



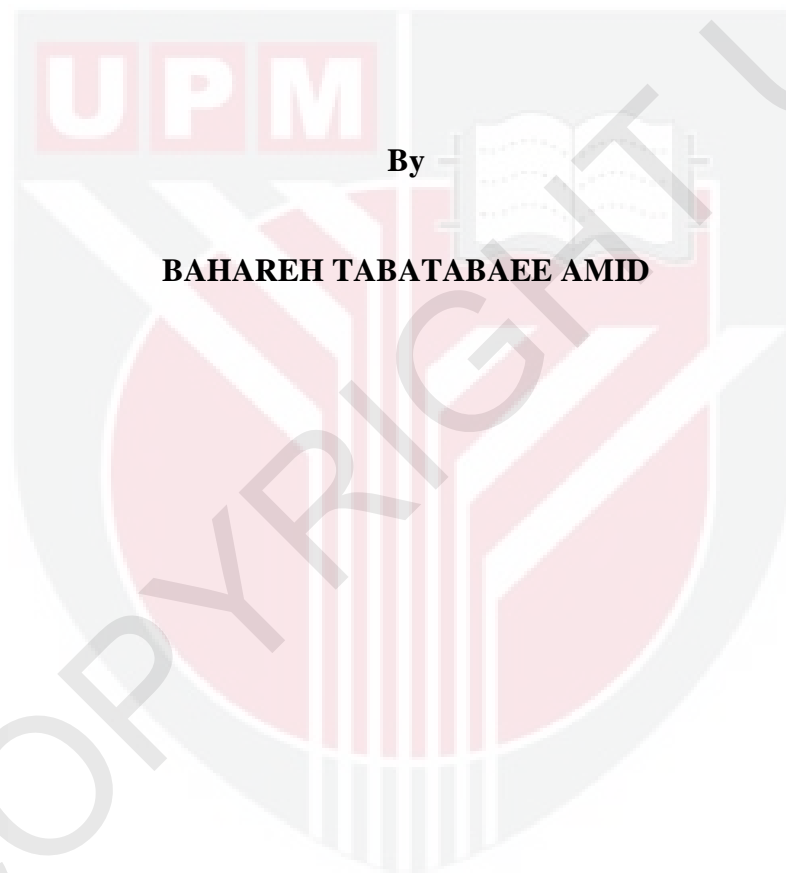
UNIVERSITI PUTRA MALAYSIA

**EFFECTS OF EXTRACTION, PURIFICATION AND DRYING
ON THE PHYSICOCHEMICAL PROPERTIES OF DURIAN
(*Durio zibethinus*) SEED GUM**

BAHAREH TABATABAEE AMID

FSTM 2011 16

**EFFECTS OF EXTRACTION, PURIFICATION AND DRYING ON THE
PHYSICOCHEMICAL PROPERTIES OF DURIAN (*Durio zibethinus*)
SEED GUM**

