}, 912.5 cm^{-1}, 1032 cm^{-1}, 1091 cm^{-1}, 1130 cm^{-1}, 1400.5 cm^{-1}, 1418 cm^{-1}, 1654 cm^{-1}, 1729 cm^{-1}, 2974.36 cm^{-1}, 3005.54 cm^{-1}, 3474.91 cm^{-1}, and 3530 cm^{-1}. These wavenumbers are related to the functional groups alkene and ester. Alkene can be found in unsaturated FFA while ester is correlated inversely with FFA since it indicates the glycerol bonded FFA. The more the ester the lesser is the FFA, and *vice versa*.

Several previous researchers have used different wavenumbers. Al-Degs *et al.* (2011) has used wavenumbers between 1109.1-1240.2 cm^{-1}, 1703.1-1724.4 cm^{-1}, 1749.4 cm^{-1}, and 2837.3 cm^{-1}, while Che Man and Setyowati (1998) have used wavenumbers between 1662-1728 cm^{-1} and Lanser *et al.* (1991) have used wavenumbers between 1600-2000 cm^{-1}. The difference in the wavenumbers is due to the differences in frying oil sampling of each research. Al Degs *et al.* (2011) has taken the sample for every three days of frying. Che Man and Setyowaty (1998) have used oleic fatty acid as standard.

From the calculation of OLS with multivariate analysis, the correlation between percentage of absorbance of the main wavenumbers and FFA value is shown in this formula:

FFA value = -161.34 + 0.74x(%A722 cm^{-1})+ 7.02x(%A872 cm^{-1})+ 3.50x(%A912.5 cm^{-1})+ 1.11x(%A 1091 cm^{-1})+ 2.30x(%A1130 cm^{-1})+ 1.44x(%A 1400.5 cm^{-1})+ 1.49x(%A 1418 cm^{-1})+ 1.01x(%A 1.654 cm^{-1}) + 1.59x(%A 1729 cm^{-1}) + 1.9x(%A 2974.36 cm^{-1}) + 1.09x(%A 3005.54 cm^{-1}) + 4.25x (%A 3474.91 cm^{-1}) -2.14x(%A3530 cm^{-1}).

This formula has coefficient of correlation (*R*) of 0.977 and *P* number (Pr>F) of 0.042 at significance level 95%. The *P* number which is less than 0.05 shows that there is a significance relationship between the percentage of absorbance of main wavenumbers and FFA value. This formula can only be applied under the condition of frying 10 litres of palm oil with 1 kg of catfish for each frying at 180°C and maximum frying time of 2.25 hr. The plot of OLS predicted FFA values and actual FFA value is shown on *Figure 4*.

Peroxide Value Profile and Correlation with FTIR Profile

From *Figure 5*, it can be seen that the PV increased on the first frying and started to decline after reaching its maximum at the third frying. According to Chatzilazarou *et al.* (2006) and Tsaknis *et al.* (1998), the PV will increase during the first stage of frying. Later on, this number will gradually decrease at prolonged frying at 180°C due to the decomposition of peroxide into secondary oxidation compound.

TABLE 1. COMPOSITION OF FATTY ACIDS OF REFINED PALM OIL BEFORE AND AFTER FISH FRYING EXPERIMENT

Type of fatty acid	Before Frying		After Frying	
	mg g^{-1}	% Area relative	mg g^{-1}	% Area relative
Saturated Fatty Acid				
C8:0	183.72	0.03	616.13	0.06
C10:0	175.93	0.02	193.77	0.02
C12:0	1 804.94	0.25	1 927.53	0.18
C13:0	0	0	0	0
C14:0	9 900.8	1.39	10 270.81	0.99
C15:0	454.42	0.06	192.45	0.02
C16:0	1 614.54	22.63	4 194.92	40.24
C18:0	81 550.48	11.43	95 082.58	9.12
C20:0	2 351.91	0.33	3 877.27	0.37
C22:0	195.41	0.03	218.53	0.02
Total	258 071.6	36.18	531 871.1	51.02
Monounsaturated Fatty Acid				
C14:1	0	0	0	0
C16:1	100 222.4	14.05	2 048.59	0.2
C18:1	232 252.7	32.56	372 743.2	35.76
C20:1	0	0	1 830.99	0.18
C22:1	0	0	1 207.16	0.12
Total	332 475.2	46.61	3 778.30	36.25
Polyunsaturated Fatty Acid				
C18:2	120 030.6	16.83	129 331.9	12.41
C18:3	2 798.05	0.39	3 393.82	0.33
Total	122 828.6	17.22	132 725.7	12.73

