OPTIMIZATION OF MICROWAVE-ASSISTED EXTRACTION OF PECTIN FROM PINEAPPLE PEEL

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ABSTRACT

Pineapple peel has the potential to become one of the sources for pectin production due to its high pectin content in its dietary fibre composition. Pectin has been widely used in food industry as food thickener, stabilizer, emulsifier and gelling agent. The conventional extraction process with long operating hours at high temperature has been identified to cause thermal degradation of pectin molecules. Microwave technology application in pectin extraction is able to expedite the extraction process and produce higher yield. Therefore, this study was conducted to optimize the microwave-assisted extraction (MAE) process of pineapple peel pectin. The extracted pectin from optimal condition was subjected to physical characterization and chemical and physicochemical properties evaluation and it was also compared to the commercial pectin. Preliminary studies on the best solvent for pectin extraction from pineapple peel showed that hot solvent sulphuric acid resulted in higher recovery in pectin yield. The optimization of MAE of pectin from pineapple peel was performed using Box-behnken design under the response surface method. The optimum operating conditions obtained were pH 1.52 of solvent, 1:10 (w/v) of solid to liquid ratio and 600 W of microwave power with 2.43 ± 0.03 % (w/w) of pectin yield and 54.61 ± 0.21 % (w/w) of anhydrouronic acid (AUA) content. Under these optimal conditions, the degree of esterification (DE) of pectin was 63.93 ± 0.30 % (w/w). The flow behaviour of pineapple peel pectin solutions at low concentration exhibited the Newtonian behaviour whilst at higher concentration it exhibited the pseudo-plastic behaviour. The elevation of temperature caused the pectin solution viscosity to decrease. The emulsifying activity of extracted pectin was 45 % and the emulsion prepared from pineapple peel pectin has high stability at 4 °C with 93.75 % and 88.24 % at 25 °C, after 30 days of storage. The Fourier transform infrared spectrum showed that the extracted pineapple peel pectin contained the same functional groups that exist in the commercial pectin. Through this study, it can be proven that extraction by using MAE could provide higher quantity and better quality of pectin from pineapple peel. Moreover, pectin produced using pineapple peel has comparable DE value, flow behaviour of pectin solution and emulsifying properties, to the commercial pectin. Hence, pineapple peel can be a potential source for pectin production with some extended studies in sample pre-treatment and pectin purification process.

ABSTRAK

Kulit nanas mempunyai potensi menjadi salah satu sumber kepada penghasilan pektin kerana kandungan pektin yang tinggi dalam komposisi serat dietnya. Pektin telah digunakan dengan meluas dalam industri makanan sebagai pemekat, penstabil, pengemulsi dan agen penggelan makanan. Proses pengekstrakan konvensional dengan tempoh masa yang panjang pada suhu yang tinggi telah dikenal pasti menyebabkan degradasi haba dalam molekul pektin. Aplikasi teknologi gelombangmikro dalam pengekstrakan pektin menjadikan proses ini lebih cepat serta menghasilkan ekstrak yang lebih tinggi. Oleh itu, kajian ini dijalankan untuk mengoptimumkan pengekstrakan berbantu gelombangmikro (MAE) dalam pengekstrakan pektin daripada kulit nanas. Pektin yang terhasil daripada pengekstrakan yang optimum telah menjalani pencirian fizikal dan penilaian sifat kimia dan fizikokimia, dan potensinya telah dibandingkan dengan pektin komersial. Kajian awal terhadap pelarut terbaik dalam pengekstrakan pektin daripada kulit nanas menunjukkan bahawa larutan asid sulfurik panas memberikan hasil perolehan pektin yang lebih tinggi. Pengoptimuman proses pengekstrakan berbantu MAE pektin daripada kulit nanas telah dilaksanakan dengan menggunakan kaedah gerak balas permukaan menerusi reka bentuk Box-benhnken. Keadaan operasi pengekstrakan optimum yang diperolehi ialah pH 1.52 pelarut, 1:10 (w/v) nisbah pepejal-cecair dan 600 W kuasa gelombangmikro dengan sebanyak 2.43 ± 0.03 % (w/w) hasil pektin dan 54.61 ± 0.21 % (w/w) kandungan asid anhidrouronik (AUA). Pada keadaan optimum, darjah pengesteran (DE) pektin ialah 63.93 ± 0.30 % (w/w). Kelakuan aliran larutan pektin kulit nanas pada kepekatan rendah menunjukkan sifat Newtonian manakala pada kepekatan tinggi ia menunjukkan sifat pseudo-plastik. Peningkatan suhu telah menyebabkan pengurangan pada kelikatan larutan pektin. Aktiviti emulsi pektin yang diekstrak ialah 45 % dan emulsi yang dihasilkan daripada pektin kulit nanas mempunyai kestabilan yang tinggi iaitu 93.75 % pada suhu 4 °C dan 88.24 % pada suhu 25 °C, selepas 30 hari penyimpanan. Spektrum inframerah jelmaan Fourier menunjukkan bahawa pektin kulit nanas mempunyai kumpulan berfungsi yang sama yang terkandung dalam pektin komersial. Kajian ini membuktikan bahawa pengekstrakan dengan menggunakan MAE boleh menghasilkan pektin kulit nanas dalam kuantiti yang lebih banyak dan kualiti yang lebih baik. Tambahan pula, pektin kulit nanas mempunyai kualiti setanding dengan pektin komersial dari segi nilai DE, kelakuan aliran larutan pektin dan sifat-sifat pengemulsi. Oleh itu, kulit nanas berpotensi menjadi sumber penghasilan pektin dengan beberapa kajian lanjutan dalam pra-rawatan sampel dan proses penulenan pektin.

