UNIVERSITI PUTRA MALAYSIA

DETECTION OF LARD ADULTERATION IN SELECTED EDIBLE OILS USING GAS CHROMATOGRAPHY MASS SPECTROMETRY AND NIR SPECTROSCOPY

MUTIA NURULHUSNA BINTI HUSSAIN

FPV 2022 8

DETECTION OF LARD ADULTERATION IN SELECTED EDIBLE OILS USING GAS CHROMATOGRAPHY MASS SPECTROMETRY AND NIR SPECTROSCOPY

By

MUTIA NURULHUSNA BINTI HUSSAIN

Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfilment of the Requirements for the Degree of Master of Science

October 2020

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

DETECTION OF LARD ADULTERATION IN SELECTED EDIBLE OILS USING GAS CHROMATOGRAPHY MASS SPECTROMETRY AND NIR SPECTROSCOPY

By

MUTIA NURULHUSNA BINTI HUSSAIN

October 2020

Chair : Prof. Jamilah Bakar, PhD
Faculty : Food Science and Technology

Adulteration of food products has become a common problem in many countries. Adulteration may take the form of substitution of one species for another, where the food products from one species have been mixed intentionally with either a similar substitute material or a cheaper species. However, the use of pork and lard is a serious matter in Islam because foods containing ingredients from pig sources are haram (unlawful or prohibited) for Muslims to consume. Therefore, a reliable technique for the detection of lard adulteration in food products is necessary to protect Muslim consumers from intentional or unintentional fraud. The objective of this study was to apply near-infrared (NIR) spectroscopy combined with certain chemometric techniques for the detection and quantification of lard adulteration in selected edible oils. An analysis of fatty acids of authentic oils (i.e., palm olein (PO), soybean oil (SB), and canola oil (CO)) due to lard adulteration can complement the NIR spectral measurements. The presence of lard adulteration in PO, SB, and CO was analysed using NIR spectroscopy combined with soft independent modelling class analogy (SIMCA) and partial least-squares (PLS) in the region of 1,350–2,450 nm. This method can discriminate between pure and adulterated samples. The results revealed that the models predicted the adulterants with error limits of ± 0.83, ± 1.67, and ± 0.99 weight/weight for PO, SB, and CO, respectively. The PCA-developed models were able to classify lard-adulterated edible oil mixtures with almost 100% certainty. The adulterants were quantified using their respective PLS models within the same error limits as mentioned above. Furthermore, the study was extended for the classification of food product systems, specifically in biscuit formulation to verify the ability of the proposed method to classify between biscuits containing lard and without lard in its formulation. The spectra from the NIR analysis showed three significant peaks in the region of interest: 1,400–1,500 nm, 1,600–1,700 nm, and 2,100–2,200 nm. In this case, the result is similar to a previous study using edible oils and lard as

samples. Using the same established model, the samples of adulterated biscuits and biscuits without lard were 100% correctly classified. A chemometric PLS method was used to derive a NIR calibration model. However, the model used previously could not predict the validation data, resulting in a high value of root mean square error of prediction (RMSEP). Therefore, several limitations of the established model and recommendations have been discussed. In conclusion, this study shows that the utilisation of NIR spectroscopy combined with suitable chemometric techniques can provide a reliable analytical tool for detecting pork and lard adulteration. The findings from this study can serve as a basis of reference for the research in halal food authentication.

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

PENGESANAN PENCEMARAN LEMAK BABI DI DALAM MINYAK MASAK TERPILIH MENGGUNAKAN KROMATOGRAFI GAS SPEKTROSKOPI JISIM DAN SPEKTROSKOPI NIR