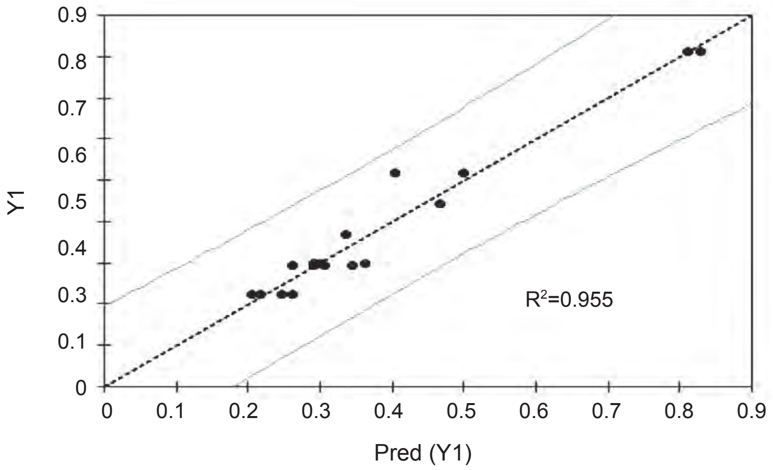

Figure 4. Plot of ordinary least square (OLS) predicted free fatty acid (FFA) value with actual FFA value of palm oil after fish frying experiment. Dot indicates the plot between Y1 and Pred(Y1). Line indicates the ideal curve when R^2=1.

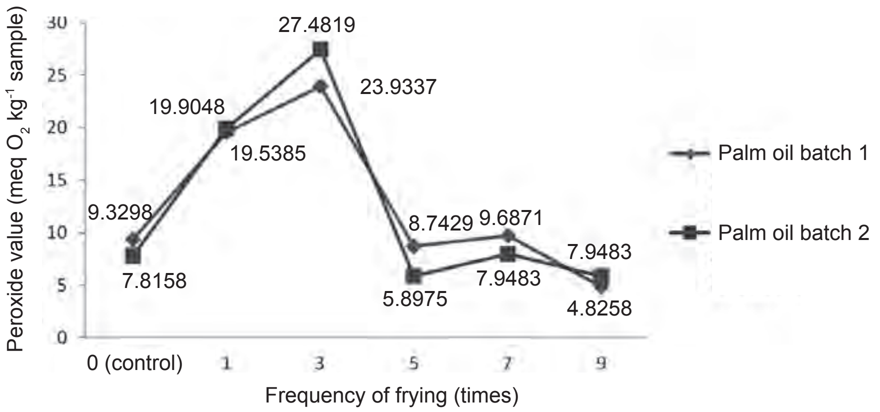

Figure 5. Peroxide value profile of palm oil after fish frying experiment at 15 min frying time and 15 min duration between frying using fryer with a temperature controller set at 180°C.

The OLS method was also used to find the correlation between spectrum profile and the PV. The selection of the wavenumbers that were highly correlated with the PV was based on the same criteria as in the FFA values. The 13 wavenumbers that meet the criteria are 722 cm^{-1}, 872 cm^{-1}, 912.5 cm^{-1}, 966 cm^{-1}, 1091 cm^{-1}, 1130 cm^{-1}, 1400.5 cm^{-1}, 1418 cm^{-1}, 1654 cm^{-1}, 1729 cm^{-1}, 2974.36 cm^{-1}, 3005.54 cm^{-1}, and 3474.91 cm^{-1}. These wavenumbers belong to carbonyl and alkene functional groups which are mostly affected by oxidation reaction. According to Lerma-Garcia *et al.*, (2011), the functional group of = C – H (*trans* and *cis*), ester C – O, and double bond $=CH_2$ have been detected by FTIR spectroscopy measurement and these groups are vulnerable to oxidation reaction.

Russin *et al.* (2004) correlated wavenumbers and PV using region 3444 cm^{-1}, 2854 cm^{-1}, 1100-1270 cm^{-1}, and 460-660 cm^{-1}. Guillen and Cabo (2002) used wavenumbers 3470 cm^{-1}, 3006 cm^{-1}, 1238 cm^{-1}, 1746 cm^{-1}, 1728 cm^{-1}, 1163 cm^{-1}, and 1118 cm^{-1}. The different wavenumbers chosen are likely because of the differences in the treatment and the samples used. Russin *et al.* (2004) used a mixture of canola, sunflower, and virgin coconut oil. Guillen and Cabo (2002) used sunflower oil.

Figure 6 shows the plot of OLS predicted PV and the actual PV. From the calculation of OLS with multivariate analysis, the correlation between percentage of absorbance of the main wavenumbers and PV is shown in this formula:

$$PV = 3284.74 - 37.89x(\%A\ 722\ cm^{-1}) + 136.08x(\%A\ 872\ cm^{-1}) - 62.67x(\%A\ 912.5\ cm^{-1}) - 183.24x(\%A966\ cm^{-1}) - 17.61x(\%A\ 1091\ cm^{-1}) - 32.70x(\%A1130\ cm^{-1}) - 27.97x(\%A1400.5\ cm^{-1}) - 53.34x(\%A1418\ cm^{-1}) + 30.66x(\%A\ 1654\ cm^{-1}) - 30.22x(\%A\ 1729\ cm^{-1}) - 40.09x(\%A\ 2974.36\ cm^{-1}) - 33.56x(\%A\,3005.54\ cm^{-1}) - 111.92x\ (\%A\,3474.91\ cm^{-1}).$$

This formula has coefficient of correlation (*R*) of 0.981 and *P* number (Pr>F) of 0.030 at significance level 95%. The *P* number which is less than 0.05 shows that there is a significance relationship between the percentage of absorbance of main wavenumbers and PV.

