

EFFECTS OF HEATING ON LARD AS ADULTERANT IN
REFINED BLEACHED DEODORIZED PALM OIL USING
PHYSICOCHEMICAL TESTS AND GAS CHROMATOGRAPHY
HEADSPACE WITH CHEMOMETRICS

UNIVERSITI SAINS ISLAM MALAYSIA
جامعة العلوم الإسلامية
ISLAMIC SCIENCE UNIVERSITY OF MALAYSIA

AMAL HUSNA DINI SULAIMAN

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Amal Husna Binti Sulaiman

(Matric No: 3130131)

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AUTHOR DECLARATION

بِسْمِ اللَّهِ الرَّحْمَنِ الرَّحِيمِ

I hereby declare that the work in this thesis is my own except for quotations and summaries which have been duly acknowledged.

Date: 19/4/2016

Signature: 

Name: Amal Husna binti Sulaiman

Matric No: 3130131

Address: No 3, Jalan Melor 6, Taman Seri Melor, 43000, Kajang, Selangor

UNIVERSITI SAINS ISLAM MALAYSIA
جامعة العلوم الإسلامية
ISLAMIC SCIENCE UNIVERSITY OF MALAYSIA

BIODATA OF AUTHOR

Amal Husna binti Sulaiman (3130131) was born on the 4th November 1988. She is currently residing at No 3, Jalan Melor 6, Taman Seri Melor, 43000, Kajang, Selangor Darul Ehsan. She previously was a student of Universiti Putra Malaysia (UPM) from the Faculty of Food Science and Technology. She is at present a Master student of USIM in Food Biotechnology.



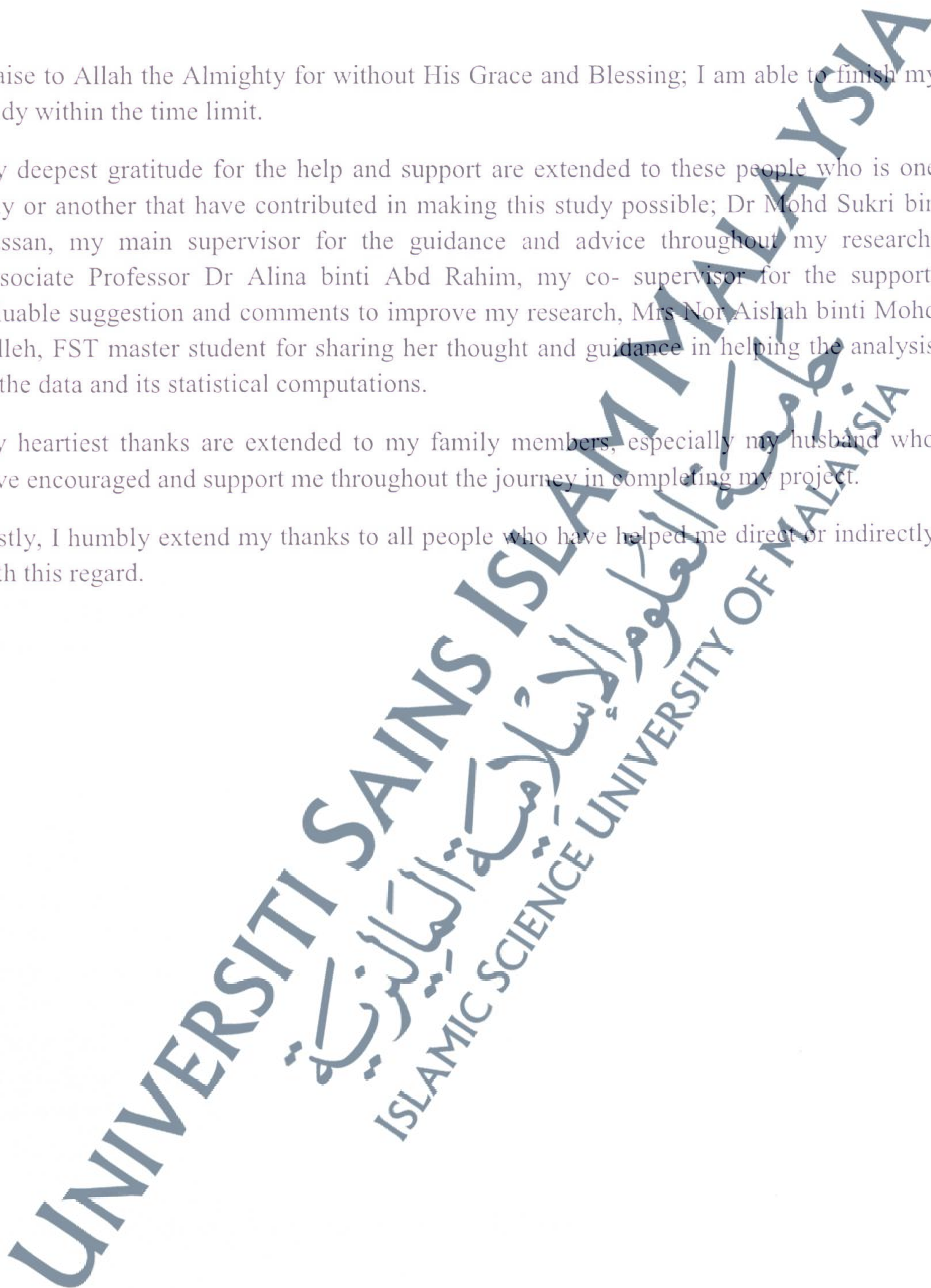
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ABSTRAK