Oleh

MUTIA NURULHUSNA BINTI HUSSAIN

Oktober 2020

Pengerusi : Prof. Jamilah Bakar, PhD
Fakulti : Sains dan Teknologi Makanan

Pencemaran produk makanan menjadi masalah bersama di kebanyakan negara. Pencemaran boleh mengambil bentuk penggantian satu spesies yang lain di mana produk makanan dari satu spesies telah dicampur dengan sengaja dengan bahan pengganti yang serupa atau spesies yang lebih murah. Dalam kebanyakan kes, pengeluar makanan sering memilih lemak babi sebagai bahan pengganti minyak kerana ia murah dan mudah didapati. Walau bagaimanapun, penggunaan babi dan lemak babi adalah perkara yang serius dalam Islam kerana makanan yang mengandungi bahan-bahan dari sumber babi adalah haram (haram atau dilarang) untuk dimakan oleh orang Islam. Oleh itu, teknik yang boleh dipercayai untuk mengesan daging babi dan lemak babi dalam produk makanan adalah perlu untuk melindungi pengguna Islam dari penipuan yang disengajakan atau tidak sengaja. Objektif kajian ini adalah untuk menggunakan spektroskopi Near-inframerah (NIR) yang digabungkan dengan teknik kimometrik tertentu untuk pengesanan dan kuantifikasi pelarut lemak babi dengan minyak makan terpilih. Analisis perubahan asid lemak minyak asli olein sawit, minyak soya dan minyak kanola disebabkan oleh pemalsuan amalan lemak babi boleh melengkapkan pengukuran spektrum NIR. Kehadiran LD yang bercampur dengan olein sawit (PO), minyak kacang soya (SB) dan minyak kanola (CO) dianalisis menggunakan spektroskopi NIR yang digabungkan dengan analogy kelas pemodelan (SIMCA) dan “partial least square” (PLS) di kawasan 1350 nm-2450 nm. Kaedah ini membolehkan mendiskriminasi sampel yang tulen dan dipalsukan. Hasil kajian menunjukkan model yang meramalkan campuran yang terlibat dalam PO, SB dan CO dengan had kesalahan masing-masing ±0.83, ±1.67, and ±0.99 berat/berat. Model pembangunan PCA dapat mengklasifikasikan campuran lemak babi dan minyak masak dengan hampir 100%. Pengukuran percampuran dilakukan dengan menggunakan model PLS masing-masing dalam had kesalahan yang sama seperti yang disebutkan

diatas. Tambahan pula, kajian itu diperluaskan untuk klasifikasi sistem produk makanan, khususnya dalam formulasi biskut untuk mengesahkan keupayaan kaedah yang dicadangkan untuk mengklasifikasikan antara biskut yang mengandungi lemak babi dan tanpa lemak dalam formulasinya. Spektrum dari NIR menunjukkan tiga puncak penting di kawasan kepentingan, 1400-1500nm, 1600-1700nm dan 2100-2200 nm. Dalam kes ini, hasilnya sama dengan kes sebelumnya apabila kita menggunakan minyak dan lemak babi yang boleh dimakan sebagai sampel. Menggunakan model yang sama ditubuhkan, sampel biskut yang bercampur dan tidak bercampur dengan lemak 100% diklasifikasikan dengan betul. Kaedah kimometrik PLS digunakan untuk memperolehi model kalibrasi NIR. Walaubagaimanapun, model yang digunakan dalam bab sebelumnya tidak dapat meramalkan data pengesahan yang menghasilkan nilai RMSEP yang tinggi. Kesimpulannya, kajian ini memperlihatkan bahawa spektroskopi NIR dengan gabungan teknik chemometrics yang sesuai dapat memberikan alat analisa yang dapat digunakan dalam mengesan daging babi dan lemak babi yang dipamerkan. Penggunaan NIR juga ditunjukkan sebagai pengenalpastian adulterasi lemak babi yang boleh dipercayai. Penemuan dari kajian ini boleh menjadi asas rujukan untuk penyelidikan dalam pengesahan makanan halal.

ACKNOWLEDGEMENTS

I would like to express my most profound and sincere appreciation to my supervisor, Prof. Dr. Jamilah Bakar from the Department of Food Technology, Faculty of Food Science And Technology for her guidance, advice, encouragement and understanding. Her cooperation and support are always highly appreciated. My appreciation also goes to my co-supervisors, Prof. Dr. Shuhaimi Mustafa, Prof Madya Ir Dr Mohd Fared bin Abdul Khir and Pn.Syariena Arshad for their encouragement, opinion, comments and valuable moral support.

A special thanks to MIMOS Berhad and the Ministry of Science, Technology, and Innovations (MOSTI), Malaysia for providing funding support through the research Grant (Science Fund 06-03-04-SF0065). The encouragement and support are very much appreciated. I would also like to thank lab assistants of Halal Research Products Institute UPM and staffs of MIMOS Berhad for their help throughout the course of this project.

I would like to take this opportunity to express my warmest gratitude to my mother, Pn Jhani Ab. Rahaman and Pn Siti Rahmah Yaacob, my husband, Muhammad Safuan Bin Shaharum, my beloved children, Nur Diyana Yusra and Muhammad Daniel Yusya' and all my family members for their encouragement, support and love. Last but not least, I would also like to thank my friends and labmates for their kindness.