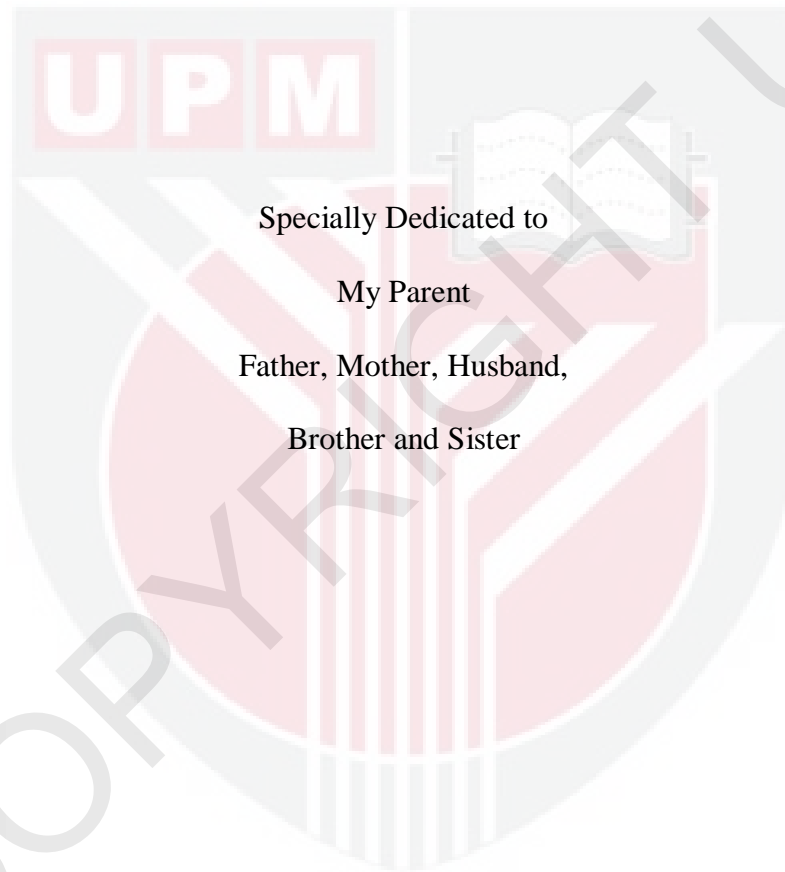


By

BAHAREH TABATABAEE AMID

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

December 2011



Specially Dedicated to

My Parent

Father, Mother, Husband,

Brother and Sister

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in
fulfilment of the requirement for the degree of Doctor of Philosophy

**EFFECTS OF EXTRACTION, PURIFICATION AND DRYING ON THE
PHYSICOCHEMICAL PROPERTIES OF DURIAN (*Durio zibethinus*)
SEED GUM**

By

BAHAREH TABATABAEE AMID

December 2011

Chairman : Seyed Hamed Mirhosseini, PhD

Faculty : Food Science and Technology

The long-term goal of the current study was to look into the possibility of applying the biorefinery process for utilizing the main biomass of durian fruit. The main objective was to investigate the effect of extraction, purification and drying on the chemical composition, physicochemical and functional properties of durian seed gum. The efficiency of two aqueous and chemical extraction techniques was compared under the optimum extraction conditions to determine the most suitable extraction condition resulted in the crude durian seed gum with desirable functional properties. For further improvement of functional properties of crude gum, the determination of most suitable purification and drying techniques resulting in the purified durian seed gum with desirable properties was carried out. The results indicated that durian seed gum exhibited a typical

'pseudoplastic or shear-thinning rheological behavior'. In fact, the viscosity of durian seed gum decreased when the shear rate was increased. The durian seed gum showed more viscous (liquid like) behavior rather than the elastic (solid like) behavior. This is typical rheological behaviour of diluted gum solution, when the molecules cannot entangle and produce a network structure during oscillatory stress. This could be due to the random-coil polysaccharide structure which behaved like a liquid when exposed to low frequencies of oscillation. The carbohydrate analysis showed that the major monosaccharide composition of crude durian seed gum include D-galactose (57.2 ± 3.2), glucose (41.8 ± 3.9), and small contents of arabinose (0.6 ± 0.03) and xylose (0.4 ± 0.06). The current study revealed that the durian seed gum may be classified as a galactan or arabinogalactans rather than a galactomannan.

The results indicated that the chemical extraction variables exhibited the highest significant ($p < 0.05$) effect on the oil-holding capacity (OHC) and extraction yield, respectively. Among all chemical extraction variables, the soaking temperature and decolouring time showed the most significant ($p < 0.05$) effect on the physicochemical and functional properties of the crude durian seed gum. The optimum chemical extraction (i.e. the decolouring time ~119 min, soaking time ~1.5 h and soaking temperature 16.5 °C) resulted in the most desirable physicochemical properties. The results indicated that the purification process significantly ($p < 0.05$) improved the solubility of crude durian seed gum. This could be explained by the fact that the purification process resulted in the significant ($p < 0.05$) decrease in the protein content and some impurities in the

chemical structure of crude durian seed gum. The current study revealed that the purification method C (using saturated barium hydroxide) resulted in the highest protein content, maximum solubility and OHC as well as the most desirable viscous modulus (G'') among all purification methods. In addition, the purified seed gum using saturated barium hydroxide also provided high WHC, large specific surface area and relatively high purification yield and viscosity. Therefore, the present study suggests that the purification process using saturated barium hydroxide is recommended as the most suitable purification technique for durian seed gum.