Pengadukan telah mendapat perhatian lebih dalam industri lemak dan minyak kerana melibatkan isu agama. Kajian ini menilai kesan pemanasan ke atas profil lemak babi (15 % dan 30 % lemak babi) di dalam minyak kelapa sawit RBD (disaring, dinyahluntur dan dinyahbau) pada 120 °C, 180 °C dan 240 °C selama 1, 2 dan 3 jam masa pemanasan. Ujian fiziko kimia yang telah dilakukan adalah nilai iodin, nilai peroksida dan kepekatan warna. Bagi ujian nilai iodin, kesemua sampel menunjukkan penurunan nilai iodin apabila masa pemanasan ditingkatkan. Pada suhu yang rendah (120 °C), tidak banyak perubahan yang berlaku pada semua sampel. Apabila dipanaskan pada suhu yang tinggi, sampel 30 % lemak babi menunjukkan perubahan nilai iodin yang paling besar. Untuk nilai peroksida pada suhu 120 °C, semua sampel menunjukkan nilai peningkatan apabila masa pemanasan ditingkatkan. Walaubagaimanapun, apabila suhu semakin tinggi (180 °C dan 240 °C), nilai peroksida menjadi semakin rendah. Hal ini mungkin disebabkan oleh penghasilan dan pengumpulan produk teroksida sekunder. Bagi keputusan warna, apabila masa pemanasan ditingkatkan, sampel menjadi semakin gelap disebabkan penghasilan bahan tidak meruap. Apabila data dianalisis menggunakan PCA, kesemua sampel (15 % dan 30 % lemak babi) dan kawalan (0 % lemak babi) telah dikumpulkan mengikut masa pemanasan tetapi tiada perbezaan untuk sampel dan kawalan. Komponen meruap telah dikenalpasti menggunakan kaedah GC-MS-HS (gas kromatografi spektrometri headspace). Kepelbagaian data daripada GC-MS-HS telah di purata tengahkan untuk Analisis Komponen Utama (PCA) menggunakan perisian Unscrambler. Keputusan daripada plot skor untuk 30 % lemak babi dan 15 % lemak babi di dalam RBD minyak sawit menunjukkan corak yang sama untuk suhu pemanasan 120 °C dan 180 °C. Pada 240 °C, kedua-dua sampel dan kawalan bertaburan pada plot skor. Walaubagaimanapun, teknik GC-MS-HS tidak dapat membezakan antara 0 % lemak babi dengan 15 % dan 30 % lemak babi di dalam RBD minyak kelapa sawit. Bahan meruap didapati berkaitan dengan suhu pemanasan spesifik adalah heksanal pada 120 °C, nonanal, oktana dan heptana pada 180 °C. Keputusan daripada kajian ini mungkin dapat memberi maklumat untuk kaedah pengenalanpastian dan penyaringan lemak babi yang terdapat dalam minyak kitar semula dan peralatan yang lebih sensitif seperti SPME berkemungkinan memberikan hasil keputusan yang lebih baik.

