

OPTIMIZATION AND CHARACTERIZATION OF PECTIN EXTRACTED
FROM SWEET POTATO RESIDUE

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ABSTRACT

Pectin has been used widely as thickener, stabilizer and gelling agent. However, the source of pectin industrially are still limited. This study aims to optimize the extraction of pectin from sweet potato residue using hydrochloric acid and analyses the properties of the pectin. In this study, the extraction of pectin from sweet potato residues using hydrochloric acid was optimized to maximise its yield and quality using response surface methodology (RSM). Then, the extracted pectin was further analyse for its degree of methylesterification (DM) using Fourier Transform Infrared Spectroscopy (FTIR). In this study, 0.5 %w/v, 1.0% w/v and 2.5% w/v concentration of pectin sugar solution were used to study their rheological properties. The antioxidant activity was assessed using DPPH radical scavenging activity. The optimum condition to extract pectin from sweet potato residues was at extraction temperature (60 °C), pH (1) and extraction time (60 minutes) yielding 24.4 % pectin using hydrochloric acid. The pectin extracted has 57.48% of DM which indicated that it is high methoxyl pectin and able to form gel with the addition of sugar. The rheological properties analysis shows that 0.5% w/v pectin sugar solution shows Newtonian fluid behaviour while 1.0% w/v sugar pectin solution and 2.5% w/v pectin sugar solution showed pseudoplastic fluid behaviour. Moreover, the pectin was show 25.5 to 37.7% of radical scavenging activity.

ABSTRAK

Pektin telah digunakan secara meluas sebagai pemekat, penstabil dan ejen gelling. Walau bagaimanapun, sumber pektin dalam perindustrian masih terhad. Kajian ini bertujuan untuk mengoptimumkan pengeluaran pektin daripada sisa keledak menggunakan asid hidroklorik dan menganalisis sifat-sifat pektin. Dalam kajian ini, pengekstrakan pektin dari sisa keledak menggunakan asid hidroklorik telah dioptimumkan untuk memaksimumkan kaedah gerak balas permukaan hasil dan menggunakan kaedah (RSM). Kemudian, pektin yang diekstrak dianalisis dengan lebih lanjut untuk mengetahui darjah pengesteran (DM) menggunakan Fourier Transform Infrared Spektroskopi (FTIR). Dalam kajian ini, 0.5% w / v, 1.0% w / v dan 2.5% w / v kepekatan larutan gula pektin telah digunakan untuk mengkaji sifat-sifat reologi. Aktiviti antioksidan dinilai menggunakan aktiviti memerangkap radikal DPPH. Keadaan optimum untuk mengekstrak pektin dari sisa keledak adalah pada suhu pengekstrakan (60 °C), pH (1) dan masa pengekstrakan (60 minit) menghasilkan 24.4% pektin menggunakan asid hidroklorik. Pektin yang telah diekstrak mempunyai 57,48% daripada DM yang menunjukkan bahawa ia adalah pektin bermetoksil tinggi dan dapat membentuk gel dengan penambahan gula. Analisis sifat-sifat reologi menunjukkan bahawa 0.5% w / v larutan gula pektin menunjukkan kelakuan bendalir Newtonian manakala 1.0% w / v larutan gula pektin dan 2.5% w / v larutan gula pektin menunjukkan kelakuan bendalir pseudoplastic. Selain itu, pektin itu menunjukkan 25,5-37,7% daripada aktiviti memerangkap radikal.

TABLE OF CONTENT

CHAPTER	TITLE	PAGE
	DECLARATION	ii
	DEDICATION	iii
	ACKNOWLEDGEMENTS	iv
	ABSTRACT	v
	ABSTRAK	vi
	TABLE OF CONTENT	vii
	LIST OF TABLES	xi
	LIST OF FIGURES	xii
	LIST OF ABBREVIATIONS	xiv
1	INTRODUCTION	1
	1.1 Background of Study	1
	1.2 Problem Statement	3
	1.3 Objectives	4
	1.4 Scope of Study	5
2	LITERATURE REVIEW	6
	2.1 Overview of Sweet Potato	6
	2.2 Pectin	10
	2.3 Structure of Pectin	11
	2.3.1 Gel Formation of Pectin	13
	2.3.2 Application of Pectin in Food Industry	15

