

**COMPARISON OF VEGETABLE OILS AND MONITORING OF RANCIDITY
AND LARD ADULTERATION IN PALM OLEIN USING ELECTRONIC NOSE**

By

GAN HOWE LIN

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

October 2004

ESPECIALLY DEDICATED TO MY BELOVED FAMILY

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

COMPARISON OF VEGETABLE OILS AND MONITORING OF RANCIDITY AND LARD ADULTERATION IN PALM OLEIN USING ELECTRONIC NOSE

BY

GAN HOWE LIN

October 2004

Chairman: Professor Yaakob bin Che Man, Ph.D.

Faculty: Food Science and Technology

Flavour analysis is typically performed by organoleptic panel, which is often expensive and less objective. A novel approach using a surface acoustic wave (SAW) sensing-based electronic nose (zNoseTM) for flavour analysis was explored in this study for determination of some parameters of edible oils and fats.

In the characterization of sixteen types of vegetable oils, the high resolution olfactory image, called VaporPrintTM, was unique for each type of vegetable oil studied. The score plot from principal component analysis (PCA) indicated that 97% of the total variance in the zNoseTM measurement data was described by PC 1 and PC 2. The loading plot revealed five compounds (*m*, *k*, *n*, *s*, and *p*) that were important to differentiate the vegetable oils.

In determining oxidative stability of RBD palm olein, the results of zNoseTM showed significant difference ($P < 0.05$) between fresh oil and rancid oil. VaporPrintTM provided the operator with a visually recognised pattern for rapid identification of rancid off-flavour. By using Pearson correlation analysis, high correlation ($r > 0.90$) was observed between electronic nose responses and chemical test data; as well as between electronic nose responses and sensory evaluation scores.

The zNoseTM technique was also employed to monitor the presence of lard as an adulterant in RBD palm olein. As the adulteration level increased from 1% to 20%, a few distinct peaks were found to gradually increase in size in the zNoseTM chromatograms. These peaks dramatically increased in 3% lard, while the corresponding VaporPrintTM was obviously an exception to the normal RBD palm olein pattern. Qualitative identification of adulterated RBD palm olein samples was possible by the characteristic VaporPrintTM. The most significant relationship occurred between percent lard and adulterant peak number 6 ($R^2 = 0.906$). An ideal correlation was also observed between the electronic nose response and chemical analyses ($r > 0.90$).

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

**PENCIRIAN MINYAK SAYURAN SERTA PEMANTAUAN KETENGIKAN DAN
PENGADUKAN MINYAK KELAPA SAWIT OLEIN DENGAN LEMAK
KHINZIR MENGGUNAKAN HIDUNG ELEKTRONIK**

Oleh

GAN HOWE LIN

October 2004

Pengerusi: Profesor Yaakob bin Che Man, Ph.D

Fakulti: Sains Makanan dan Teknologi

Pengujian citarasa biasanya dijalankan oleh panel ujideria yang selalunya mahal dan kurang objektif. Satu pendekatan baru menggunakan hidung elektronik (zNoseTM) yang berasaskan pengesanan “surface acoustic wave” (SAW) untuk menganalisa citarasa telah diterokai dalam kajian ini untuk menentukan beberapa parameter minyak dan lemak masakan.

Dalam pencirian enam belas jenis minyak sayuran yang berbeza, didapati imej resolusi tinggi yang dikenali sebagai VaporPrintTM adalah unik untuk setiap jenis minyak masak yang dikaji. Lakaran skor dari “principal component analysis” (PCA) menunjukkan 97% daripada jumlah varian dapat dijelaskan oleh PC 1 dan PC 2. Lakaran loading pula

menunjukkan lima komponen (m , k , n , s , and p) yang penting dalam pembezaan minyak sayuran yang berlainan.

