



UNIVERSITI PUTRA MALAYSIA

**APPLICATION OF FOURIER TRANSFORM INFRARED
SPECTROSCOPY FOR DETERMINING SOME
TRADING PARAMETERS OF PALM OIL**

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**APPLICATION OF FOURIER TRANSFORM INFRARED
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TRADING PARAMETERS OF PALM OIL**

By

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**Thesis Submitted in Fulfilment of the Requirements for the
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Especially dedicated to my beloved parents....



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TABLE OF CONTENTS

	Page
ACKNOWLEDGEMENTS.....	iii
LIST OF TABLES.....	ix
LIST OF FIGURES.....	xi
LIST OF PLATE.....	xiv
LIST OF ABBREVIATIONS.....	xv
ABSTRACT.....	xviii
ABSTRAK.....	xx
 CHAPTER	
I INTRODUCTION.....	1
II LITERATURE REVIEW.....	5
Palm Oil.....	5
Production.....	5
Palm Oil Trade.....	6
Chemical Properties and Characteristics of Palm Oil.....	7
Test Method for Palm Oil.....	12
Some Trading Parameters Relating to the Quality and Characteristics of Palm Oil.....	12
Fourier Transform Infrared Spectroscopy.....	19
Infrared Spectroscopy.....	19
Vibration of Molecules.....	20
Intensity of Infrared Bands.....	22
Representation of Spectra.....	22
The Advantages of FTIR Over Disper- sive Spectroscopy.....	23
Transmission Technique.....	28
Spectrum Interpretation.....	31
Multivariate Calibration Methods.....	35
FTIR in Food Analysis.....	49
Application of FTIR in Oils and Fats.....	50



III DETERMINATION OF IODINE VALUE OF PALM OIL BY FOURIER TRANSFORM INFRARED SPECTROSCOPY.....	67
Introduction.....	67
Materials.....	70
Methods.....	71
Instrumentation and Sample Handling..	71
Calibration and Validation.....	73
Results and Discussion.....	74
Spectral Analysis.....	74
Calibration Assessment.....	76
PLS Calibration and Validation.....	80
Summary.....	86
IV MULTIVARIATE CALIBRATION OF FOURIER TRANSFORM INFRARED SPECTRA IN DETERMINING IODINE VALUE OF PALM OIL PRODUCTS.....	87
Introduction.....	87
Materials.....	89
Methods.....	89
Instrumentation.....	89
Statistical Multivariate Methods.....	91
Results and Discussion.....	92
Baseline Correction.....	96
Partial Least Squares.....	98
Principle Component Regression.....	98
Summary.....	107
V DETERMINATION OF FREE FATTY ACID CONTENTS IN PALM OLEIN BY FOURIER TRANSFORM INFRARED SPECTROSCOPY	108
Introduction.....	108
Materials.....	110
Methods.....	112
Instrumentation.....	112
Spectra Collection.....	113



Calibration and Validation.....	113
Results and Discussion.....	114
Spectral Basis for Analysis.....	114
Statistical Analysis.....	117
Summary.....	123
VI QUANTITATIVE DETERMINATION OF PEROXIDE VALUE IN THERMALLY OXIDIZED PALM OLEIN BY FOURIER TRANSFORM INFRARED SPECTROSCOPY..	125
Introduction.....	125
Materials.....	127
Sample and Chemical.....	127
Reference Oil.....	127
Methods.....	128
Sample/Oxidation.....	128
Sample Handling.....	129
Calibration and Validation.....	130
Results and Discussion.....	131
Spectroscopy of Thermally Oxidized Palm Olein.....	131
Development of Calibration Model.....	134
Summary.....	143
VII DETERMINATION OF ANISIDINE VALUE IN THERMALLY OXIDIZED PALM OLEIN BY FOURIER TRANSFORM INFRARED SPECTROSCOPY.....	145
Introduction.....	145
Materials.....	147
Methods.....	148
Chemical Analyses.....	148
FTIR Scanning and Calibration.....	148
Model Validation.....	150
Results and Discussion.....	150
Anisidine Value Measured by the Reference Method.....	150
The Absorption Bands of Aldehydes.....	151
Selection of the Optimal Region for Anisidine Value Prediction.....	153



Expanded FTIR Calibration Model for Predicting Anisidine Value.....	158
Summary.....	162
VIII SUMMARY, CONCLUSION AND RECOMMENDATION.....	163
Summary.....	163
Conclusion and Recommendation.....	165
BIBLIOGRAPHY.....	167
APPENDIX.....	185
A Plate	186
BIOGRAPHICAL SKETCH.....	187