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LIST OF ABBREVIATIONS

ANOVA	-	Analysis of Variance
AUA	-	Anhydrouronic Acid
BBD	-	Box-Behnken Design
CCD	-	Central Composite Design
CP	-	Commercial Pectin
DE	-	Degree of Esterification
FTIR	-	Fourier Transform Infrared
GalA	-	Galacturonic Acid
HCl	-	Hydrochloric Acid
H_2SO_4	-	Sulphuric Acid
HMP	-	High Methoxyl Pectin
LMP	-	Low Methoxyl Pectin
MAE	-	Microwave-Assisted Extraction
MeO	-	Methoxyl
MW	-	Molecular Weight
NaCl	-	Sodium Chloride
NaOH	-	Sodium Hydroxide
O/W	-	Oil-in-Water
PPP	-	Pineapple Peel Pectin
RSM	-	Response Surface Methodology
S/L	-	Solid to Liquid
UAE	-	Ultrasound Assisted Extraction

LIST OF SYMBOLS

α	-	Alpha
β	-	Beta
°C	-	Degree Celcius
%	-	Percentage
>	-	More than
<	-	Less than
cm	-	Centimeter
g	-	Gram
g/mL	-	Gram per mililitre
mg	-	Miligram
mm	-	Milimeter
mL	-	Mililitre
min	-	Minute
N	-	Normality
pH	-	Power of hydrogen
R^2	-	Determination coefficient
rpm	-	Revolutions per minute
s	-	Second
w/w	-	Weight per weight
w/v	-	Weight per volume
W	-	Watt power

CHAPTER 1

INTRODUCTION

1.1 Background of Study

Pineapple is one of the tropical fruit that can be considered as the most important in the world market and become the top three most preferable fruit juices after orange and apple (Upadhyay *et al.*, 2010). Pineapple can be consumed in various way either by raw itself, cooked in meal or processed into jam or juices. The exportation of pineapple from Malaysia mainly involved fresh fruit or canned product. According to the statistical data by Food and Agriculture Organization (FAO), Malaysia has ranked the 8th place in Asia for the greatest pineapple producer after Philippines, Thailand, India, Indonesia, China, Vietnam and Taiwan (FAO, 2017). The pineapple industry shows exportation of canned pineapple increase enormously with 8,474.90 tons metric per year in 2014 to 8,853.37 tons metric per year in 2015 (LPNM, 2016). Demand of pineapple in worldwide also has shown a good growth of production from 2013 to 2014 with 24.5 million tons per year to 25.4 million tons per year of production (FAO, 2017).