2.3.3	Pectin Potential in Pharmaceutical and Healthcare	16
2.3.4	Antioxidant Properties of Pectin	17
2.4	Extraction of Pectin	18
2.4.1	Methods of Pectin Extraction	18
2.4.2	Factors Affecting Yield of Extraction	22
2.4.2.1	Solvent pH	22
2.4.2.2	Extraction Temperature	23
2.4.2.3	Extraction Time	23
2.5	Characterization Properties of Pectin	23
2.5.1	The Degree of Methyl Esterification of Pectin	24
2.5.2	Rheological Properties of Pectin	25
2.6	Response Surface Methodology	26
3	RESEARCH METHODOLOGY	28
3.1	Introduction	28
3.2	Materials and Methods	28
3.3	Pre-treatment of Sweet Potato Residue	30
3.4	Analysis of Sweet Potato Residue	30
3.4.1	Moisture Content	31
3.4.2	Carbohydrate Content	32
3.4.3	Ash Analysis	32
3.5	Extraction of Pectin	32

32	3.5.1	Preparation of Cell Wall Material	32
	3.5.2	Extraction of Pectin	33
	3.6	Design of Experiment	34
	3.6.1	Optimization of Pectin Extraction from Sweet Potato Residue	34
	3.7	Analysis of Pectin	36
	3.7.1	Degree of Methylesterification	36
	3.7.2	Rheological Properties of Pectin	36
	3.7.3	Antioxidant Properties of Pectin	37
4		RESULT AND DISCUSSION	38
	4.1	Introduction	38
	4.2	Analysis of Sweet Potato Residue	38
	4.3	Optimization of Pectin Extraction	39
	4.3.1	Model Fitting	39
	4.3.2	Optimization using Design Expert	39
	4.3.3	Main Effects and Interaction between Parameters	42
	4.3.4	Model Verification	44
	4.4	Analysis of Pectin	46
	4.4.1	Degree of Methyl Esterification	47
	4.4.2	Rheological Properties of Pectin	49
	4.4.3	Antioxidant Properties of Pectin	52

5	CONCLUSION	53
	5.1 Introduction	53
	5.2 Conclusion	53
	5.3 Recommendation for Future Work	54
	REFERENCE	55-60

LIST OF TABLES

TABLE NO	TITLE	PAGE
2.1	Some of sweet potato varieties in Malaysia	7
2.2	Composition of pectin in different fruits and vegetables.	11
2.3	Common extraction technique in pectin extraction.	21
3.1	Condition of extraction.	33
3.2	Independent Variables and levels used in the response surface design	34
3.3	Actual Experimental Design	34
4.1	Analysis of sweet potato residue.	38
4.2	ANOVA for pectin yield	40
4.3	Coefficients of determination (R^2), adjusted R^2 values, probability values (p) and lack-of-fit values for dependent variable	41
4.4	Pectin yield under different extraction condition.	42
4.5	Actual and predicted value of pectin yield.	46
4.6	Actual and predicted value of response of optimized condition	46
4.7	Degree of Methyl esterification of pectin sample.	48
4.8	Value of parameters obtained for pectin sugar solution	50
4.9	DPPH scavenging activities of extracted pectin	51

LIST OF FIGURES

FIGURE NO	TITLE	PAGE
2.1	Graph of sweet potato production in Malaysia per year.	8
2.2	Sweet potato residues	8
2.3	Pectin in plant cell wall.	10
2.4	Primary structure of pectic substances	12
2.5	Structure of pectin	12
2.6	Schematic representation of pectin structural elements.	14
2.7	Covalent cross link by calcium bridges	15
2.8	Graph of shear stress versus shear rate.	25
3.1	Sweet potato variety (Serdang 1).	28
3.2	Work flow of the study.	29
3.3	The sweet potato residues.	30
4.1	Graph of predicted values versus actual values of response (pectin yield).	40
4.2	Response surface plots for the effect of temperature and pH at constant time of 60 minutes.	43
4.3	Response surface plots for the effect of extraction temperature and extraction time at constant pH of 2.	44
4.4	Response surface plots for the effect of pH of and extraction time at constant temperature of 75°C.	45
4.5	FTIR spectra of pectin sample.	48
4.6	Graph of $\ln(\text{shear stress})$ versus $\ln(\text{shear rate})$ of 0.5% w/v Pectin Sugar Solution	49
4.7	Graph of $\ln(\text{shear stress})$ versus $\ln(\text{shear rate})$ of 1.0% w/v Pectin Sugar Solution	50

