

**EXTRACTION AND CHARACTERIZATION OF LOW METHOXYL
PECTIN FROM DURIAN RIND**

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A thesis submitted in
fulfilment of the requirement for the award of the
Doctor of Philosophy in Science

Faculty of Applied Sciences and Technology
Universiti Tun Hussein Onn Malaysia



MARCH 2023

DEDICATION

This thesis is dedicated to my Lord and Saviour, Jesus Christ.

For from Him and through Him and to Him are all things.

May all the glory be to God.

*I dedicate this thesis to my beloved parents,
who have been my greatest support.*



ACKNOWLEDGEMENT

First and foremost, I would like to give thanks to my Lord Jesus Christ for giving me strength, courage, and hope throughout my Ph.D. journey. Without Him, I could have given up halfway. I give thanks for all the provisions from God that were poured down to me, the research resources, finances, and helps from the people around me. All my achievements belong to Him. I give all glory to God.

Words cannot express my sincere appreciation to my supervisor, Ts. Dr. Norazlin binti Abdullah, who had been there with me through the thin and thick of my research journey. She is a dedicated teacher and an understanding friend. She gave me much support, guidance, and affirmation whenever I needed it. Not to mention, my co-supervisor, Chm. Dr. Norhayati binti Muhammad, who had extended her help and advice to me despite her heavy workload.

I am so grateful and lucky to have many good friends and lab mates on this journey. They had made the dull lab works more enjoyable. Thank you for all the prayers, the good food sent to me when I had no time to eat, and the help and company throughout my lab work, sometimes even until nighttime. Additionally, I would like to extend my gratitude to all the laboratory technicians for being kind, helpful, and supportive throughout my research journey. Not to forget Uncle Lee, a very kind and helpful uncle who had supplied me with the durian rinds for my research.

Finally, from the bottom of my heart, I am genuinely thankful for my family, who have been my greatest support and backup throughout my life. Without their continuous moral and financial support, I can't be where I am today. I love my parents so much. I am grateful for their support and trust in my decision to pursue a Ph.D. I hope I have made my parents proud of what I have achieved.

ABSTRACT

Durian is a tropical fruit in Southeast Asia. Durian rind, comprising 65-70% of durian total weight, may serve as a potential source of pectin, a gelling agent commonly used in the food and pharmaceutical industries. This study aims to optimize the pectin extraction from durian rinds and characterize its physicochemical and rheological properties. The factors affecting pectin extraction: durian rind parts and varieties, acid type and concentration, temperature, duration, and solid-to-liquid ratio (S:L) were screened and optimized based on yield, esterification degree (DE), and purity (AUA). Pectin was characterized by intrinsic viscosity, molecular weight, structural, thermal, morphological, and rheological analysis. The durian rind's white part has higher pectin yield than the rind with thorns, while variety D168 has lower yield than other varieties. The parts and varieties did not significantly affect DE and AUA. Pectin yield and DE deteriorated at higher acid concentrations (0.1, 1.0 M). Mineral acids (hydrochloric, sulphuric, and nitric acids) were more effective in extracting pectin of higher purity (AUA > 65%). Organic acids-extracted pectins (citric, acetic, and tartaric acids) had AUA < 65%, indicating poor purity. 0.001M sulphuric acid was selected for extraction optimization due to its good yield (9.13%) and purity (AUA = 66.72%) of low-methoxyl pectin (LMP) (DE = 20.11%). According to response surface methodology, temperature was the most significant variable affecting yield and purity. DE was not pronouncedly affected by temperature (75 – 95 °C), time (30 – 270 minutes), and S:L (1:20 – 1:60 g/mL). The optimal extraction conditions (93.3 °C, S:L 1:50, 185 minutes) yielded 12.12% pectin with AUA = 77.01%, DE = 18.99%, intrinsic viscosity = 148.74 mL/g, and molecular weight = 42.12 kDa. The strongest gel was obtained with 3% (w/v) pectin at pH 3, showing better gel strength than commercial LMP. The gel showed good thermal stability throughout heating and cooling. Therefore, durian rind is a potential LMP source, which could be a gelling and thickening agent for weak-acid-low-calorie food and pharmaceutical applications. Future studies could be done to explore durian rind pectin's applications in the pharmaceutical and food industries.