This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfilment of the requirement for the degree of Master of Sciences. The members of the Supervisory Committee were as follows:

Jamilah binti Bakar, PhD
Professor
Faculty of Food Science and Technology
Universiti Putra Malaysia
(Chairman)

Shuhaimi bin Mustapha, PhD
Professor
Faculty of Biotechnology and Biomolecular Science
Universiti Putra Malaysia
(Member)

Syariena bt Arshad
Research Officer
Halal Products Research Institute
Universiti Putra Malaysia
(Member)

Mohd Fared bin Abdul Khir, PhD
Associate Professor
Faculty of Science and Technology
Universiti Sains Islam Malaysia
(Member)

_____
ZALILAH MOHD SHARIFF, PhD
Professor and Dean
School of Graduate Studies
Universiti Putra Malaysia

Date: 10 February 2022

Declaration by graduate student

I hereby confirm that:

- this thesis is my original work;
- quotations, illustrations and citations have been duly referenced;
- this thesis has not been submitted previously or concurrently for any other degree at any other institutions;
- intellectual property from the thesis and copyright of thesis are fully-owned by Universiti Putra Malaysia, as according to the Universiti Putra Malaysia (Research) Rules 2012;
- written permission must be obtained from supervisor and the office of Deputy Vice-Chancellor (Research and Innovation) before thesis is published (in the form of written, printed or in electronic form) including books, journals, modules, proceedings, popular writings, seminar papers, manuscripts, posters, reports, lecture notes, learning modules or any other materials as stated in the Universiti Putra Malaysia (Research) Rules 2012;
- there is no plagiarism or data falsification/fabrication in the thesis, and scholarly integrity is upheld as according to the Universiti Putra Malaysia (Graduate Studies) Rules 2003 (Revision 2012-2013) and the Universiti Putra Malaysia (Research) Rules 2012. The thesis has undergone plagiarism detection software.

Signature: _____ Date: _____

Name and Matric No.: Mutia Nurulhusna Binti Hussain

Declaration by Members of Supervisory Committee

This is to confirm that:

- the research conducted and the writing of this thesis was under our supervision;
- supervision responsibilities as stated in the Universiti Putra Malaysia (Graduate Studies) Rules 2003 (Revision 2012-2013) are adhered to.

Signature: _____
Name of Chairman of Supervisory Committee: _____

Signature: _____
Name of Member of Supervisory Committee: _____

Signature: _____
Name of Member of Supervisory Committee: _____

Signature: _____
Name of Member of Supervisory Committee: _____

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF ABBREVIATIONS

°C	degree celcius
ANOVA	analysis of variance
AOCS	American Oil Chemists' Society
c	Concentration
C10:0	Capric acid
C12:0	lauric acid
C14:0	myristic acid
C14:1	Myristoleic acid
C15:0	Pentadecanoic acid
C16:0	palmitic acid
C16:1	palmitoleic acid
C17:0	Margaric acid
C17:1	Margaroleic acid
C18:0	stearic acid
C18:1	oleic acid
C18:2	linoleic acid
C18:3	linolenic acid
C20:0	arachidic acid
C20:1	Gadoleic acid
C20:2	Eicosadienoic acid
C20:4	Eicosatetraenoic acid
C22:0	Docosanoic acid
C22:1	Erucic acid

cm	centimeter
CO	Canola Oil
Cv	cuvette
CVA	canonical variable analysis
DAG	Diacylglycerols
DSC	differential scanning calorimetry
ELISA	Enzyme-linked immunosorbent assay
EN	Electronic nose
EVM	evaluation model
FAME	fatty acid methyl ester
FTIR	Fourier Transform Infrared
g	Gram
GC	gas chromatograph
kg	kilogram
g/cm^2	gram per centimeter square
GC/MS	Gas chromatography Mass Spectrometry
GC-FID	Gas chromatography with flame ionization detector
HPLC	high performance liquid chromatography
InGaAs	Indium Gallium Arsenide
JAKIM	Department of Islamic Development Malaysia
l	length
LC/MS	liquid chromatography-mass spectrometry
LD	lard
LDA	Linear Discriminant Analysis
LOO	leave-one-out
LS	Light source

mL/min	milliliter/minute
mm	Milimeter
MPOB	Malaysian Palm Oil Board
MS	Mass spectrometer
N	Refractive Index
Na_2SO_4	anhydrous sodium sulphate
NIR	Near-Infrared
nm	Nano-meter
PC	Principal component
PCA	principle component analysis
PCR	Polymerase chain reaction
PO	palm olein
PUFA	polysaturated fatty acid
PUFA	polyunsaturated fatty acid
PV	Peroxide value
R^2	Coefficient of determination
RBD	refined bleach deodorized
RMSEP	root mean square error of prediction
rpm	revolution per minute
RT	Retention time
SBO	Soybean oil
SIMCA	Soft Independent Modeling of Class Analogy
TAG	triacylglycerol
TIC	total ion chromatograms
UKM	University Kebangsaan Malaysia
UPM	University Putra Malaysia

v/v	volume/volume
w/v	weight/volume
w/w	weight/weight
ε	absorptivity
µL	Microliter
µm	micrometer

CHAPTER 1

INTRODUCTION

1.1 Background of study

Vegetable oils and fats have a big contribution in our diet or in food products formulation. Fats and oils differ in composition, length, and unsaturation degree of the fatty acids, as well as their double-bond position in the chain. The vegetables oil and animal fats composed primarily from triacylglycerol (TAG), diacylglycerols (DAGs), free fatty acids and also varying amounts of minor components like phospholipids, sterols, tocopherols, carotenoids and fat soluble vitamins (O'Brien, 2009).

