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Application News

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Halal Authentication Analysis / IRTracer-100

Quantitative Determination of Lard Adulteration by FTIR Spectroscopy with Chemometrics Method - Vegetable Palm Oil

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❑ Introduction

Food adulteration is a persistent problem which could occur either accidentally or intentionally. Among the food products, edible oil is the most prone to adulteration [1] and this poses a major concern in terms of economical and religious point of view. For example, the Islamic law prohibits Muslims from consuming pork in any form, including lard in food products [2]. Therefore, it is necessary to develop analytical techniques to identify and quantify lard adulterated edible oil.

Fourier Transform Infrared (FTIR) spectroscopy is an effective technique to differentiate fats and oils as different compounds have unique fingerprint region in the infrared (IR) region. FTIR spectroscopy in combination with chemometrics data analysis such as multi-linear regression (MLR) or partial least squares (PLS) regression is a fast and simultaneous quantitative analysis of multi-component. In this application news, we introduce a method for quantitative determination of lard adulterated vegetable palm oil using FTIR spectroscopy and PLS quantitative calibration model.

❑ Experimental

Pork lard and commercially available palm oil were purchased from local markets. The lard was extracted based on the procedure by Rohman and Che Man [3]. Adipose tissue of pork was cut into small pieces and heated at 90-100°C for 2 hours. The melted fat was strained through sieve cloth and dried by addition of anhydrous Na_2SO_4. The extracted fat was centrifuged at 3000 rpm and for 20 minutes at 30°C. The fat layer was decanted and centrifuged again, followed by filtering through filter paper to remove solid residue.

A set of 13 standards containing 1-90% (w/w) lard in palm oil was prepared. A PLS calibration model for lard was established with 11 of these standards using LabSolutions IR workstation with Chemometrics PLS function. The remaining 2 standards were used as samples for quantitative determination.

The sample was measured with horizontal type attenuated total reflection attachment (HATR) with zinc selenide (ZnSe) prism. Each sample was measured 3 times. The IR spectra were acquired in the wavenumber range from 4000 cm^{-1} to 650 cm^{-1}. The measurement conditions are shown in Table 1.

Table 1: Instruments and Analytical Conditions

Instruments	: IRTracer-100, ATR-8200H (ZnSe)
Resolution	: 4.0 cm^{-1}
Accumulation	: 45
Apodization	: Happ-Genzel
Detector	: DLATGS

❑ Results and Discussion

Figure 1 shows the IR spectra of palm oil, lard and 50% w/w lard in palm oil. The IR spectra for palm oil and lard were quite similar. This is due to the similar chemical compositions between palm oil and lard. A closer examination of the IR region in the range of 1500-1000 cm^{-1} revealed slight differences in terms of peak intensities ratio at around 1160 cm^{-1}, 1117 cm^{-1} and 1097 cm^{-1} as marked by arrows in the overlay spectrum of palm oil and lard (Figure 2).

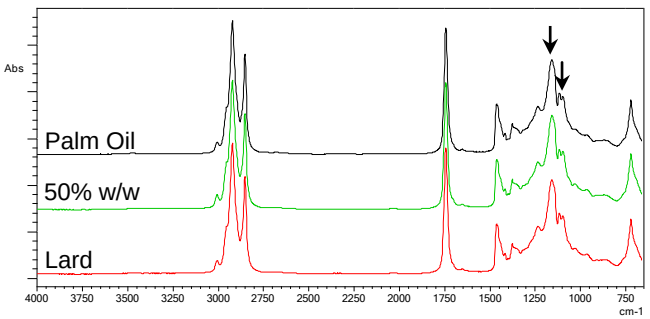

Figure 1: IR spectra of palm oil, lard and 50% w/w lard in palm oil.