ABSTRAK

Durian ialah buah tropika di Asia Tenggara. Kulit durian, terdiri daripada 65 – 70% berat buah durian, berpotensi menjadi sumber pektin, agen pembentuk gel yang biasa dipakai dalam industri makanan dan farmaseutikal. Objektif kajian ini adalah untuk mengoptimumkan pengekstrakan pektin daripada kulit durian dan mencirikan sifat fizikokimia dan reologinya. Faktor-faktor yang menpengaruhi pengekstrakan pektin: bahagian dan varieti kulit durian, jenis dan kepekatan asid, suhu, tempoh dan nisbah pepejal kepada cecair (S:L) telah disaring dan dioptimumkan berdasarkan hasil, darjah pengesteran (DE) dan ketulenan (AUA) pektin. Analisis pektin yang dilakukan adalah termasuk analisis kelikatan intrinsik dan berat molekul, struktur, terma, morfologi dan reologi. Bahagian putih kulit durian mempunyai hasil pektin yang lebih tinggi daripada kulit berduri, manakala varieti D168 mempunyai hasil yang lebih rendah berbanding varieti lain. Bahagian dan varieti tidak mempengaruhi DE dan AUA dengan ketara. Dapatan kajian juga menunjukkan hasil dan DE pektin menurun pada kepekatan asid yang lebih tinggi (0.1, 1.0M). Selain itu, asid mineral (asid hidroklorik, sulfurik dan nitrik) lebih berkesan dalam mengekstrak pektin dengan ketulenan yang lebih tinggi ($AUA > 65\%$). Pektin yang diekstrak oleh asid organik (asid sitrik, asetik dan tartarik) mempunyai $AUA < 65\%$, ini menunjukkan ketulenan yang rendah. Asid sulfurik 0.001M telah dipilih untuk proses pengoptimuman pengekstrakan disebabkan oleh perolehan hasil (9.13%) dan ketulenan ($AUA = 66.72\%$) pektin metoksil rendah (LMP) ($DE = 20.11\%$) yang baik. Menurut kaedah permukaan tindak balas, suhu adalah faktor yang paling ketara dalam mempengaruhi hasil dan ketulenan pektin. DE tidak menunjukkan perubahan yang ketara pada suhu (75 – 95 °C), masa (30 – 270 minit) dan S:L (1:20 – 1:60 g/mL). Keadaan pengekstrakan optimum (93.3 °C, S:L 1:50, 185 minit) memperolehi 12.12% pektin dengan $AUA = 77.01\%$, $DE = 18.99\%$, kelikatan intrinsik = 148.74 mL/g dan berat molekul = 42.12 kDa. Gel yang paling teguh telah diperolehi pada 3% (w/v) pektin dan pH 3 dan gel tersebut mempunyai keteguhan yang lebih baik daripada LMP komersial. Gel ini juga menunjukkan kestabilan haba yang

baik sepanjang pemanasan dan penyejukan berterusan. Oleh itu, kulit durian merupakan sumber LMP yang berpotensi dan boleh dipakai sebagai agen pembentuk gel dan pemekat produk makanan dan farmaseutik yang berciri asid lemah-rendah kalori. Kajian masa depan boleh dilakukan untuk meneroka aplikasi pektin kulit durian dalam industri farmaseutikal dan makanan.



PTTA UTHM
PERPUSTAKAAN TUNKU TUN AMINAH

TABLE OF CONTENTS

TITLE	i
DECLARATION	ii
DEDICATION	iii
ACKNOWLEDGEMENT	iv
ABSTRACT	v
ABSTRAK	vi
TABLE OF CONTENTS	viii
LIST OF TABLES	xiv
LIST OF FIGURES	xvii
LIST OF SYMBOLS AND ABBREVIATIONS	xxi
LIST OF APPENDICES	xxiv
LIST OF PUBLICATIONS	xxvi
LIST OF AWARDS	xxvii
CHAPTER 1 INTRODUCTION	1
1.1 Research background	1
1.2 Problem statement	3
1.3 Significance of study	5
1.4 Research objectives	6
1.5 Scope of study	6
1.6 Limitation of study	7
CHAPTER 2 LITERATURE REVIEW	8
2.1 Overview	8
2.2 Durian	8
2.2.1 Malaysian durian production, distribution and market demand	11
2.2.2 Durian rind and its impact to environment	13