Over the past decades, vegetable oils have supplanted lard and beef tallow as the major source of dietary fat mostly due to consumers demanding food products that combine a pleasant flavor with nutritional benefits. On the basis of the expanding market for vegetables oils, the authentication become an important subject from both commercial and health perspective (Aparicio & Aparicio-Ruiz, 2000). Therefore, food authenticity issue including adulteration is a major issue in the food industry, and is causing concerns among costumers and food manufacturers.

One of the major issues concerning adulteration is where high value raw materials are substituted by cheaper materials. Lard is one of the pig derivatives which is added into the food products where food producers in some countries prefer to mix vegetable oils with lard to reduce the production cost (Mansor, Che Man, & Shuhaimi, 2011). The detection of food adulterants also important for religion concern where some religions like Islam, Judaism and Hinduism forbid the follower to consume any food containing lard and its derivatives (A. Rohman, Che Man, Hashim, & Ismail, 2011). Adulteration of haram or 'shubhah' ingredient like lard in food products has been widespread and difficult to identify with the naked eye (Fadzlillah et al., 2011). Thus, a sensitive, easily performable and reliable scientific method is required to verify the ingredients in Halal food product.

Several analytical methods either physical or chemical based methods have been successfully applied to detect and identify lard based ingredients adulteration. Technique such as analysis of fatty acid methyl ester (FAME) by gas chromatography (GC) is used widely in food analysis including checking for food adulteration (Lehotay & Hajslova, 2002). Furthermore, several techniques also have been reported such as high-performance liquid chromatographic

(HPLC) (Marikkar, Ghazali, Che Man, Peiris, & Lai, 2005), differential scanning calorimetry (DSC) (Mansor et al., 2011), and gas chromatography with flame ionization detector (GC-FID) (Dahimi, Hassan, Rahim, Abdulkarim, & A, 2014) as the analytical techniques for detection and quantification of pig derivatives in food systems.

The developments of rapid and reliable methods are required for the authentication and detection of adulteration in food products. Therefore, there have been several attempts to find a rapid and efficient method for detection of adulteration such as the use of infrared spectrometry both in the mid- and near-infrared regions. The mid-infrared (MIR) region where the fundamentals of functional groups appear has long been used for food analysis as reported by Che Man, Rohman, & Mansor (2010) and Xu, Cai, Cui, Ye, & Yu, (2012) using Fourier Transform Infrared (FTIR) Spectroscopy.

The potential of NIR spectrometry has been explored. There are a multitude of reviews on NIR spectroscopy however only few deal exclusively with its halal food authentication. A few works done by the researcher and food analyst in food industry using NIR spectroscopy method such as Christy, Kasemsumran, Yiping, & Ozaki (2004) has investigated the possibility of detecting adulteration in olive oil containing different adulterants, and for quality authentication of meat product by Prieto, Roehe, Lavín, Batten, & Andrés (2009). These study focused on the investigation of the possibility of the NIR technique for detecting non-halal adulterant specifically in the case of lard in several oil mixtures as an attractive alternative technique due to straightforward and speedy characterization of samples.

NIR spectra generally contain a number of broad and overlapping bands. The nature of these bands make quantitative analysis difficult. Therefore, this problem overcome by development of methods by chemometrics analysis such as Partial Least Square (PLS), Linear Discriminant Analysis (LDA), Soft Independent Modelling of Class Analogy (SIMCA) and others applied to vibrational spectroscopic data to assess the adulteration and authentication (Nunes, 2014).

1.2 Problem statement

Research and development in halal food authentication are meant to find alternatives to recent non halal and doubtful ingredients. Several analytical methods are available but most are require sophisticated laboratory procedures, lack in terms of practicality and requires high capital cost (Kamruzzaman, Sun, ElMasry, & Allen, 2013). One such example is chromatography-based techniques and polymerase chain reaction (PCR). Previous studies on fats and oils using the FTIR spectroscopy combined with chemometrics can be alternative for a rapid and non-destructive technique. However, the requirement for a bulky detector set coming from the need to cover the mid-infrared range has prevented the use of FTIR spectroscopy system for on-site screening application. Therefore, a rapid and efficient method for detection of adulteration should be developed and a possible way out is to use the near-infrared (NIR) region spectroscopy combined with chemometrics analysis.