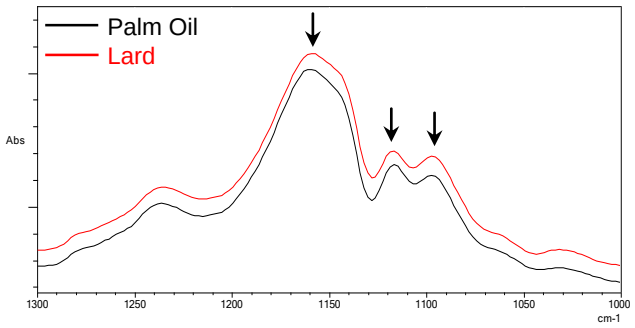

Figure 2: Overlay spectrum of lard and palm oil

Second derivative spectra were used in the PLS data analysis for better resolution of overlapping and shoulder peaks. Table 2 and Figure 3 show the PLS calibration parameter and result of lard in palm oil. A good square correlation coefficients of more than 0.999 was obtained for the PLS calibration modelling with low Mean Squared Error of Prediction (MSEP) and Standard Error of Prediction (SEP) as shown in Table 2.

Table 2: PLS calibration parameters of lard in palm oil

Calibration Table	
Algorithm	PLS I
Number of references	33 (three measurement per sample)
Range (cm^{-1})	1000 – 1490
Pre-process	Derivative, Order = 2, Points = 15
Scale	Autoscale
Number of factors	5
Square of correlation coefficient (R^2)	0.9993
MSEP	0.0007
SEP	0.0258

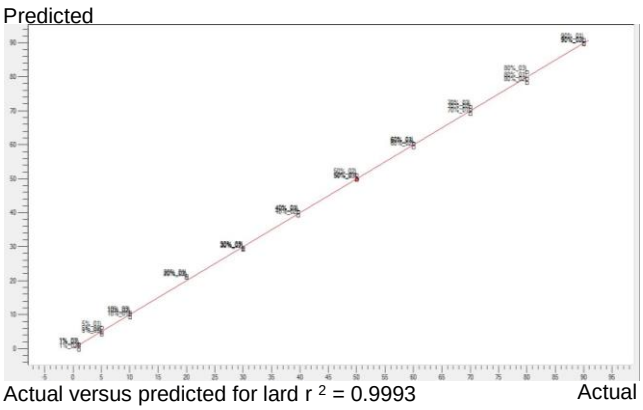

Figure 3: PLS calibration for lard predicted versus actual values.

Table 3 shows the quantitation results of lard in various types of edible oils by PLS method. The measurement results are within ±10% of the expected values for lard in palm oil. For lard in olive oil and palm soy oil, the mean predicted values differed greatly from the expected values. This is due to the difference in sample matrix of the edible oils.

Table 3: Predicted values of lard in different edible oils

Edible Oil		Brand A Palm Oil		Brand B Palm Oil		Olive Oil	Palm Soy Oil
Expected Value (% w/w)		8.0	25.0	8.0	25.0	25.0	25.0
Predicted Value (% w/w)	**1**	8.90	24.08	9.06	26.1	64.41	32.32
	2	8.24	23.54	7.60	25.82	64.56	32.13
	3	8.60	24.43	8.36	26.18	62.95	33.66
	Mean	8.58	24.02	8.34	26.03	63.97	32.70
Recovery (%)		107.2	96.1	104.2	104.1	255.9	130.8

❑ Conclusions

FTIR spectroscopy in combination with PLS data analysis is a rapid technique which has potential in determination of lard adulteration in palm oil without excessive sample pre-treatment. In this application news, a percentage of verification sample within ±10 % of the expected value was obtained. For lard adulteration in other types of edible oils, respective PLS calibration models have to be prepared to account for the difference in sample matrix.

❑ References

1. Johnson, R. (2014). Food Fraud and economically motivated adulteration of food and food ingredients. Congressional Research Services R43358, 1–40.
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3. Rohman, A. and Che Man, Y. B. (2009). Analysis of cod-liver oil adulteration using FTIR spectroscopy. Journal of the American Oil Chemists' Society 86: 1149–1153.

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