2.2.3 Durian rind utilization as a potential source of pectin	15
2.3 Pectin	16
2.3.1 Pectin chemical structure	17
2.3.2 Commercial pectin and its classification	19
2.3.3 Commercial pectin application	20
2.3.4 Market demand of pectin	22
2.3.5 Pectin source: Durian rind as a potential source of pectin	23
2.4 Pectin production	24
2.4.1 Raw material pre-treatment	24
2.4.2 Pectin extraction	26
2.4.3 Key performance indicators of pectin extraction	29
2.4.4 Factors affecting pectin extraction	30
2.4.5 Research gap of durian rind pectin extraction	39
2.4.6 Screening of acid type and concentration using balanced factorial design	41
2.4.7 Optimization of pectin extraction parameters using response surface methodology (RSM)	42
2.4.8 Precipitation and purification of pectin	43
2.4.9 Drying of pectin	44
2.5 Physicochemical and rheological properties characterization of pectin	44
2.5.1 Degree of esterification and structural analysis by Fourier transform infrared spectroscopy (FT-IR)	45
2.5.2 Anhydrouronic acid content analysis by colorimetric assay	47
2.5.3 Intrinsic viscosity and viscosity-average molecular weight	48

2.5.4 Thermal analysis by thermogravimetric analysis (TGA)	50
2.5.5 Morphological analysis by scanning electron microscopy (SEM)	51
2.5.6 Rheological analysis	52
CHAPTER 3 METHODOLOGY	53
3.1 Overview	53
3.2 Research methodology layout	53
3.2.1 Raw material	55
3.3 List of chemicals and ingredients	57
3.4 Apparatus and instruments	57
3.5 Raw material preparation	58
3.5.1 Collection of durian rinds	58
3.5.2 Drying of durian rinds	58
3.5.3 Alcohol insoluble residue preparation	60
3.6 Screening of durian rind parts for pectin extraction	61
3.6.1 Pectin extraction	61
3.6.2 Pectin yield determination	61
3.6.3 Equivalent weight determination	62
3.6.4 Methoxyl content determination	62
3.6.5 Anhydrouronic acid content determination	62
3.6.6 Degree of esterification determination	63
3.6.7 Statistical analysis of pectin extraction by durian rind parts	63
3.7 Screening of durian rind varieties for pectin extraction	63
3.7.1 Pectin extraction	64
3.7.2 Pectin yield determination	64
3.7.3 Degree of esterification determination	64
3.7.4 Anhydrouronic acid content determination	65

3.7.5 Statistical analysis of pectin extraction by durian rind varieties	66
3.8 Screening of acid type and concentration for pectin extraction	66
3.8.1 Experimental design of pectin extraction based on the acid type and concentration	66
3.8.2 Pectin extraction	67
3.8.3 Pectin yield determination	67
3.8.4 Degree of esterification determination	67
3.8.5 Anhydrouronic acid content determination	68
3.8.6 Statistical analysis of pectin extraction based on the acid type and concentration	68
3.9 Optimization of pectin extraction by response surface methodology	68
3.9.1 Experimental design of pectin extraction optimization	68
3.9.2 Pectin extraction	69
3.9.3 Pectin yield determination	70
3.9.4 Degree of esterification determination	70
3.9.5 Anhydrouronic acid content determination	70
3.9.6 Statistical analysis of pectin extraction parameters	71
3.9.7 Optimization of pectin extraction parameters	71
3.9.8 Validation of the model	72
3.10 Physicochemical and rheological properties characterization of pectin	72
3.10.1 Pectin extraction	73
3.10.2 Physicochemical properties characterization	73
3.10.3 Rheological properties characterization	76

3.10.4 Statistical analysis of pectin's physicochemical and rheological properties	78
CHAPTER 4 RESULTS & DISCUSSIONS	79
4.1 Overview	79
4.2 Effect of durian rind parts on durian rind pectin yield, purity, and degree of esterification	79
4.2.1 Effect of durian rind parts on pectin yield	79
4.2.2 Effect of durian rind parts on pectin equivalent weight	80
4.2.3 Effect of durian rind parts on pectin purity	80
4.2.4 Effect of durian rind parts on pectin methoxyl content and degree of esterification	81
4.2.5 Screening of durian rind parts based on pectin yield, purity, and degree of esterification	81
4.3 Effect of durian rind varieties on durian rind pectin yield, purity, and degree of esterification	82
4.3.1 Effect of durian rind varieties on pectin yield	82
4.3.2 Effect of durian rind varieties on pectin purity	83
4.3.3 Effect of durian rind varieties on pectin degree of esterification	83
4.3.4 Screening of durian rind varieties based on pectin yield, purity, and degree of esterification	85
4.4 Effect of acid type and concentration on durian rind pectin yield, purity, and degree of esterification	85

