

**DETECTION OF LARD IN SELECTED FOOD MODEL SYSTEMS USING
FOURIER TRANSFORM INFRARED SPECTROSCOPY**

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ESPECIALLY DEDICATED TO MY BELOVED FAMILY

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfillment of the requirement for the degree of Master of Science

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The determination of food authenticity and the detection of adulteration are major issues in the food industries, and are of concern among consumers. In some countries, the food manufacturers choose to blend vegetable oil with lard to reduce production cost because lard is the cheapest fat commonly available for food industries. Pork and lard are serious matters in view of some religions, biological complications and health risks associated with daily intake. Therefore, a rigorous method is urgently needed in order to detect the presence of lard in food.

Fourier Transform Infrared (FTIR) spectroscopy has been widely used in many food authentication studies such as in coffee, extra virgin olive oil, jam, and fruit purees. It is a rapid analytical technique that measures the vibrations of bonds within their functional

groups. In this study, rapid methods using FTIR techniques were developed to detect and quantify the level of lard adulteration in selected food model systems.

In the first study, FTIR spectroscopy in combination with attenuated total reflectance (ATR) and Partial Least Square (PLS) regression was used to detect the presence of lard in chocolate formulation. A semi quantitative approach is proposed to measure the percent of lard in blends on the basis of spectral data at frequency region $4000-650\text{cm}^{-1}$. A high correlation of $R^2=0.9872$ was obtained with a standard error (SE) of 1.305.

In this second study, detection of lard in cake formulation was conducted. The lard was added to the shortening in the cake recipe at 0-100% level. FTIR spectra were recorded using ATR cell. A chemometric PLS regression was used to derive FTIR spectroscopic calibration model in regions of $1117-1097\text{cm}^{-1}$ and $990-950\text{cm}^{-1}$. For full cross validation, the R^2 obtained was 0.9937 with standard error (SE) of 2.257. The result was compared to a test set validation; which gave slightly lower R^2 value but better SE value (SE= 1.752).

In the third study, the same FTIR technique was used to detect the presence of lard in biscuits. A linear plot ($R^2=0.9974$) was obtained with SEC of 2.819 using calibration model, developed using region $3500-2900\text{cm}^{-1}$, $1780-1700\text{cm}^{-1}$, and $1500-800\text{cm}^{-1}$. The high correlation obtained indicated a good accuracy, reflecting a close relationship between actual and FTIR predicted value.

From these studies, the potential of FTIR spectroscopy as a rapid analytical tool for the quantitative determination of lard in selected food model systems were demonstrated. The finding from this study will serve as a basis in developing a database for monitoring food authentication, especially for Halal authentication purposes.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Master Sains

**PENGESANAN LEMAK KHINZIR DI DALAM MODEL SISTEM MAKANAN
TERPILIH MENGGUNAKAN SPEKTROSKOPI FOURIER
TRANSFORM INFRARED**

Oleh

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Penentuan ketulenan makanan dan pengesanan pencampuran adalah isu utama di dalam industri makanan dan mendapat perhatian para pengguna. Di sesetengah negara, pihak pengeluar makanan memilih untuk mencampurkan minyak sayuran dengan lemak babi bagi mengurangkan kos pengeluaran kerana lemak babi adalah paling murah dan mudah diperolehi pada ketika ini. Daging dan lemak babi merupakan isu sensitif dan serius bagi sesetengah agama, terutamanya Islam, komplikasi biologi serta risiko kesihatan yang dikaitkan dengan pengambilan hariannya. Oleh itu, satu teknik berkesan adalah sangat diperlukan untuk mengesan kehadiran lemak babi di dalam makanan.

Spektroskopi Fourier Transform Infrared (FTIR) telah digunakan secara meluas dalam kajian ketulenan makanan seperti untuk kopi, minyak zaiton dara, jem serta puri buah.

Spektroskopi FTIR adalah teknik analitikal pantas yang mengukur getaran ikatan di dalam kumpulan berfungsi mereka. Kaedah pantas menggunakan teknik FTIR telah di reka untk mengesan dan menentukan tahap pencampuran lemak babi di dalam model sistem makanan yang terpilih.

Spektroskopi FTIR, digabungkan dengan Attenuated Total Reflectance (ATR) dan persamaan 'Partial Least Squares (PLS)' telah digunakan untuk mengesan kehadiran lemak babi di dalam formulasi coklat serta hasilnya. Pendekatan semi kuantitatif telah dicadangkan untuk menentukan peratus lemak babi di dalam campuran berdasarkan data spektra pada frekuensi $4000-650\text{cm}^{-1}$. Korelasi yang baik ($R^2= 0.9872$) telah di capai dengan nilai sisihan (SE) bersamaan 1.305.

Bagi kajian kedua, pengesanan pencampuran lemak babi di dalam formulasi kek telah dijalankan. Lemak babi telah di tambah kepada 'shortening' di dalam resepi kek dalam julat 0-100%. Spektra FTIR telah direkod dengan menggunakan sel ATR. Pendekatan kemometrik PLS telah digunakan untuk membentuk model kalibrasi di dalam julat $1117-1097\text{cm}^{-1}$ dan $990-950\text{cm}^{-1}$. Bagi pengesanan lintang, R^2 yang diperolehi adalah 0.9937 dengan nilai sisihan (SE) 2.257. Keputusan ini telah dibandingkan dengan pengesanan set ujian, yang telah memberikan nilai R^2 yang lebih rendah tetapi menunjukkan nilai sisihan yang lebih baik (SE= 1.752).

Di dalam kajian ketiga, teknik FTIR yang sama telah digunakan untuk mengesan kehadiran lemak babi di dalam biskut. Satu plot linear ($R^2= 0.9974$) telah dicapai dengan nilai SEC iaitu 2.819, menggunakan model kalibrasi yang dibentuk pada julat 3500-2900 cm^{-1} , 1780-1700 cm^{-1} , dan 1500-800 cm^{-1} . Nilai korelasi yang tinggi menggambarkan ketepatan yang baik, sekaligus menunjukkan hubungan yang rapat antara nilai sebenar dan nilai anggaran menggunakan spektroskopi FTIR.

Daripada kajian ini, potensi spektroskopi FTIR sebagai alatan analitikal pantas untuk penentuan kuantitatif pencampuran lemak babi didalam model makanan terpilih telah ditunjukkan. Hasil daripada kajian ini akan menjadi asas bagi pembentukan pangkalan data untuk mengawal pengesahan makanan, terutamanya bagi tujuan pengesahan makanan Halal.

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I certify that an Examination Committee has met on 7th March 2006 to conduct the final examination of Syahariza binti Zainul Abidin on her Master of Science thesis entitled “Detection of Lard in Selected Food Model Systems using Fourier Transform Infrared Spectroscopy” in accordance with Univesiti Pertanian Malaysia (Higher Degree) Act 1980 and Universiti Pertanian Malaysia (Higher Degree) Regulations 1981. The Committee recommends that the candidate be awarded the relevant degree. Members of the Examination Committee are as follows:

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DECLARATION

I hereby declare that the thesis is based on my original work except for quotations and citations which have been duly acknowledged. I also declare that it has not been previously or concurrently submitted for any other degree at UPM or other institutions.

SYAHARIZA BINTI ZAINUL ABIDIN

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