Finally, the effect of four different drying methods (namely oven drying (105 °C), vacuum oven drying, freeze drying and spray drying) on the chemical composition and physicochemical properties of purified durian seed gum was investigated. In this study, the drying process led to improve the solubility of purified durian seed gum. This observation could be explained by the significant ($p < 0.05$) effect of drying process on the carbohydrate composition and protein content of purified durian seed gum. In fact, the drying process resulted in (i) decrease the protein, xylose and arabinose contents and (ii) increase the glucose and galactose contents. The present study revealed that the freeze drying resulted in the durian seed gum with smallest average droplet size. In addition, the freeze-dried seed gum also provided the highest protein content and maximum solubility, specific surface area, viscosity and OHC among other samples. Therefore, the present study recommends the freeze drying as the most suitable drying technique for the durian seed gum.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi sebahagian keperluan untuk ijazah Doktor Falsafah

**KESAN PENGEKSTRAKAN, BERSUCI DAN PENGERINGAN
TERHADAP CIRI FIZIKOKIMIA DURIAN (*Durio zibethinus*) SEED GUM**

Oleh

BAHAREH TABATABAEE AMID

Disember 2011

Pengerusi : Seyed Hamed Mirhosseini, PhD

Fakulti : Sains dan Teknologi Makanan

Matlamat jangka panjang kajian semasa adalah untuk mengkaji kemungkinan menggunakan proses biorefinery untuk menggunakan biomass utama buah durian. Objektif utama adalah untuk menyiasat kesan pengekstrakan, penulenan dan pengeringan pada komposisi kimia, sifat-sifat fizikokimia dan berfungsi gusi benih durian. Kecekapan dua teknik pengekstrakan akueus dan kimia berbanding di bawah syarat-syarat yang pengekstrakan optimum untuk menentukan pengekstrakan keadaan yang paling sesuai yang menyebabkan gusi benih durian mentah dengan ciri-ciri yang diinginkan berfungsi. Untuk penambahbaikan sifat-sifat fungsi gula mentah, penentuan penyucian yang paling sesuai dan pengeringan teknik-teknik yang menyebabkan gusi disucikan benih durian dengan sifat-sifat yang diinginkan telah dijalankan. Keputusan menunjukkan bahawa gula-gula getah benih durian yang dipamerkan 'tingkah laku pseudoplastic atau penipisan ricih

reologi' tipikal. Malah, kelikatan gusi benih durian berkurangan apabila kadar ricih meningkat. Gusi benih durian menunjukkan tingkah laku yang lebih likat (cecair seperti) bukannya kelakuan anjal (pepejal seperti). Ini adalah tingkah laku yang biasa reologi larutan gula dicairkan, apabila molekul tidak boleh menjerat dan menghasilkan struktur rangkaian semasa tekanan ayunan. Analisis karbohidrat menunjukkan bahawa komposisi monosakarida utama gusi benih durian mentah termasuk D-galactose (57.2 ± 3.2), glucose (41.8 ± 3.9), dan kandungan kecil arabinose (0.6 ± 0.03) dan xylose (0.4 ± 0.06). Kajian semasa menunjukkan bahawa gula-gula getah benih durian boleh dikelaskan sebagai galactan atau arabinogalactans bukannya satu galactomannan.

Keputusan menunjukkan bahawa pemboleh ubah pengekstrakan kimia mempamerkan tertinggi yang signifikan ($p < 0.05$) kesan ke atas keupayaan minyak pegangan (OHC) dan hasil pengekstrakan, masing-masing. Antara semua pemboleh ubah pengekstrakan kimia, suhu jering dan masa decolouring menunjukkan kesan yang paling ketara ($p < 0.05$) pada sifat-sifat fizikokimia dan berfungsi gusi benih durian mentah. Pengekstrakan kimia yang optimum (iaitu masa decolouring ~ 119 min, jering masa ~ 1.5 h dan merendam suhu 16.5°C) menyebabkan sifat-sifat fizikokimia yang paling wajar. Keputusan menunjukkan bahawa proses penulenan yang signifikan ($p < 0.05$) meningkatkan kebolehlarian gusi benih durian mentah. Ini dapat dijelaskan oleh fakta bahawa proses penulenan menyebabkan penurunan yang signifikan ($p < 0.05$) di dalam kandungan protein dan beberapa kekotoran dalam struktur kimia gusi benih durian mentah. Kajian semasa menunjukkan bahawa kaedah penulenan C