Dalam menentukan kestabilan pengoksidaan minyak kelapa sawit olein, keputusan zNoseTM menunjukkan perbezaan yang bermakna ($P < 0.05$) di antara minyak segar dengan minyak tengik. VaporPrintTM membolehkan pengguna mengenalpasti bau tengik dalam minyak dengan pantas. Dengan menggunakan korelasi Pearson, korelasi yang tinggi ($r > 0.90$) telah didapati di antara respons zNoseTM dan data ujian kimia, serta diantara respons zNoseTM dan skor ujideria.

Kaedah zNoseTM juga digunakan untuk menguji kehadiran lemak khinzir dalam minyak kelapa sawit olein. Apabila lemak khinzir meningkat dari 1% ke 20%, saiz beberapa puncak didapati bertambah secara beransur-ansur. Puncak-puncak ini meningkat secara mendadak dalam 3% lemak khinzir sementara VaporPrintTM yang berkaitan adalah suatu pengecualian yang ketara kepada corak normal minyak kelapa sawit olein. Pengenalpastian kualitatif terhadap sampel RBD olein sawit yang tercemar dapat dilakukan dengan ciri-ciri VaporPrintTM yang tersendiri. Perhubungan yang paling ketara wujud antara peratus lemak khinzir tercemar dengan puncak ke-enam ($R^2 = 0.906$). Korelasi yang baik juga diperhatikan di antara respons zNoseTM dan analisis kimia ($r > 0.90$).

ACKNOWLEDGEMENTS

I would like to express my most sincere gratitude and appreciation to my supervisory committee chairman, Professor Dr. Yaakob bin Che Man, for his invaluable guidance, advice and constant encouragement throughout the course of my research study. His constructive criticisms and suggestions provided me the strength and perseverance to complete this thesis despite several obstacles encountered throughout the course of this research, which at times seemed insurmountable. Appreciation also goes to the members of my supervisory committee, Dr. Nazimah Sheikh Abdul Hamid, Dr. Tan Chin Ping, and Dr. Nor Aini Idris for their support and invaluable suggestions to guide me during my study.

I wish to express my gratitude to all members of the Faculty of Food Science and Biotechnology, UPM for providing the research facilities and technical assistance during my graduate study. Acknowledgment is also due to all my friends, Lee Wai Cheng, Pua Chun Kiat, Kenny Loh Seng Kean, Ms. Mariam binti Abdul, Ms Wanna Ammawath, Syahariza Zainul Abidin, Reni Rahayou Mohd. Zain; and those who have given me the moral encouragement and support to complete my graduate study.

I would like to acknowledge the financial support provided by IRPA short term fund (53098) awarded to Dr. Tan Chin Ping and IRPA fund (03-02-040172-EA001) awarded to Professor Dr. Yaakob bin Che Man for this study. Acknowledgement is also due to the PASCA scholarship for granting me the opportunity to pursue my master degree. I would

like to thank STRIDE of Ministry of Defence, Malaysia, and Mr. Tibby Lim (Electronic Sensor Technology Malaysia representative) for technical support.

I am also greatly indebted to my beloved parents and sisters, for their love, spiritual encouragement and support. I wish also to express my deepest appreciation to my boy friend, for his understanding, concern, faith and love.

I certify that an Examination Committee met on 27 October 2004 to conduct the final examination of Gan Howe Lin on her Master of Science thesis entitled “Comparison of Vegetable Oils and Monitoring of Rancidity and Lard Adulteration in Palm Olein using Electronic Nose” in accordance with Universiti Pertanian Malaysia (Higher Degree) Act 1980 and Universiti Pertanian Malaysia (Higher Degree) Regulation 1981. The committee recommended that the candidate be awarded the relevant degree. Members of the Examination Committee are as follows:

AZIZAH HAMID, Ph.D.

Associate Professor
Faculty of Food Science and Technology
Universiti Putra Malaysia
(Chairman)

SALMAH YUSOF, Ph.D.

Professor
Faculty of Food Science and Technology
Universiti Putra Malaysia
(Member)

LAI OI MING, Ph.D.