4.4.1 Effect of acid type and concentration on pectin yield	86
4.4.2 Effect of acid type and concentration on pectin purity	89
4.4.3 Effect of acid type and concentration on pectin degree of esterification	92
4.4.4 Screening of acid type and concentration based on pectin yield, purity, and degree of esterification	94
4.5 Optimization of extraction parameters of durian rind pectin using response surface methodology	98
4.5.1 Statistical analysis and model evaluation	98
4.5.2 Effect of extraction parameters on pectin yield	102
4.5.3 Effect of extraction parameters on pectin purity	106
4.5.4 Optimization of the experiment and validation of the model	110
4.6 Physicochemical and rheological characterization of durian rind pectin	111
4.6.1 Physicochemical properties of durian rind pectin	112
4.6.2 Rheological properties of durian rind pectin	120
CHAPTER 5 CONCLUSION	139
5.1 Conclusion	139
5.2 Recommendations for future studies	141
REFERENCES	143
APPENDICES	172
VITA	184

LIST OF TABLES

2.1	Botanical classification of durian (Manzoor <i>et al.</i> , 2020)	9
2.2	Malaysian durian production volume from 2010 to 2020	12
2.3	Summary table of durian rind's potential utilization	16
2.4	Pectin application in different industries	20
2.5	Food application of HMP and LMP	21
2.6	Food Hydrocolloid Market 2020 (Seisun & Zalesny, 2021)	22
2.7	Pectin yield and type of commercial and other feasible sources for pectin production	23
2.8	Comparison between alkaline, chelating agent and acid pectin extraction	27
2.9	Summary table of the yield, DE, and purity of pectin based on plants' parts	31
2.10	Summary table of the yield, DE, and purity of pectin based on plants' varieties	32
2.11	Advantages and drawbacks of mineral and organic acid pectin extraction	34
2.12	Summary of the investigated effect of acid extraction parameters on yield, purity of DE of pectin	36
2.13	Extraction parameters of durian rind pectin extraction from previous studies	40
2.14	The range of extraction parameters of durian rind pectin and their justifications	40
2.15	Chemical formula, chemical structure and pKa based on acid type (Comuzzo & Battistutta, 2019; Papagianni, 2007; Solomons <i>et al.</i> , 2016)	41
2.16	Frequencies and intensities of functional groups from the FT-IR spectra of pectin (Gnanasambandam & Proctor, 2000)	45

2.17	The parameters for flow behavior and viscoelastic properties analysis of durian rind pectin solutions and gels in the current study	52
3.1	Experimental range and level of each variable	69
3.2	Experimental design for pectin extraction based on the central composite rotatable design	70
4.1	Yield and chemical properties of pectin	81
4.2	Yield, AUA content, and DE of pectin extracted from different durian rind varieties	85
4.3	Extraction parameters and responses of the central composite design for durian rind pectin extraction using 0.001M H ₂ SO ₄	99
4.4	Fit statistics of regression models of yield and AUA	100
4.5	Effect of extraction parameters on pectin yield, analysed by ANOVA of the yield linear model	103
4.6	Influence of the main effects and interaction effects of extraction parameters on pectin AUA content (%), analyzed by ANOVA of the AUA quadratic model	107
4.7	Predicted and experimental value at optimum extraction condition (93.3 °C, 1:50, 185 min)	110
4.8	Chemical properties of durian pectin and commercial low methoxyl pectin	112
4.9	Intrinsic viscosity and viscosity-average molecular weight of durian pectin and commercial low methoxyl pectin	113
4.10	Quantitative parameters from TGA curves of DP and CLMP	119
4.11	Linear viscoelastic region (LVR) of DP gels at different concentrations (1 – 3% w/v) and pH (2 – 6) with 30% sucrose (w/v) and 50 mg Ca ²⁺ /g pectin measured at 6.283 rad/s and temperature 25 °C	128
4.12	Power-law constants for the relationship between storage modulus (G') and frequency (ω) of DP at different concentrations	131
4.13	Power-law constants for the relationship between storage modulus (G') and frequency (ω) of DP at different pH	135