(menggunakan saturated barium hydroxide) menyebabkan kandungan protein tertinggi, kelarutan maksimum dan OHC serta modulus likat yang paling dikehendaki (G'') di kalangan semua kaedah penulenan. Oleh itu, kajian ini mencadangkan bahawa proses penulenan menggunakan saturated barium hydroxide tepu disyorkan sebagai teknik penulenan yang paling sesuai untuk gusi benih durian.

Akhir sekali, kesan daripada empat kaedah pengeringan yang berlainan (iaitu pengeringan oven ($105\text{ }^{\circ}\text{C}$), ketuhar vakum pengeringan pengeringan beku, dan semburan pengeringan) pada komposisi kimia dan sifat-sifat fizikokimia suci gusi benih durian disiasat. Dalam kajian ini, proses pengeringan membawa kepada meningkatkan kebolehlarutan gusi benih durian. Pemerhatian ini boleh dijelaskan oleh kesan yang signifikan ($p < 0.05$) proses pengeringan pada komposisi karbohidrat dan kandungan protein gusi benih durian. Malah, proses pengeringan yang mengakibatkan (i) mengurangkan protein, xylose dan kandungan arabinose dan (ii) meningkatkan kandungan glukosa dan galaktosa. Kajian ini menunjukkan bahawa pembekuan pengeringan menyebabkan gusi benih durian dengan kelantangan-wajaran minimum bermakna (atau terkecil purata saiz titisan). Di samping itu, pembekuan kering benih gusi juga menyediakan kandungan protein yang tertinggi dan kelarutan maksimum, kawasan permukaan yang tertentu, kelikatan dan OHC di kalangan sampel lain. Oleh itu, kajian ini mencadangkan pembekuan pengeringan sebagai teknik yang paling sesuai kering gusi benih durian.

ACKNOWLEDGEMENTS

I am grateful to have this opportunity to express my sincere appreciation and thanks to my supervisor Associate Professor Dr. Seyed Hamed Mirhosseini for his valuable guidance, suggestion and stimulating discussion. This thesis could not have been completed if not for the worth mentioning involvement and support of the following people, to whom I am deeply indebted. His intellectual expertise, excellent support and professional guidance made this undertaking manageable. From the inception of the project until its completion, his invaluable comments and ideas have contributed a great deal to the refinement of this thesis. I would also like to express my appreciation and gratitude to my committee members, Prof. Dr. Yazid Abd Manap, Associate Prof. Dr. Tan Chin Ping and Associate Prof. Lasekan Olusegun for their remarkable ideas, constructive comments, continuous support and guidance throughout of the duration of this research project.

I would like to express my sincere feeling towards my husband, Mr. Farhad Farivar, since he was very patient and helpful during my PhD work. The completion of this study would not be possible without the assistance and support from Mr. Halim from Chromatography Lab, Mr. Azman from biochemistry lab and all the laboratory staff especially Mr. Amran and Madam Jamaliah for their kindness and technical assistance. Last but not least, I would like to express my gratitude to my parents and family for their fullest encouragement and concern throughout the entire course of my studies.

I certify that an Examination Committee has met on December 2011 to conduct the final examination of Bahareh Tabatabaee Amid on his Doctor of Philosophy thesis entitled " **EFFECTS OF EXTRACTION, PURIFICATION AND DRYING ON THE PHYSICOCHEMICAL CHARACTERISTICS OF DURIAN (*Durio zibethinus*) SEED GUM**" in accordance with Universiti Pertanian Malaysia (Higher Degree) Act 1980 and Universiti Pertanian Malaysia (Higher Degree) Regulations 1981. The Committee recommends that the student be awarded the degree of Doctor of Philosophy.