LIST OF TABLES

Table		Page
1	Fatty Acid (%) Composition of Palm Oil.....	8
2	Fatty Acid Composition of Processed Palm Oil and Palm Oil Fractions.....	9
3	Characteristics of Palm Oil.....	11
4	The Frequency Bands of Edible Oils.....	34
5	Actual Iodine Value for the RBD Palm Stearin, Superolein and Their Blends.....	71
6	Statistical Comparison of IV of RBD Palm Stearin, Superolein and Their Blends Obtained by AOCS Reference and FTIR Methods.....	83
7	Standard Error of Prediction (SEP), Number of Factor, Coefficient of Determination (R^2) and Accuracy of IV of Palm Oil Products Obtained by PLS and PCR Methods.....	105
8	SEP Ratios of Different Methods and F Critical at 95% Confidence Level.....	106
9	Mean Duplicate Readings of FFA Content (%) by AOCS Reference Method for the RBD Palm olein.....	111
10	Statistical Comparison of FFA Content (%) of RBD Palm Olein Obtained by AOCS Reference and FTIR Methods.....	122
11	Result of Mean Duplicate Analyses of RBD Palm Olein Obtained from AOCS Reference Method.....	137



12	Calibration and Validation Statistics for Anisidine Value Prediction by the AOCS Method.....	151
13	Effect of Different Wavelength Regions in Development the Calibration Model in RBD Palm Olein Samples.....	157
14	Statistical Comparison of Anisidine Value of Blends of Oxidized and Unoxidized RBD Palm Olein Obtained by AOCS Reference and FTIR Methods.....	160



LIST OF FIGURES

Figure		Page
1	(a) Glycerol Molecule; (b) Triglyceride Molecule.	7
2	Autoxidation Chain Reaction.....	14
3	A Generalized Mechanism for Hydroperoxide Formation.....	15
4	Schematic Diagram of the Essential Components of an Interferometer.....	26
5	FTIR Spectrometer.....	28
6	Semi Permanent Cell.....	29
7	Theoretical Illustration of Model Optimization..	49
8	A Typical Spectrum of Palm Stearin and Superolein Blends.....	75
9	Variance Spectrum Obtained from the Calibration Standards.....	77
10	Correlation Spectrum Obtained from the Calibration Standards.....	79
11	The Spectral Region Used for the Best Calibration.....	81
12	IV Calibration Plot Yielded from RBD Palm Stearin and Superolein Blends Calibration Standards.....	84
13	IV Validation Plot Yielded from RBD Palm Stearin and Superolein Blends Calibration Standards.....	85



14	Transmission/Fourier Transform Infrared Spectra of Palm Oil at 4000-750 cm^{-1}	94
15	Overlay Spectra of Palm Oil at Wavelength of 3025 to 2992 cm^{-1}	95
16	Predicted Versus Actual Iodine Values Calculated with PLS Calibration Method.....	99
17	Predicted Versus Actual Iodine Values Calculated with PLS Calibration Method Using Linear Removed Baseline Type.....	100
18	Predicted Versus Actual Iodine Values Calculated with PLS Calibration Method Using One Point Baseline Type at 3100 cm^{-1}	101
19	Predicted Versus Actual Iodine Values Calculated with PCR Calibration Method.....	102
20	Predicted Versus Actual Iodine Values Calculated with PCR Calibration Method Using Linear Removed Baseline Type	103
21	Predicted Versus Actual Iodine Values Calculated with PCR Calibration Method Using One Point Baseline Type at 3100 cm^{-1}	104
22	Fourier Transform Infrared Spectrum of the 4000-600 cm^{-1} Region of Oleic Acid-Spiked Palm Olein (Oleic Acid of 0.498%).....	115
23	The Variance Spectrum for the Calibration Samples.....	118
24	The Correlation Spectrum for the Calibration Samples.....	119