1.3 Scope of study

In this particular study, NIR spectroscopy was used to develop a rapid detection method coupled with chemometrics approach to detect the presence of lard in selected edible oils as a means for halal screening technique. Long wave NIR spectrometer in the region of 1350-2450 nm was used using transmittance measurement mode as the sample is of liquid type. This study focused on the lard adulteration. This work includes different types of vegetable oils (palm olein, canola and soybean oil) adulterated with lard. Furthermore, the change of fatty acid profiles in samples due to the addition of adulterants (lard) was also reported by in order to confirm the NIR spectroscopy results. Biscuit formulation serving as a model system for food products which contain fats and oils is used to verify the ability of NIR spectroscopy to detect lard in complex matrix.

1.4 Objectives

This study focuses on the possibility of Near-Infrared (NIR) spectroscopy combined with chemometrics analysis technique as a halal screening tool and develops this method particularly for the case of oil and lard mixture detection. Therefore, the specific objectives of the study were:

1. To analyse fatty acid composition of lard adulteration in selected edible oils using gas chromatography mass spectrometry (GC/MS)
2. To detect lard adulteration in selected edible oils for halal screening application using NIR spectroscopy and chemometrics analysis.
3. To validate lard adulteration in biscuit formulation using NIR spectroscopy.

1.5 Significance of study

Detecting adulteration in fats and oils especially the most widely used such as palm oil, soybean and canola oil are indeed needed. The attempt to find the effective, simple, straightforward analysis is essential. The NIR spectroscopy based screening system can be used for on-site application to identify food products that contains lard mixture. The findings from this experiment will be a valuable input in fine tuning the portable halal screening system which could later be adapted for other adulteration in fats and oil, and food products.

REFERENCES

A.N. Ahmad, R. Abdul Rahman, M. Othman, and U. F. Ungku Zainal Abidin, "Critical success factors affecting the implementation of halal food management systems: Perspective of halal executives, consultants and auditors," *Food Control*, vol. 74, pp. 70–78, 2016

Aenugu, H., & Kumar, D. (2011). Near Infra Red Spectroscopy- An Overview.*International Journal of ChemTech Research*, *3*(2), 825–836.

Ali, M. E., Kashif, M., Uddin, K., Hashim, U., Mustafa, S., & Che Man, Y. Bin. (2012). Species Authentication Methods in Foods and Feeds: the Present, Past, and Future of Halal Forensics. *Food Analytical Methods*, *5*(5), 935–955. doi:10.1007/s12161-011-9357-3

An Overview of the Current Analytical Methods for Halal Testing- Irwandi Jaswir, Muhammad Elwathig S. Mirghani, Hamzah M. Salleh, Noriah Ramli, Fitri Octavianti, Ridar Hendri

Aparicio, R., & Aparicio-Ruiz, R. (2000). Authentication of vegetable oils by chromatographic techniques. *Journal of Chromatography A*, *881*, 93–104.

Azmi, Aida Azrina (2007) *Detection Of Pork And Lard Adulteration In Food Products Using Molecular Biology Techniques For Halal Authentication.* Masters thesis, Universiti Putra Malaysia.

BERNAMA. (2015, February 5). Jakim appoints TPM Biotech as halal panel laboratory. *Daily Express.*

Blanco, M., & Pagès, J. (2002). Classification and quantitation of finishing oils by near infrared spectroscopy. *Analytica Chimica Acta*, *463*(2), 295–303. doi:10.1016/S0003-2670(02)00382-3

Boschetti, L., Ottavian, M., Facco, P., Barolo, M., Serva, L., Balzan, S., & Novelli, E. (2013). A correlative study on data from pork carcass and processed meat (Bauernspeck) for automatic estimation of chemical parameters by means of near-infrared spectroscopy. *Meat Science*, *95*(3), 621–8. doi:10.1016/j.meatsci.2013.06.001

C. E. Stauffer, *Fats and Oils*. St.Paul,Minnesota,USA: American Association of Cereal Chemist, Inc, 1996

Cen, H., & He, Y. (2007). Theory and application of near infrared reflectance spectroscopy in determination of food quality. *Trends in Food Science & Technology*, (18), 72–83.

Che Man, Y. B., Aida, a. a., Raha, a. R., & Son, R. (2007). Identification of pork derivatives in food products by species-specific polymerase chain

reaction (PCR) for halal verification. *Food Control*, 18(7), 885–889. doi:10.1016/j.foodcont.2006.05.004

Chou, C.-C., Lin, S.-P., Lee, K.-M., Hsu, C.-T., Vickroy, T. W., & Zen, J.-M. (2007). Fast differentiation of meats from fifteen animal species by liquid chromatography with electrochemical detection using copper nanoparticle plated electrodes. *Journal of Chromatography B*, *846*(1-2), 230–9. doi:10.1016/j.jchromb.2006.09.006

Christy, A. A., Kasemsumran, S., Yiping, D., & Ozaki, Y. (2004). The Detection and Quantification of Adulteration in Olive Oil by Near-Infrared Spectroscopy and Chemometrics. *Analytical Sciences*, *20*(June), 935–940.

chromatography mass spectrometer with headspace analyzer. Meat Science, 88(4), 638–44. doi:10.1016/j.meatsci.2011.02.022

Codex Alimentarius Commision, 2001

Colombo, Carlos & Berton, Luiz & Diaz, Brenda & Ferrari, Roseli. (2017). Macauba: A promising tropical palm for the production of vegetable oil. OCL. 25. 10.1051/ocl/2017038.