Lecturer
Faculty of Food Science and Technology
Universiti Putra Malaysia
(Member)

AMINAH ABDULLAH, Ph.D.

Professor
Faculty of Science and Technology
Universiti Kebangsaan Malaysia
(Independent Examiner)

GULAM RUSUL RAHMAT ALI, Ph.D.

Professor/ Deputy Dean
School of Graduate Studies
Universiti Putra Malaysia

Date :

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

YAAKOB BIN CHE MAN, Ph.D.

Professor
Faculty of Food Science and Biotechnology
Universiti Putra Malaysia
(Chairman)

NAZIMAH SHEIKH ABDUL HAMID, Ph.D.

Lecturer
Faculty of Food Science and Biotechnology
Universiti Putra Malaysia
(Member)

TAN CHIN PING, Ph.D.

Lecturer
Faculty of Food Science and Biotechnology
Universiti Putra Malaysia
(Member)

NOR AINI BINTI IDRIS, Ph.D.

Principal Research Officer
Malaysia Palm Oil Board (MPOB)
(Member)

AINI IDERIS, Ph.D.

Professor/ Dean
School of Graduate Studies
Universiti Putra Malaysia

Date :

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.

GAN HOWE LIN

Date:

TABLE OF CONTENTS

	Page
DEDICATION	ii
ABSTRACT	iii
ABSTRAK	v
ACKNOWLEDGEMENTS	vii
APPROVAL	ix
DECLARATION	xi
LIST OF TABLES	xiv
LIST OF FIGURES	xv
LIST OF ABBREVIATIONS	xvii
CHAPTER	
I GENERAL INTRODUCTION	1
II LITERATURE REVIEW	5
Aroma Analysis Techniques	5
Sensory Analysis	6
Gas Chromatography/Mass Spectrometry	7
Electronic Nose	8
History	8
Definition	9
Principles of Olfaction	9
Sensor Technology	11
Statistical Analysis Techniques	17
Applications of Electronic Nose	19
Application of Electronic Nose in Fats and Oils Industry	24
Characterization of Vegetable Oils	24
Monitoring the Oxidation of Edible Oil	28
Monitoring the Authenticity of Edible Oil	30
Vegetable Oils	30
Rancidity in Fats and Oils	32
Oils and Fats Authentication	38
III CHARACTERIZATION OF VEGETABLE OILS BY SURFACE ACOUSTIC WAVE SENSING ELECTRONIC NOSE	44
Introduction	44
Materials and Methods	47
Oil Samples	47
Chemical Analyses	47
Electronic Nose Apparatus	48

	Electronic Nose Analysis	50
	Data Analysis	51
	Principal Component Analysis (PCA)	51
	Results and Discussion	52
	Chemical Analyses	52
	Electronic Nose Analysis	56
	Principal Component Analysis (PCA)	63
	Conclusion	66
IV	MONITORING THE STORAGE STABILITY OF RBD PALM OLEIN USING THE ELECTRONIC NOSE	67
	Introduction	67
	Materials and Methods	68
	Oil Samples	68
	Chemical Analyses	69
	Sensory Evaluation	69
	Electronic Nose Analysis	69
	Data Analysis	70
	Results and Discussion	70
	Chemical Analyses	70
	Sensory Evaluation	76
	Electronic Nose Analysis	77
	Conclusion	89
V	MONITORING THE LARD ADULTERATION IN RBD PALM OLEIN BY ELECTRONIC NOSE	90
	Introduction	90
	Materials and Methods	92
	Oil Samples	92
	Blend Preparations	93
	Chemical Analyses	93
	Electronic Nose Analysis	93
	Data Analysis	93
	Results and Discussion	94
	Chemical Analyses	94
	Electronic Nose Analysis	97
	Conclusion	105
VI	GENERAL DISCUSSION AND RECOMMENDATION	106
	Conclusions	106
	Recommendations	109
	REFERENCES	112
	APPENDICES	125
	BIODATA OF THE AUTHOR	136