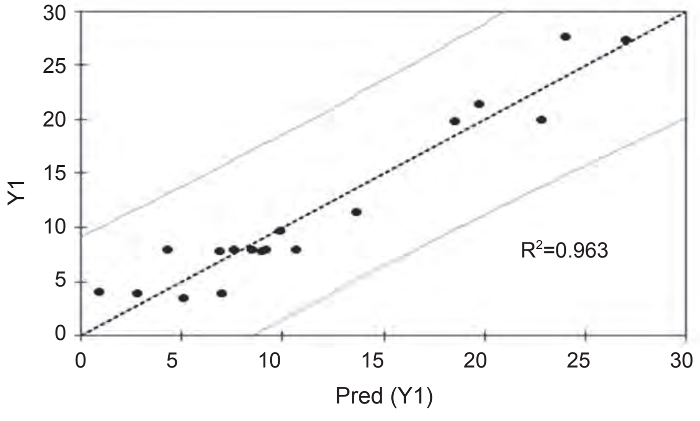

Figure 6. Plot of ordinary least square (OLS) predicted peroxide value [Pred (Y1)] with actual peroxide value (Y1) of palm oil after fish frying experiment. Dot indicates the plot between Y1 and Pred (Y1). Line indicates the ideal curve when R^2=1.

CONCLUSION

There is a significant correlation between the analysis of palm oil by FTIR and that by the conventional methods for peroxide and FFA values. The correlation formula derived from this study can be used for a rapid analysis to determine the palm oil quality from a food processing such as in fish frying.

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Appendix 1

COMPARISON OF THE MAIN WAVENUMBERS OF FRYING OILS, ANALYSED BY FOURIER TRANSFORM INFRA-RED SPECTROSCOPY (FTIR)

Researcher	Type of frying oil	Wavenumber	Functional group
Che Man and Setyowaty (1998)	Palm oil	3 550	OH
		3 473	Triglyceride ester
		3 006	C = C
		2 900 – 2 800; 1 465 and 1 377	CH_3 and CH_2
		2 677	ester C = O
		723	C = O
Vlachos *et al.* (2006)	Olive oil	3 009; 2 925; 2 854; 1 377; 723	C = C
		2 962; 2 872 and 1 654	CH_3
		1 746	ester C = O
		1 700	Free fatty acid
		1 465	CH_3 and CH_2
		1 418 and 1 397	*cis* C = C
		1 238 and 1 163	C = O in ester
Mossoba *et al.* (2007)	Hydrogenated soyabean oil	966	*trans* C = C
Rohman A *et al*. (2010)	Virgin coconut oil	2 954 and 1 377	CH_3
		2 924; 2 852 and 1 465	CH_2
		1 743	ester C = O
		1 417 and 721	*cis* C = C
		1 228 and 1 155	C – O
		962	*trans* C = C
		872	C = C
Al Degs *et al.* (2011)	Palm oil	3 491.2	OH in carboxylic
		3 005.1; 2 974.1; 2 837.1; 1 452.4; 1 379.1	C – H
		and 1 234.6	C – O and C=O
		1 762.9 and 1 753.2; 1 192.2 and 1 118.7	C = O
		721.4	
Hocevar *et al*. (2011)	Soyabean oil, palm oil and hydrogenated oil	2 915 and 2 845	C – H
		1 741	C = O in ester
		1 154	C – O and CH_2
Samples	Palm oil	722; 872; 912.5; 1 650; 1 402; 1 418 and 3 005.54	*cis* C = C
		966	*trans* C = C
		1 032; 1 091; 1 130 and 1 729	C – O in ester
		2 974	C – H
		3 474.91	C = O in ester
		3 536	Secondary oxidation product (alcohol, aldehyde, keton)

Appendix 2

COMPARISON OF FREE FATTY ACID (FFA) VALUES OF VEGETABLE OILS AFTER FRYING FROM DIFFERENT RESEARCHERS

Researcher	Type of frying oil	Time per frying (min)	Total frying time (hr)	Time of first sample taken (hr)	FFA value of control sample (% oleic acid)	FFA value of first sample (% oleic acid)	Interval (% oleic acid)	Trend
Tyagi and Vasishta (1996)	Vanaspati oil	30	70	6.00	0.12	0.25	0.13	Increasing
Abdulkarim *et al.* (2007)	*Moringa olifeira* oil	3	30	6.00	0.19	0.25	0.06	Increasing
Manral *et al.* (2008)	Sunflower oil	6	14	2.00	0.50	0.10	0.40	Decreasing
Kress-Rogers *et al.* (1990)	Partially hydrogenated vegetable oil	4	13.5	0.50	0.20	0.10	0.10	Decreasing
Kalapathy and Proctor (1996)	Soyabean oil	10	0.66	0.66	0.81	0.80	0.01	Decreasing
Sampels	Palm oil	15	2.25	0.25	0.57	0.21	0.36	Decreasing