- 4.14 Power-law constants for the relationship between storage modulus (G') and frequency (ω) of DP and CLMP gels prepared at 3% (w/v) pectin concentration, pH 3, 30% sucrose (w/v) and 50 mg Ca^{2+} /g pectin 137



PTTA UTHM
PERPUSTAKAAN TUNKU TUN AMINAH

LIST OF FIGURES

2.1	Morphological structure of durian fruit, Musang King (D197), weighing about 2 kg (photo taken by the author, 14 August 2022)	10
2.2	Cross-section of a half-cut durian fruit (about 6 to 7 weeks) after successful pollination and fertilization (Chung, 2017)	10
2.3	Statistics of durian exports in Malaysia from 2002 – 2019 (in USD'000 and metric tonnes (Mt)) (Safari <i>et al.</i> , 2021)	13
2.4	Structure of a durian rind with three layers: exocarp (thorns), mesocarp, and endocarp (photo taken by the author, 14 August 2022)	14
2.5	Primary plant cell wall structure (Dranca & Oroian, 2018)	17
2.6	Schematic structure of pectin (Zdunek <i>et al.</i> , 2021)	18
2.7	Chemical structure of pectin: (a) A repeating segment of pectin molecules and different functional groups of pectin units: (b) carboxyl; (c) ester; (d) amide (Sriamornsak, 2003)	19
2.8	General pectin production process	24
2.9	Illustration of a) porous spherical solid phase and b) mass transfer mechanism of the hydrolysis and transport of the pectin to the particle surface (Minkov <i>et al.</i> , 1996)	28
2.10	Central composite rotatable design (CCRD) for three factors	42
2.11	FT-IR spectra of pectin at varying DE (Gnanasambandam & Proctor, 2000)	46
2.12	Absorbance change over time of MHDP-GalA-H ₂ SO ₄ solution	48
2.13	a) Estimation of intrinsic viscosity for scleroglucan in water by Huggin's and Kraemer's equations (Lapasin & Pricl, 1995) b) Concentric cylinder of rheometer (Anton Paar)	49

2.14	TGA curves of pectin from sugar beet pulp (Combo <i>et al.</i> , 2013)	50
2.15	Examples of typical pectin SEM images: a) commercial HMP, b) pectin from alkaline demethoxylation, c) pectin from acidic demethoxylation, and d) pectin from enzymatic demethoxylation (Einhorn-Stoll, 2018)	51
3.1	Flowchart of pectin extraction process	54
3.2	Layout of research methodology	56
3.3	a) Cross section of durian rind; b) Chunks of durian rinds with thorns attached (TDR); c) Chunks of the white part of durian rinds, without thorns (WDR)	59
3.4	Summary of AIR preparation	60
3.5	Overview of rheological and physicochemical properties of pectin characterization	72
4.1	Effect of durian rind varieties on pectin yield, AUA content, and DE	84
4.2	Effect of acid type and concentration on the yield (%) of pectin	95
4.3	Effect of acid type and concentration on pectin purity, represented by AUA content (%) with $\geq 65\%$ considered pure (primary y-axis, bar) and pectin yield (%) (secondary y-axis, line)	96
4.4	Effect of acid type and concentration on pectin DE (%)	97
4.5	Model adequacy graphs of developed models: Actual vs. predicted plots of a) yield and b) AUA; Normal plots of residuals of c) yield and d) AUA; Residuals vs. run plots of e) yield and f) AUA	101
4.6	One-factor plots of yield (%): Effect of a) temperature at 150 min and S:L 1:40, b) S:L at 85 °C and 150 min, c) time at 85 °C and S:L 1:40; d) perturbation plot on pectin yield (%)	103
4.7	3D response surface plots of yield (%): Effect of a) temperature and S:L at 150 min, b) temperature and time at S:L 1:40, c) S:L and time at 85 °C. Color gradients indicate the level of optimization (red = high, green = intermediate, and blue = low)	105