Katakunci: Pengadukan, Kesan pemanasan, Profil lemak babi, PV, IV, Warna, GC-MS-HS, PCA

ABSTRACT

Adulteration has gained much concern in the oils and fats industry due to religious issues. This study assessed the effect of heating on the profiling of lard (15 % and 30 % lard) spiked in refined bleached deodorized (RBD) palm oil at three different temperatures (120 °C, 180 °C and 240 °C), each for 1, 2 and 3 hours of heating time. The physicochemical test done were iodine value (IV), peroxide value (PV) and color intensity. For iodine value test, all samples showed decreased in iodine value as heating time was prolonged. At low heating temperature (120 °C), not much changes were observed for all samples. When samples were heated at higher temperature, 30 % lard showed the biggest changes in iodine value. For peroxide value, at 120 °C, all samples showed an increased as the time was prolonged. However, at higher heating temperature (180 °C and 240 °C), the value decreased. This might be due to the formation and accumulation of secondary oxidation products. In terms of colour, as time was prolonged, all samples becomes darker due to formation of non volatile decomposition. When data of physicochemical tests were analyzed using PCA, all samples (15 and 30 % lard) and control (0 % lard) were clustered according to the heating temperature but showed no difference for samples and control. The volatile compounds released were identified using (gas chromatography mass spectrometry headspace) GC-MS-HS method. Multivariate data from GC-MS-HS were mean centred prior to Principal Components Analysis (PCA) using Unscrambler software. The result obtained from the scores plot for 30 % lard and 15 % lard in the RBD palm oil showed the same pattern for heating temperature at 120 °C and 180 °C. Both samples and control were scattered in the scores plot at 240 °C. However, the GC-MS-HS technique was not able to differentiate between 0 % lard with 15 % or 30 % lard in RBD palm oil. Certain volatile compounds were found related to spesific heating temperature such as hexanal at 120 °C, while nonanal, heptane and octane at 180°C. The result of this study might be used to provide information for the method designed in the detection and screening of lard in recycled cooking oil and a more sensitive equipment such as SPME might give a better results.

Keywords: Adulteration, Heating effect, Lard profiling, PV, IV, Colour, GC-MS-HS, PCA