4.8	Graph of $\ln(\text{shear stress})$ versus $\ln(\text{shear rate})$ of 2.5% w/v Pectin Sugar Solution	50
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LIST OF ABBREVIATION

DM	-	Degree of Methyl Esterification
FT-IR	-	Fourier-transform Infrared Spectroscopy
HM	-	High Methoxyl
LM	-	Low Methoxyl
DPPH	-	1,1-diphenyl-2-picrylhydrazyl
RSM	-	Response Surface Methodology
CHE	-	Conventional Heating Extraction
MAE	-	Microwave assisted Heating Extraction
UAHE	-	Ultrasonic-assisted Heating Extraction

CHAPTER 1

INTRODUCTION

1.1 Background of Study

Sweet potato, *Ipomoea batatas* L. (Lam.), a dicotyledonous plant that belongs to the family Convolvulaceae contains high dietary fibers, minerals, vitamin and antioxidant such as phenolic acid and carotene. It is also categorised as short term food crops. They are staple food crop in some developing countries. They are grown widely in tropical and subtropical region. Sweet potato also serves as animal feed and as raw product for industrial purposes. Moreover, sweet potato ranked as one of the most important crops in developing countries besides rice, wheat, maize and cassava (Hue & Low, 2015).

Pectin are natural hydrocolloids that are found in higher plants as main structural elements of cell walls. Basically, they are complex polysaccharides containing 1,4- linked- α -D galacturonic acid residues. Neutral sugar units are attached to the backbone and concentrated in highly branched “hairy” regions. Part of the carboxylic groups in the galacturonic chain exists in methyl ester form, and the degree of methylation (DM) divides pectin into two types. They are high methoxyl pectin (HM) and low methoxyl (LM) pectin. In the high-methoxyl (HM) form, more than 50% of the carboxyl groups are methylated, meanwhile in the low-methoxyl (LM) form less than 50% are methylated. The degree of methylation is very important for the gel formation of pectin.

Pectin is mainly used in food processing industry as gelling and thickening agents, emulsifiers as well as stabilizers in jams, jellies, confectionery products, and beverages. Other than that, pectin is also used in pharmaceutical and cosmetic industry as natural prophylactic, as binding agent in tablet formulations and as carrier of a variety of drugs. Pectin is also a component of soluble dietary fibres and plays a significant role in many physiological processes as it is poorly digested in the small intestine but ferments in the colon. For now, the source of commercial pectin are apple pomace and citrus peel, and to a certain extent sugar beet roots and sunflowers. However, there is a requirement for a novel source of pectin to manufacture pectin with tailored structures and beneficial properties, particularly adaptable for biomedical application. Therefore, the isolation of pectin from a cheap and abundant renewable resource, such as agricultural and food waste is a concern, moreover improve waste management and create another prospect of income to the economy.

The manufacture of pectin typically comprises extraction, purification, and drying. The use of a suitable method for pectin extraction is significant to maximize its yield and quality. Pectin can be extracted using various techniques. The most frequently used techniques are: i) solvent extraction by stirring and heating, ii) heat refluxing extraction and iii) microwave heating extraction. In solvent extraction of pectin by stirring and heating, different type of solvent was used. In this experiment, hydrochloric acid (HCl) was used as the solvent. The yield and properties of pectin usually depends on the raw materials used to extract pectin and the extraction conditions, such as temperature, extraction time, pH, and type of extraction solvents (Yeoh, Shi, & Langrish, 2008).