4.8	One factor plot of AUA (%): Effect of a) temperature at 150 min and S:L 1:40, b) S:L at 85 °C and 150 min, c) time at 85 °C and S:L 1:40; d) perturbation plot on pectin yield (%)	108
4.9	3D response surface plots of AUA (%): Effect of a) temperature and S:L at 150 min; b) temperature and time at S:L 1:40; c) S:L and time at 85 °C. Color gradients indicate the level of optimization (red = high, green = intermediate, and blue = low)	109
4.10	Appearances of a) durian pectin powder and b) commercial low methoxyl pectin powder	111
4.11	a) Huggin's, b) Martin's, and c) Kraemer's plots of DP and CLMP for intrinsic viscosity determination	114
4.12	FT-IR spectra of DP and CLMP, showing the chemical groups presented in the pectins	116
4.13	Morphological structures of DP and CLMP, captured by SEM at $\times 200$ magnification	117
4.14	Thermogravimetric analysis (TGA) curves of DP and CLMP	119
4.15	Viscosity-shear rate profiles of a) DP and b) CLMP solutions at different concentrations (1 – 3% w/v) ($T = 25^\circ\text{C}$), and c) Effect of concentration on the viscosities of DP and CLMP solutions ($T = 25^\circ\text{C}$, shear rate = 10 s^{-1})	122
4.16	Viscosity-shear rate profiles of 1% (w/v) a) DP and b) CLMP solutions at different pH (2 – 6) ($T = 25^\circ\text{C}$), and c) Effect of pH on the viscosities of 1% (w/v) DP and CLMP solutions ($T = 25^\circ\text{C}$, shear rate = 10 s^{-1})	124
4.17	Effect of temperature on the viscosities of a) DP and b) CLMP solutions at different concentrations (1 – 3% w/v)	125
4.18	Effect of temperature on the viscosities of 1% (w/v) a) DP and b) CLMP solutions at different pH (2 – 6)	126
4.19	Amplitude sweep studies of DP gels at different a) concentrations (1 – 3% w/v) and b) pH (2 – 6) with 30% sucrose (w/v) and 50 mg Ca^{2+}/g pectin measured at 6.283 rad/s and 25°C	129

4.20	Appearances of DP gels prepared at different concentrations (1 – 3 % w/v), pH 3, 50 mg Ca ²⁺ /g pectin and 30% sucrose (w/v)	130
4.21	a) Frequency sweep studies of DP at different concentrations (1 – 3% w/v), pH 3, 50 mg Ca ²⁺ /g pectin and 30% sucrose (w/v); b) Viscoelastic properties at 6.283 rad/s as a function of concentration	132
4.22	Appearances of 3% (w/v) DP gels prepared at different pH (2 – 6), 30% sucrose (w/v) and 50 mg Ca ²⁺ /g pectin	133
4.23	a) Frequency sweep studies of 3% w/v DP at different pH (2 – 6), 50 mg Ca ²⁺ /g pectin, 30% sucrose (w/v); b) Viscoelastic properties at 6.283 rad/s as a function of pH	135
4.24	Appearances of DP and CLMP gels prepared at 3% (w/v) pectin concentration, pH 3, 30% sucrose (w/v) and 50 mg Ca ²⁺ /g pectin	136
4.25	Frequency sweep studies of DP and CLMP gels at 3% (w/v) concentration, pH 3, 50 mg Ca ²⁺ /g pectin and 30% sucrose (w/v)	137
4.26	Temperature ramp studies (heating and cooling: 5 – 95 °C) of DP and CLMP gels prepared at 3% (w/v), pH 3, 50 mg Ca ²⁺ /g pectin and 30% sucrose (w/v)	138

LIST OF SYMBOLS AND ABBREVIATIONS

%	-	Percentage
°C	-	Celsius
AA	-	Acetic acid
ADI	-	Acceptance daily intake
AGA	-	Apiogalacturonan
AIR	-	Alcohol insoluble reside
ANOVA	-	Analysis of variance
AUA	-	Anhydrouronic acid
CA	-	Citric acid
Ca ²⁺	-	Calcium ion
CAGR	-	Compound annual growth rate
CCRD	-	Central composite rotatable design
CeDS	-	Centre for Diploma Studies
CLMP	-	Commerical low methoxyl pectin
D	-	Desirability function
DE	-	Degree of esterification
DOA	-	Department of Agriculture of Malaysia
DP	-	Durian rind pectin
DTG _{max}	-	Maximum decomposition rate temperature
EtOH	-	Ethanol
FAST	-	Faculty of Applied Sciences and Technology
FCC	-	Food Chemical Codex 1996
FT-IR	-	Fourier Transform Infrared spectroscopy
FTK	-	Faculty of Engineering Technology
g	-	gram
G'	-	Storage modulus
G''	-	Loss modulus