ملخص البحث

الغش تسبب في الكثير من القلق في صناعة الزيوت والدهون بسبب القضايا الصحية والدينية. في هذه الدراسة نقوم بتقييم تأثير التسخين على مواصفات شحم الخنزير (15% و 30% شحم الخنزير) ارتفعت في المكرر ابيض (RBD) لزيت النخيل في 120 درجة مئوية، 180 درجة مئوية و 240 درجة مئوية لمدة 1 و 2 و 3 ساعات. استخدمت تقنية الفصل الكروماتوغرافي (GC-MS-HS) للتعرف على المركبات الطيارة. وكانت الاختبارات الفيزيائية لقيمة اليود، والرقم البيروكسيد (PV) وكثافة اللون (IV). لاختبار قيمة اليود، أظهرت جميع العينات انخفاض في قيمة اليود كلما كان وقت التسخين لفترة طويلة. في انخفاض درجة حرارة التسخين (120 درجة مئوية)، في حين لم يتم ملاحظة أي تغييرات كبيرة لجميع العينات في ارتفاع درجة الحرارة، قد لوحظ أكبر التغيرات في قيمة اليود والرقم البيروكسيد بنسبة 30% شحم الخنزير، في 120 درجة مئوية، وأظهرت جميع العينات انخفاض قيمة اليود والرقم البيروكسيد كلما كان وقت التسخين أطول (180 درجة مئوية و 240 درجة مئوية)، كلما كان وقت التسخين أطول تصبح العينات أكثر قتامة (أقل L * القيمة) هذا يرجع إلى كمية عالية من الأحماض الدهنية غير المشبعة في شحم الخنزير. وتشكيل النحل الغير متقلبة. عندما تم تحليل البيانات باستخدام PCA، لجميع العينات (15 و 30% شحم الخنزير) والتحكم (0% شحم الخنزير) تم التجميع حسب درجة حرارة التسخين ولم تظهر أي فروق للعينات و التحكم. قد تم شرح النتائج المتحصل عليها من GC-MS-HS باستخدام تحليل المكونات الرئيسية (PCA). أظهرت نتيجة تم الحصول عليها من مؤامرة درجات 30% شحم الخنزير وشحم الخنزير 15% في زيت النخيل RBD نفس النمط لدرجة الحرارة للتسخين في 120 درجة مئوية و 180 درجة مئوية. وتناثرت كل من العينات والتحكم في المؤامرة العشرات في 240 درجة مئوية. ومع ذلك، فإن تقنية GC-MS-HS غير قادرة على التفريق بين 0% شحم الخنزير مع 15% أو 30% شحم الخنزير في زيت النخيل RBD. تم العثور على مركبات طيارة معينة تتعلق درجة الحرارة للتحكم مثل hexanal في 120 درجة مئوية، في حين nonanal، هيبتان والأوكتان في 180°C. ونتيجة لهذه الدراسة يمكن أن تستخدم لتوفير المعلومات عن طريقة تصميم في الكشف والفحص من شحم الخنزير في زيت الطبخ المعاد تدويره والمعدات أكثر حساسية مثل SPME قد تعطي نتائج أفضل. كلمات المفتاحية: الغش، وتأثير التسخين، مواصفات شحم الخنزير، PV، IV، اللون، GC-MS-HS، PCA.

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ABBREVIATIONS

RBD	Refined Bleached Deodorized
CPO	Crude Palm Oil
ENL	Enshi No 1. Native Breed
DAG	Diacylglycerol
DSC	Differential Scanning Calorimetry
DTA	Differential Thermal Analysis
FA	Fatty Acids
FAME	Fatty Acid Methyl Esters
FFA	Free Fatty Acids
FDA	Food and Drug Administration
FID	Flame Ionization Detector
FTIR	Fourier Transform Infrared
GC	Gas Chromatography
GC-MS-HS	Gas Chromatography Mass Spectrometry Headspace
GC –TOF	Gas Chromatography Time of Flight
HPLC	High Performance Liquid Chromatography
IRMS	Isotope Radio Mass Spectrometry
IV	Iodine Value
MAG	Monoacylglycerols
MS	Mass Spectroscopy
MUFA	Mono unsaturated fatty acid

NIST	National Institute of Standard and Technology
NMR	Nuclear Magnetic Resonance
PCA	Principal Components Analysis
PKO	Palm Kernel Oil
PLS	Partial Least Square
POO	Palmitoolein
POS	Palmitooleostearin
POP	Palmitooleopalmitin
PUFA	Poly unsaturated Fatty Acid
RBD	Refined Bleached Deodorized
SBO	Soybean Oil
SPME	Solid Phase Microextraction
TAG	Triacylglycerol
TMA	Thermo Mechanical Analysis
TCL	Three way crossbred
TSyF	Total Synchronous Fluorescence
USDA	United States Department of Agriculture
UV	Ultraviolet
VCO	Virgin Coconut Oil