Cozzolino, D., & Murray, I. (2004). Identification of animal meat muscles by visible and near infrared reflectance spectroscopy. *LWT - Food Science and Technology*, *37*(4), 447–452. doi:10.1016/j.lwt.2003.10.013

Dahimi, O., Hassan, M. S., Rahim, A. A., Abdulkarim, S. M., & A, S. M. (2014). Differentiation of Lard from Other Edible Fats by Gas Chromatography-Flame Ionisation Detector (GC-FID) and Chemometrics. *Journal of Food and Pharmaceutical Sciences*, *2*, 27–31.

Demirhan, Y., Ulca, P., & Senyuva, H. Z. (2012). Detection of porcine DNA in gelatine and gelatine-containing processed food products—Halal/Kosher authentication. *Meat Science*, *90*(3), 686–689. doi:10.1016/j.meatsci.2011.10.014

DLP450NIR Evaluation Module, http://www.ti.com (Accessed 2 Aug. 2016)

Donald A. Burns and Emil W. Ciurczak, Handbook of Near-Infrared Analysis, Third Edit. Boca Raton: Taylor & Francis Group, 2008.

Ekwu, F.C., Nwagu, A., 2004. Effect of processing on the quality of cashew nut oils. J. Sci. Agric. Food Tech. Environ. 4, 105–110

Fernández Pierna, J. A., Abbas, O., Lecler, B., Hogrel, P., Dardenne, P., & Baeten, V. (2014). NIR fingerprint screening for early control of non-conformity at feed mills. *Food Chemistry*. doi:10.1016/j.foodchem.2014.09.105

Global Vegetable Oil Market Professional Survey Report 2019, Publish Date : 02-Sep-2019, No.Of Pages : 126

H. N. Lubis, N. F. Mohd-Naim, N. N. Alizul, and M. U. Ahmed, “From market to food plate: Current trusted technology and innovations in halal food analysis,” Trends Food Sci. Technol., vol. 58, pp. 55–68, 2016.

Harwood John and Ramón Aparicio. 2000. Handbook of olive oil. Analysis and Properties. Gaithersburg: Aspen publications

Hourant, Pierre & Baeten, V. & Morales, Maria & Meurens, Marc & Aparicio, Ramón. (2000). Oil and Fat Classification by Selected Bands of Near-Infrared Spectroscopy. Applied Spectroscopy. 54. 1168-1174. 10.1366/0003702001950733.

J. Z. K. Khattak et al., “Concept of Halal food and biotechnology,” Adv. J. Food Sci. Technol., vol. 3, no. 5, pp. 385–389, 2011.

Jia Xie• Tianshun Liu• Yingxin Yu• Guoxin Song• Yaoming Hu. Rapid Detection and Quantification by GC–MS of Camellia Seed Oil Adulterated with Soybean Oil. 2013, J Am Oil Chem Soc (2013) 90:641–646. DOI 10.1007/s11746-013-2209-0

Jirasubkunakorn, W., Bell, A.E., Gordon, M.H. and Smith, K.W., (2007). Effects of variation in the palm stearin: Palm olein ratio on the crystallisation of a low-trans shortening, *Food Chemistry, 103,* 477-485.

Jorfi, R., Mustafa, S., Man, Y. B. C., Hashim, D. B. M., & Awis, Q. (2012). Differentiation of pork from beef, chicken, mutton and chevon according to their primary amino acids content for halal authentication. *African Journal of Biotechnology*, *11*(32), 8160–8166. doi:10.5897/AJB11.3777

Kamruzzaman, M., Sun, D.-W., ElMasry, G., & Allen, P. (2013). Fast detection and visualization of minced lamb meat adulteration using NIR hyperspectral imaging and multivariate image analysis. *Talanta*, *103*, 130–6. doi:10.1016/j.talanta.2012.10.020

Kesmen, Z., Sahin, F., & Yetim, H. (2007). PCR assay for the identification of animal species in cooked sausages. *Meat Science*, *77*(4), 649–53. doi:10.1016/j.meatsci.2007.05.018