GalA	-	Galacturonic acid
GRAS	-	Generally recognized as safe
GTMP	-	Green Technology Master Plan
H ₂ SO ₄	-	Sulphuric acid
H ₃ PO ₄	-	Phosphoric acid
HCl	-	Hydrochloric acid
HG	-	Homogalacturonan
HMP	-	High methoxyl pectin
HNO ₃	-	Nitric acid
INS	-	International Numbering System
IV	-	Intrinsic viscosity
LA	-	Lactic acid
LMP	-	Low methoxyl pectin
LVR	-	Linear viscoelastic region
M	-	Molarity
mg	-	milligram
MHDP	-	Metahydroxydiphenyl
min	-	Minutes
Mt	-	Metric tonnes
MW	-	Molecular weight
N	-	Normality
NaOH	-	Sodium hydroxide
pKa	-	Acid dissociation constant
rad/s	-	Radian per second
RM	-	Ringgit Malaysia
RG1	-	Rhamnogalacturonan I
RGII	-	Rhamnogalacturonan II
Rha	-	Rhamnose
RSM	-	Response surface methodology
s ⁻¹	-	Per second
S:L	-	Solid to liquid ratio
SEM	-	Scanning electron microscopy
T _{50%}	-	Temperature at which 50% mass loss

TA	-	Tartaric acid
TGA	-	Thermogravimetric analysis
USD	-	United States Dollar
UTHM	-	Universiti Tun Hussein Onn Malaysia
VMW	-	Viscosity-average molecular weight
w/w	-	Weight/weight
w/v	-	Weight/volume
XGA	-	Xylogalacturonan



PTTA UTHM
PERPUSTAKAAN TUNKU TUN AMINAH

LIST OF APPENDICES

APPENDIX	TITLE	PAGE
A	Standard curve of galacturonic acid (0 – 80 µg/mL) against absorbance 520 nm	172
B	Yield (%) of acid-extracted pectin from durian rinds using different acid types and concentrations	173
C	Influence of the main effect and interaction effect of acid type and concentration on pectin yield (%), analysed by two-way ANOVA with significant effect considered at $p<0.05$	174
D	pH value of extractants according to acid types at different concentrations	175
E	Tukey pairwise comparisons of pectin yield (%) according to acid types	176
F	AUA content (%) of acid-extracted pectin from durian rinds using different acid types and concentrations	177
G	Influence of the main effect and interaction effect of acid type and concentration on pectin purity (AUA content %), analysed by two-way ANOVA with significant effect considered at $p<0.05$	178
H	Tukey pairwise comparisons of AUA content (%) according to acid types	179
I	DE (%) of acid-extracted pectin from durian rinds using different acid types and concentrations	180
J	Influence of the main effect and interaction effect of acid type and concentration on pectin DE (%), analysed by two-way ANOVA with significant effect considered at $p<0.05$	181

K	Tukey pairwise comparisons of DE (%) according to acid types	182
L	Product specification of commercial low methoxyl pectin	183



PTTA UTHM
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VITA

Jong Sze Hui was born in Kuching, Sarawak, Malaysia on 3rd September 1993. She attended Universiti Tun Hussein Onn Malaysia (UTHM) in 2013 and graduated with a bachelor's degree in Science (Food Technology) with honours in 2017 with a good CGPA. In the same year, she was offered a fast-track course for a Doctor of Philosophy degree in Science (research mode) by the same university. Her research interest falls in the fields of biopolymer and rheology, which has led her to her current research topic regarding the fundamental studies of pectin extraction and characterization of physicochemical and rheological properties. Throughout her doctorate studies, she had involved, assisted, and participated in a few other research, for example, the development of starch-based edible coating using modified tapioca starch for mushroom shelf-life extension and the product development of a healthy snack, bitter gourd pudding. The fields covered were food science, food preservation, food processing, and food product development. She was also a teaching assistant for the laboratory works of food technology-related subjects in UTHM, for example, Food Technology Unit Process, Food Engineering, Food Processing Technology, and Physical Properties of Food.