25	Calibration Plot of the Predicted Free Fatty Acids Versus the Chemical Free Fatty Acid Content	121
26	A Typical Spectrum of Oxidized Refined-Bleached-Deodorized Palm Olein.....	133
27	The Difference Spectrum Obtained by Ratioing the Single Beam Spectrum of the Oxidized Palm Olein against that of the Reference Palm Olein.....	135
28	Variance Spectrum Obtained from the Calibration Standards.....	138
29	Correlation Spectrum Obtained from the Calibration Standards.....	139
30	A Calibration Plot of Infrared Predicted PV Versus Chemically Determined PV for 30 RBD Oxidized and Unoxidized Palm Olein Blends....	140
31	A Cross-Validation Plot of Infrared Predicted PV Versus Chemically Determined PV for 30 RBD Oxidized and Unoxidized Palm Olein Blends.....	142
32	Mean Spectrum of Calibration Set.....	154
33	Correlation Spectrum at 2760-2600 cm^{-1} Obtained from the Calibration Standards.....	155
34	Correlation Spectrum at 1750-1600 cm^{-1} Obtained from the Calibration Standards.....	156
35	Anisidine Value Calibration Plot Yielded from Blends of Oxidized and Fresh Palm Olein.....	159
36	Anisidine Value Validation Plot Yielded from Blends of Oxidized and Fresh Palm Olein.....	161



LIST OF PLATE

Plate		Page
1	Fourier Transform Infrared Spectroscopy (Perkin-Elmer 1600 Series).....	186



LIST OF ABBREVIATIONS

AnV	anisidine value
AOAC	Association of Official Analytical Chemists
AOCS	American Oil Chemist' Society
ATR	attenuated total reflectance
AV	acid value
BSI	British Standards Institute
CLS	classical least-squares
CV	coefficient of variation
DTGS	deuterated triglycine sulfate
FAMEs	fatty acid methyl esters
FAO	Food and Agriculture Organization
FFA	free fatty acid
FT	Fourier transformation
FTIR	Fourier transform infrared
hs	hours
ILS	inverse least-squares
IR	infrared
IRDM	infrared data management



ISO	International Standards Organization
IUPAC	International Union of Pure and Applied Chemistry
IV	iodine value
MD	mean difference
MIR	mid infrared
NaCl	sodium chloride
NIR	near infrared
OPD	optical path difference
PCR	principle component regression
PLS	partial least-squares
PORAM	Palm Oil Refiners Association of Malaysia
PORLA	Palm Oil Registration and Licensing Association
PORIM	Palm Oil Research Institute Malaysia
PRESS	prediction error sum of squares
PTFE	polytetra fluoroethylene
PV	peroxide value
R^2	coefficient of determination
RMSECV	root mean square error of cross-validation
SDD	standard deviation of difference
SEC	standard error of calibration



SEP	standard error of prediction
UV	ultraviolet
WHO	World Health Organization



Abstract of the thesis presented to the Senate of Universiti Putra
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**APPLICATION OF FOURIER TRANSFORM INFRARED
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June 1999

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Malaysia is the largest producer and exporter of palm oil products. For trading purposes, iodine value (IV), free fatty acid (FFA), peroxide value (PV) and anisidine value (AnV) are some parameters used to check the quality of palm oil. They are normally analyzed based on the chemical methods. However, many of these standard methods are time consuming. Therefore, simpler and faster methods, such as IR spectroscopy are necessary.

In this study, Fourier transform infrared (FTIR) calibration models have been developed to correlate the IV, FFA content, PV and AnV in palm oil, that were obtained from the standard methods. In IV determination, a calibration standard was prepared by blending palm stearin and palm superolein. A validation approach yielded a good



coefficient of determination (R^2). Subsequently, 42 palm oil samples with IV ranging from 53 to 65 were also investigated using partial least squares (PLS) and principle component regression (PCR). The results gave R^2 of 0.94443 to 0.98853. In the FFA determination, a calibration set was prepared by spiking different amount of oleic acid to a series of palm olein. The cross-validation procedure gave R^2 of 0.997. In the fourth study, FTIR method to determine PV of palm olein was studied. A wide calibration range of PV was prepared by adding oxidized palm oil into the unoxidized palm olein. The SEP and R^2 gave 0.172 and 0.996, respectively. In the final study, the FTIR spectroscopy was described to predict AnV of palm olein. The calibration set was prepared by mixing the thermally oxidized palm olein and the unoxidized palm olein. The precision of this method was shown to be comparable to chemical method used for measurement of AnV.

The results of this study showed that FTIR methods can be used as alternative to measure a number of trading parameters in palm oil to replace the tedious, time-consuming chemical method.



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Master Sains.

**PENGGUNAAN TRANSFORMASI FOURIER SPEKTROSKOPI
INFRAMERAH (FTIR) UNTUK PENENTUAN PARAMETER-
PARAMETER DAGANGAN MINYAK SAWIT**

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Malaysia merupakan pengeluar dan pengeksport terbesar produk-produk minyak sawit. Untuk kepentingan dagangan, nilai iodin (IV), asid lemak bebas (FFA), nilai peroksida (PV) dan nilai anisidin (AnV) merupakan parameter-parameter yang digunakan dalam pemeriksaan kualiti minyak sawit. Biasanya, parameter-parameter tersebut dianalisa dengan menggunakan kaedah-kaedah kimia. Walau bagaimanapun, kebanyakan kaedah piawai ini memerlukan penggunaan masa yang panjang. Oleh sebab itu, kaedah-kaedah yang mudah dan pantas seperti spektroskopi inframerah amat diperlukan.