Kuswandi, B., Irmawati, T., Hidayat, M. A., Jayus, & Ahmad, M. (2014). A Simple Visual Ethanol Biosensor Based on Alcohol Oxidase Immobilized Onto Polyaniline Film for Halal Verification of Fermented Beverage Samples. *Sensors (Basel, Switzerland)*, *14*(2), 2135–49. doi:10.3390/s140202135

Larissa Rocha dos Santos(1), Marcela de Souza Zangirolami,Núbia Oliveira Silva, Patrícia Valderrama and Paulo Henrique Março, Rapid non-invasive assessment of quality parameters in ground soybean using near-infrared spectroscopy, (2018) , v.53, n.1, p.97-104, Jan. 2018. DOI: 10.1590/S0100-204X2018000100011

Lehotay, S. J., & Hajslova, J. (2002). Application of gas chromatography in food analysis. *Trends in Analytical Chemistry*, *21*(02), 686–697.

Lima, Emerson. (2002). High-performance liquid chromatography of fatty acids in biological samples. Analytica Chimica Acta. 465. 81-91. 10.1016/S0003-2670(02)00206-4.

Lukitaningsih, E., Sa, M., & Rohman, A. (2012). Quantitative analysis of lard in cosmetic lotion formulation using FTIR spectroscopy and partial least square calibration. *Journal of the American Oil Chemists' Society*, *89*, 1–15.

Macedo-silva, A., Barbosa, S. F. C., Alkmin, M. G. A., Vaz, A. J., & Shimokomaki, M. (2000). Hamburger meat identification by dot-ELISA. *Meat Science*, *56*, 189–192.

Manley, D. (2000). Technology of Biscuits, Crackers and Cookies, CRC Press. USA. pp 130-150.

Mansor, T. S. T., Che Man, Y. B., & Shuhaimi, M. (2011). Employment of Differential Scanning Calorimetry in Detecting Lard Adulteration in Virgin Coconut Oil. *Journal of the American Oil Chemists' Society*, *89*(3), 485–496. doi:10.1007/s11746-011-1936-3

Marikkar, J. M. N., Ghazali, H. M., Che Man, Y. B., Peiris, T. S. G., & Lai, O. M. (2005). Distinguishing lard from other animal fats in admixtures of some vegetable oils using liquid chromatographic data coupled with multivariate data analysis. *Food Chemistry*, *91*(1), 5–14. doi:10.1016/j.foodchem.2004.01.080

Marikkar, J. M. N., Ghazali, H. M., Long, K., & Lai, O. M. (2003). Lard uptake and its detection in selected food products deep-fried in lard. *Food Research International*, *36*(9-10), 1047–1060. doi:10.1016/j.foodres.2003.08.003

Ng, Choo Lum, Randy L. Wehling, and S. L. C. (2007). Method for Determining Frying Oil Degradation by Near-Infrared Spectroscopy. *Journal of Agricultual and Food Chemistry*, (55), 593–597.

Nor Hayati, I., Yaakob, C.M., Tan, C.P. and Nor Aini, I., (2009). Physicochemical characteristics of soybean oil, palm kernel olein, and their binary blends, International Journal of Food Science and Technology, 44, 152–161.

Nunes, C. A. (2014). Vibrational spectroscopy and chemometrics to assess authenticity, adulteration and intrinsic quality parameters of edible oils and fats. *Food Research International*, *60*, 255–261. doi:10.1016/j.foodres.2013.08.041

Nurjuliana, M., Che Man, Y. B., Mat Hashim, D., & Mohamed, a K. S. (2011). Rapid identification of pork for halal authentication using the electronic nose and gas

O. Mba, P. Adewale, M. J. Dumont, and M. Ngadi, “Application of near-infrared spectroscopy to characterize binary blends of palm and canola oils,” Ind. Crops Prod., vol. 61, pp. 472–478, 2014.

O’Brien, R. D. (2009). *Fats and Oils: Formulating and Processing for Applications*. (3rd ed, Ed.) (pp. 43–46). New York: Taylor & Francis.

Pocketbook Of Oil Palm Uses / Coordinators Tang Thin Sue, T.P. Pantzar-Is - Seventh Edition 2016 ISBN 978-967-961-228-8 1. Oil palm--Malaysia. 2. Oil palm--Economic aspects--Malaysia. I. Tang, Thin Sue. II.Pantzaris, T.P. 633.85109595

Prieto, N., Roehe, R., Lavín, P., Batten, G., & Andrés, S. (2009). Application of near infrared reflectance spectroscopy to predict meat and meat products quality: A review. *Meat Science*, *83*(2), 175–86. doi:10.1016/j.meatsci.2009.04.016

R. Shaik, “Analytical Tool for Rapid Analysis of Edible Oil,” Aalborg University Esbjerg, 2008.

Rohman, A., & Che Man, Y. B. (2012). Analysis of Pig Derivatives for Halal Authentication Studies. *Food Reviews International*, *28*(1), 97–112. doi:10.1080/87559129.2011.595862

Rohman, A., Gupitasari, I., Triyana, K., Salleh, A., Ahmad, S., & Ahmad, S. (2014). Quantification of Lard in the Mixture with Olive Oil in Cream Cosmetics Based on FTIR Spectra and Chemometrics for Halal Authentication. *Jurnal Teknologi*, *1*, 113–119.