Examination Committees are as follows:

SON RADU, PHD

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

HASANAH MOHD GHAZALI, PHD

Professor
Department of Food Science
Faculty of Food Science and Technology
Universiti Putra Malaysia
(Internal Examiner)

ALFI KHATIB, PHD

Lecturer
Department of Food Science
Faculty of Food Science and Technology
Universiti Putra Malaysia
(Internal Examiner)

SEOW HENG FONG, PhD

Professor and Deputy Dean
School of Graduate Studies
Universiti Putra Malaysia

Date: December 2011

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

Syed Hamed Mirhosseini, PhD

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

Mohd Yazid Abd Manap, PhD

Professor
Faculty of Science and Technology
Universiti Sains Islam Malaysia
(Member)

Tan Chin Ping, PhD

Associate Prof.
Faculty of Food Science and Technology
Universiti Putra Malaysia
(Member)

Lasekan Olusegun, PhD

Associate Prof.
Faculty of Food Science and Technology
Universiti Putra Malaysia
(Member)

BUJANG BIN KIM HUAT, PhD

Professor and Dean
School of Graduate Studies
Universiti Putra Malaysia

Date: 2 March 2012

DECLARATION

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

BAHAREH TABATABAEE AMID

Date: 9 December 2011

TABLE OF CONTENTS

	Page
DEDICATION	ii
ABSTRACT	iii
ABSTRAK	vi
ACKNOWLEDGEMENTS	ix
APPROVAL	x
DECLARATION	xii
LIST OF TABLES	xVii
LIST OF FIGURES	xviii
LIST OF ABBREVIATIONS	xxii
 CHAPTER	
I	
GENERAL INTRODUCTION	1
II	
LITERATURE REVIEW	8
2.1 Durian Fruit	8
2.1.1 Durian (<i>Durio zibethinus</i> Murray)	8
2.1.2 Durian Species	10
2.1.3 Nutritional and Medicinal Application	11
2.1.4 Edible and Waste Portion	12
2.1.5 Biorefinery Process	13
2.2. Natural Gums and Their Sources	15
2.2.1 Natural Sources of Gums	16
2.2.1.1 Plant-Based Gums	17
2.2.2 Basic Structure of Gum	19
2.2.3 Chemical Composition of Gum	20
2.2.3.1 Carbohydrate Composition	20
2.2.3.2 Protein Fraction	23
2.2.4 Functions and Applications of Gum	26
2.2.5 Physicochemical Properties	27
2.2.5.1 Emulsifying Properties	28
2.2.5.2 Droplet Size and Distribution	30
2.2.5.3 Rheological Properties	32
2.2.5.4 Solubility	35
2.2.5.5 Water-Holding Capacity (WHC) and Oil-Holding Capacity (OHC)	39
2.2.6 Factors Affecting Physicochemical Properties of Gum	42
2.2.6.1 Effect of Extraction on Physicochemical Properties of Gum	43
2.2.6.2 Effect of Purification Process on Physicochemical Properties of Gum	49
2.2.6.3 Effect of Drying Process on Physicochemical Properties of Gum	54

III	MATERIALS AND METHODS	58
3.1.	Materials and Methods	58
3.1.1.	Chemicals and Standards	58
3.1.2.	Extraction Process	59
3.1.2.1	Aqueous Extraction	59
3.1.2.2	Chemical Extraction	60
3.1.3.	Purification Process	61
3.1.3.1.	Purification Using Isopropanol and Ethanol (Method A)	61
3.1.3.2.	Purification Using Isopropanol and Acetone (Method B)	62
3.1.3.3.	Purification Using Saturated Barium Hydroxide (Method C)	62
3.1.3.4.	Purification Using Fehling Solution Plus Hydrochloric Acid and Ethanol (Method D)	63
3.1.4	Drying Process	63
3.1.4.1	Oven Drying	64
3.1.4.2	Vacuum Oven Drying	64
3.1.4.3	Spray Drying	65
3.1.4.4	Freeze Drying	66
3.1.5.	Analytical Tests	66
3.1.5.1	Extraction Yield	66
3.1.5.2	Purification Yield	67
3.1.5.3	Protein Analysis	67
3.1.5.4	Volume-Weighted Mean (D [4, 3])	68
3.1.5.5	Span	69
3.1.5.6	Specific Surface Area	70
3.1.5.7	Solubility	71
3.1.5.8	Water-Holding Capacity (WHC) and Oil-Holding Capacity (OHC)	72
3.1.5.9	Apparent Viscosity	73
3.1.5.10	Elastic modulus (G') and Viscous Modulus (G'')	73
3.1.5.11	Sugar Composition	74
3.1.5.12	Moisture Content	75
3.1.5.13	Molecular weight	75
3.1.6.	Experimental Design and Data Analysis	78
3.1.6.1	Aqueous Extraction	78
3.1.6.2	Chemical Extraction	81
3.1.6.3	Purification Process	82
3.1.6.4	Drying Process	84
3.1.7.	Optimization and Validation Procedures	85