Dalam kajian ini, model-model tentukuran FTIR yang dibina untuk IV, FFA, PV dan AnV dalam minyak sawit telah dihubungkan dengan keputusan-keputusan yang didapati dari kaedah piawai. Dalam penentuan IV, satu piawaian tentukuran telah disediakan dengan mencampurkan minyak sawit stearin dan minyak sawit superolein. Pendekatan untuk mengesahkan model tentukuran menghasilkan nilai penentuan angkali yang baik (R^2). Selanjutnya, 42 sampel minyak sawit dengan IV yang berkisar antara 53 dengan 65 telah diselidiki dengan menggunakan “partial least-squares (PLS)” dan “principle component regression (PCR)”. Keputusan-keputusan memberikan R^2 berkisar antara 0.94443 dengan 0.98853. Dalam penentuan FFA, satu piawaian tentukuran telah disediakan dengan mencampurkan asid oleik pada pelbagai kuantiti ke dalam satu siri minyak sawit olein. Prosedur pengesahan silang memberikan nilai R^2 pada 0.997. Dalam kajian keempat, kaedah FTIR telah diselidiki untuk menentukan nilai PV minyak sawit olein. Satu lingkungan tentukuran PV yang luas telah disediakan dengan menambahkan minyak teroksida ke dalam minyak sawit olein tanpa teroksida. Nilai SEP dan R^2 yang didapati dari prosedur pengesahan silang masing-masing berjumlah 0.172 dan 0.996. Dalam kajian terakhir, spektroskopi FTIR telah dihuraikan untuk meramalkan AnV minyak sawit olein. Set tentukuran telah disediakan dengan campuran minyak sawit olein

teroksida secara pemanasan dan minyak sawit tanpa teroksida (0.131 hingga 17.097). Kejituan kaedah ini telah didapati setanding dengan kaedah kimia yang digunakan untuk menentukan AnV.

Keputusan-keputusan dalam kajian ini telah menunjukkan bahawa kaedah FTIR boleh digunakan sebagai kaedah alternatif untuk mengukur parameter-parameter dagangan dalam minyak sawit. Kaedah FTIR mampu menggantikan kaedah kimia yang membosankan dan mengambil masa yang panjang.

CHAPTER I

INTRODUCTION

The African oil palm (*Elaeis guineensis* jacq.) was first introduced to Malaya in 1917. Then, the cultivated lands have increased dramatically from 55000 ha in 1960 to 2.3 million ha in 1993. The production of palm oil has grown from 90000 tonnes in 1960 (Rajanaidu, 1994) to 9.07 million tonnes in 1997 (PORLA, 1998).

Palm oil contributes significantly in supplying the world's requirement for oils and fats. Total world production in 1997 was 17.5 million tonnes and this accounted for 17.5% of the world's total oils and fats output of 99.9 million tonnes. About 31.6 million tonnes of the world's production of oils and fats enter the import-export trade, where palm oil (11.9 million tonnes) leads the other oils with a 37.7% share of the market (PORLA, 1998).



Crude palm oil and crude palm kernel oil are extracted from the oil palm fruit. They are processed through physical refining to yield either refined, bleached and deodorized (RBD) or neutralized, bleached and deodorized (NBD) oils. About 90% of palm oil/palm kernel oil products are used for food purposes while the other 10% for non-food applications (Salmiah, 1994).

Overall, the price of palm oil is reflected by its chemical and physical qualities specified by Palm Oil Registration and Licensing Association (PORLA). Therefore for trading purposes in Malaysia, these specifications have to be complied. With the growing emphasis on quality, governments are introducing more new legislation to control the quality of oils and fats traded in the world market. Trade interest requires agreeable and reliable methods of analysis so that specifications quoted in contractual agreements can be assured. In addition, these methods must also be able to validate sample in cases of trade disputes. To meet this growing need for standardization of analytical techniques, a few organizations were especially involved in the development and publication of standard methods of analysis for oils and fats such as American Oil Chemists' Society (AOCS), International Standards Organization (ISO), Association of Official Analytical Chemists (AOAC), Oils, Fats and