Rohman, A., Sismindari, Erwanto, Y., & Che Man, Y. B. (2011). Analysis of Pork Adulteration in Beef Meatball Using Fourier transform infrared (FTIR) Spectroscopy. *Meat Science*, *88*(1), 91–5. doi:10.1016/j.meatsci.2010.12.007

Rohman,A. and Che Man, Y. B. (2010). FTIR spectroscopy combined with chemometrics for analysis of lard in the mixtures with body fats of lamb , cow , and chicken. *International Food Research Journal*, *526*(17), 519–526.

S. Alang Mahat, “The Palm Oil Industry From The Perspective of Sustainable Development: A Case Study of Malaysian Palm Oil Industry,” Ritsumeikan Asia Pacific University Japan, 2012.

Soares, S., Amaral, J. S., Oliveira, M. B. P. P., & Mafra, I. (2013). A SYBR Green real-time PCR assay to detect and quantify pork meat in processed poultry meat products. *Meat Science*, *94*(1), 115–20. doi:10.1016/j.meatsci.2012.12.012

Study of physicochemical properties of edible oil and evaluation of frying oil quality by Fourier Transform-Infrared (FT-IR) Spectroscopy Erum Zahir *, Rehana Saeed, Mehwish Abdul Hameed, Anjum Yousuf, 2014 , Arabian Journal of Chemistry, http://dx.doi.org/10.1016/j.arabjc.2014.05.025 1878-5352

Syahariza, Z., Man, Y., Selamat, J., & Bakar, J. (2005). Detection of lard adulteration in cake formulation by Fourier transform infrared (FTIR) spectroscopy. *Food Chemistry*, *92*(2), 365–371. doi:10.1016/j.foodchem.2004.10.039

Third Industrial Master Plan (IMP3) 2006 – 2020, Ministry of International Trade and Industry (MITI), Malaysia

Ulberth, F. and M. Buchgraber 2000. Authenticity of fats and oils. European Journal of Lipid Science Technology 102:687–694.

Wolf, C., & Luthy, J. (2001). Quantitative competitive (QC) PCR for quantification of porcine DNA. *Meat Science*, *57*, 161–168.

Wood, J. D., Enser, M., Fisher, a V, Nute, G. R., Sheard, P. R., Richardson, R. I., … Whittington, F. M. (2008). Fat deposition, fatty acid composition and meat quality: A review. *Meat Science*, *78*(4), 343–58. doi:10.1016/j.meatsci.2007.07.019

Wu, Shubin & Lyu, Gaojin & Lou, Rui. (2012). Applications of Chromatography Hyphenated Techniques in the Field of Lignin Pyrolysis. 10.5772/32446.

X. Zhang et al., "Improvement of near infrared spectroscopic (NIRS) analysis of caffeine in roasted arabica coffee by variable selection method of stability competitive adaptive reweighted sampling (SCARS)," Spectrochim. Acta - Part A Mol. Biomol. Spectrosc., vol. 114, pp. 350–356, 2013.

Xu, L., Cai, C. B., Cui, H. F., Ye, Z. H., & Yu, X. P. (2012). Rapid Discrimination of Pork in Halal and Non-Halal Chinese Ham Sausages by Fourier transform infrared (FTIR) spectroscopy and chemometrics. *Meat Science*, *92*(4), 506–10. doi:10.1016/j.meatsci.2012.05.019

Xu, L., Cai, C. B., Cui, H. F., Ye, Z. H., & Yu, X. P. (2012). Rapid Discrimination of Pork in Halal and Non-Halal Chinese Ham Sausages by Fourier transform infrared (FTIR) spectroscopy and chemometrics. *Meat Science*, *92*(4), 506–10.doi:10.1016/j.meatsci.2012.05.019

Y. B. . Man, H. . Gan, I. Noraini, S. A. . Nazimah, and C. . Tan, "Detection of lard adulteration in RBD palm olein using an electronic nose," Food Chem., vol. 90, no. 4, pp. 829–835, 2005.

Yanty, N.A.M., Marikkar, J.M.N. and Abdulkarim, S. M. (2014). Determination of types of fat ingredient in some commercial biscuit formulations. *International Food Research Journal*, *21*(1), 277–282.