IV	RESULTS AND DISCUSSION	87
	4.1. Process Optimization of Aqueous Extraction of Crude Durian (<i>Durio zibethinus</i>) Seed gum	87
	4.1.1. Extraction Yield	90
	4.1.2. Protein Content	95
	4.1.3. Volume-Weighted Mean (D [4, 3])	99
	4.1.4. Span	101
	4.1.5. Specific Surface Area	103
	4.1.6. Solubility	109
	4.1.7. Water-Holding Capacity (WHC)	116
	4.1.8. Oil-Holding Capacity (OHC)	121
	4.1.9. Apparent Viscosity	124
	4.1.10. Elastic Modulus (G') and Viscous Modulus (G'')	133
	4.1.11. Multiple Optimization and Validation Procedures	137
	4.1.12. Carbohydrate Profile	141
	4.2. Process Optimization of Chemical Extraction of Crude Durian (<i>Durio zibethinus</i>) Seed gum	145
	4.2.1. Extraction Yield	148
	4.2.2. Protein Content	151
	4.2.3. Volume-Weighted Mean (D [4, 3])	154
	4.2.4. Span	156
	4.2.5. Specific Surface Area	157
	4.2.6. Solubility	160
	4.2.7. Water-Holding Capacity (WHC)	165
	4.2.8. Oil-Holding Capacity (OHC)	167
	4.2.9. Apparent Viscosity	169
	4.2.10. Elastic Modulus (G') and Viscous Modulus (G'')	173
	4.2.11. Multiple Optimization and Validation Procedures	176
	4.2.12. Carbohydrate Profile	182
	4.2.13. Comparison of Physicochemical Properties of Aqueous and Chemically Extracted Gums	182
	4.3. Determination of the Most Suitable Method for Purification of Durian (<i>Durio zibethinus</i>) Seed Gum	184
	4.3.1. Purification Yield	184
	4.3.2. Protein Content	186
	4.3.3. Volume-Weighted Means (D [4, 3])	189
	4.3.4. Span	191
	4.3.5. Specific Surface Area	193
	4.3.6. Solubility	195
	4.3.7. Water-Holding Capacity (WHC)	201
	4.3.8. Oil-Holding Capacity (OHC)	203
	4.3.9. Apparent Viscosity	205
	4.3.10. Elastic Modulus (G') and Viscous Modulus (G'')	209
	4.3.11. Carbohydrate Profile	212
	4.3.12. Screening of the Most Efficient Purification Method	217
	4.4. Determination of the Most Suitable Drying Technique for Durian (<i>Durio zibethinus</i>) Seed Gum	220

4.4.1. Protein content	220
4.4.2. Volume-weighted mean	223
4.4.3. Span	224
4.4.4. Specific surface area	226
4.4.5. Solubility	229
4.4.6. Water-Holding Capacity (WHC)	233
4.4.7. Oil-Holding Capacity (OHC)	236
4.4.8. Apparent Viscosity	238
4.4.9. Elastic Modulus (G') and Viscous Modulus (G'')	242
4.4.10. Carbohydrate Profile	245
4.4.11. Moisture Content	251
4.4.12. Screening of the Most Efficient Drying Method	253
4.4.13. Molecular weight	254
V CONCLUSIONS AND RECOMMENDATIONS	257
REFERENCES	263
APPENDIX	298
LIST OF PUBLICATIONS	319
BIODATA OF STUDENT	323