Traditionally, an optimization process has been conducted by monitoring the influencing of one factor at a time. However, the pectin extraction is a physical-chemical process in which hydrolysis and extraction of pectin macromolecules from plant tissue, and their solubilization take place continuously under the influence of different factors, mainly temperature, pH, and time. As such, the influence of these parameters, not only the single one but also the interaction between them, on the optimal extraction condition is necessary to be established. In order to provide a

solution to this problem, the response surface methodology was consequently chosen as the tool to determine and to solve multivariable equations for optimizing the extraction processes of pectin from the sweet potato residues. The main objective of this study was to determine effects of the pectin extraction parameters (pH, temperature, and time) on their responses (yield). Such relationships finally led to an optimum condition for pectin extraction from sweet potato residue. In addition, the optimized sweet potato residue pectin was analysed for its degree of methylesterification, rheological properties and antioxidant properties.

1.2 Problem Statement

Sweet potatoes are mainly used to produce starch and starchy foods. Other than that, sweet potato also is being consumed directly. Thus, considerable quantity of residues is generated. Some of these residues are used as animal feed and the majorities of these residues are directly thrown out and consequently pollute the environment. The peels are mostly considered as waste materials. They are produced from the sweet potato processing industry and are normally discarded. Environmental problem, particularly these discarded peels of sweet potatoes can cause water pollution. Thus, in addition to being fed to animals, the peels can be utilized to produce pectin, which would then increase the potential return for the sweet potato processing industry.

Pectin is a complex polysaccharide that makes up about one third of the cell wall of higher plant. Pectin are found to be at its highest concentration in the middle lamella of cell wall. At present, commercial pectin are derived from citrus peel and apple pomace. They are by product from juice manufacturing. Apple pomace contains about 10- 15% of pectin on dry matter basis. Meanwhile, citrus peels contain relatively higher 20- 30% of pectin. Sugar beet waste from sugar manufacturing and sunflower head are alternatives sources of pectin. On the other hand, it is reported that sweet potato residues contain about 15 percent of pectin on its dry matter basis (Mei *et al.*, 2010). Hence, sweet potato residue can be used as an alternative source of pectin.

The molecular size and degree of esterification (DE) of pectin play a vital role in the ability of pectin to form gel. Different sources of pectin do not necessarily have the same gelling ability due to variations in numerous parameters. Thus, the number of sources for commercial pectin manufacture is limited even though it occurs in most plant tissues (Srivastava & Malviya, 2011). In this study, the DE of pectin is identified using Fourier transform infrared (FTIR).

When many factors and interactions affect desired response, response surface methodology (RSM) is an effective tool for optimizing the process, which was originally described by Box and Wilson (1951). RSM is a collection of statistical and mathematical techniques that has been successfully used for developing, improving and optimizing processes. The main advantage of RSM is the reduced number of experimental trials needed to evaluate multiple parameters and their interactions. Therefore, it is less laborious and time-consuming than other approaches required to optimize a process (Giovanni, 1983). Response surface methodology has been successfully used to model and optimize the extraction of pectin from apple pomace (Wang *et al.*, 2007), lemon byproduct (Masmoudi *et al.*, 2008), passion fruit peel (Kliemann *et al.*, 2009a), dragon fruit peel (Thirugnanasambandham *et al.*, 2014), , grapefruit peel (Wang *et al.*, 2015) and banana peels (Oliveira *et al.*, 2016).

1.3 Objectives

This study embarks on the following objective:

- 1 To optimise the effect of acid, pH, temperature, and extraction time on yield of pectin from sweet potato residues (*Ipomoea batatas* var. Serdang 1) residues using response surface methodology.
- 2 To characterise the degree of methylesterification (DM), rheological properties and the antioxidant properties of pectin from sweet potato residues.

1.4 Scope of Study

- 1 Optimization of pectin yield extraction from sweet potato residue at different condition of pH (1-3), temperature (60- 90 °C) and time (30-90 minutes) using 0.1 M Hydrochloric acid (HCl) by Response Surface Methodology (RSM).
- 2 Determination of the degree of methyl esterification of extracted pectin using Fourier Transform Infrared (FTIR) Spectroscopy.
- 3 Analysing the rheological properties of the extracted pectin using viscometer at 0.5 % w/v, 1.0% w/v and 2.5% w/v concentration of pectin sugar solution.
- 4 Evaluation of antioxidant properties of pectin yield using 1,1-diphenyl-2-picrylhydrazyl (DPPH) analysis.

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