Activity from pineapple industry has led to the huge waste production. Wastes from pineapple usually accounted 50 % (w/w) from the whole fruit mass which 29-40 % (w/w) came from peel, 9-10 % (w/w) from core, 2-5 % (w/w) from stem and 2-4 % (w/w) from crown (Ketnawa *et al.*, 2012). This waste has potential to become a renewable resources due to its high biodegradable content. The peel itself can be a promising resources since it is the largest proportion of the waste. According to Ketnawa *et al.* (2012), other than bromelain extraction, the peel could be utilized for further industrial application of its fiber. On the other hand, according to Huang *et al.* (2011), the fiber-rich fractions in pineapple peel potential to be exploited for food functional ingredients. The fiber-rich fractions were said to exhibit great water- and oil- holding capacity, swelling properties and cation-exchange capacities that can modify the food texture, stabilize foods with high percentage of fat, and destabilize, entrap, and disintegrate the micelles formed by emulsion of lipid (Huang *et al.,* 2011).

Previous studies have been reported that pineapple peel can be a potential source for pectin (Ukiwe and Alinnor, 2011; Karim *et al.*, 2014). Pectin is a complex polysaccharides that made up from monomers of D-galacturonic acid joined by α-(1-4) glycosidic linkages (Schols and Voragen, 1996). The galacturonic acid (GalA) or anhydrouronic acid (AUA) content in pectin was suggested to be not less than 65 % to be specified as pectin (FAO, 2007). Pectin can be divided into two main groups according to its degree of esterification (DE) which were high-methoxyl pectin (HMP) (DE > 50 %) and low-methoxyl pectin (LMP) (DE < 50 %) which dependent on the source of raw material and method of extraction (Thakur *et al.,* 1997; Wang *et al.,* 2016). Commercial pectin usually produced from either citrus peel or apple pomace. Citrus peel and apple pomace produce different types of pectin which respectively are HMP and LMP that make them preferable to specific applications (May, 1990). Different types of pectin also has different method preparations for gel formation.

Pectin in food industry uses as food thickener, stabilizer, emulsifier and gelling agent (May, 1990). The addition of pectin polysaccharide into foods and beverages also help to improve the mouthfeel and increase the satiety, thus can reduce the consumption of foods and beverages (Thakur *et al.,* 1997). Pectin usually added in fruit jams, confectionary jellies, fruit juices and dairy products. In pharmaceutical industry, pectin has been reported able to lower the cholesterol levels in blood, treat diarrheal disease and duodenal ulcers extensively, and remove mercury and lead from respiratory organs and gastrointestinal tracts effectively. The application of pectin in tablet formulations can act as a binding agent and delayed the drug delivery (Sriamornsak, 2003).

Pectin usually obtained from two steps production which were acid extraction of raw material and isolation of pectin from the extracted solution with alcohol precipitation or aluminum salts (May, 1990). Commercially, the acid extraction use hot dilute mineral acid such as nitric acid, hydrochloric acid and sulphuric acid at pH around 1-3 with various length of time. There are also some studies reported the uses of organic acid such citric acid and salt such as ammonium oxalate as extracting solvents in order to improve the pectin yield and physicochemical properties of pectin (Mohd. Ismail *et al.,* 2012). The alternative citric acid extractors have been said to carry better in economic value and more environmental friendly (Canteri-Schemin *et al.*, 2005).

The traditional extraction method can be time consuming that took around 6 to 10 hours in soxhlet method and 60 to 120 minutes in hot dilute acid extraction. These method has less efficiency and produce limited yield of pectin. Excessive time of extraction somehow can lead to pectin degradation (Wang *et al.*, 2016). Therefore, new technologies of microwave-assisted extraction (MAE) and ultrasound-assisted extraction (UAE) have been introduced earlier in pectin extraction in order to increase the pectin yield and its quality (Maran *et al.,* 2014; Wang *et al.*, 2016). The MAE technique is a powerful technique and has potential to replace the existing conventional method since it has the ability to extract samples with shorter time of extraction, less solvent consumption and higher rate of extraction with low cost equipment due to its localized heat mechanism (Mandal *et al.*, 2007; Maran *et al.*, 2013). MAE also offered protection to thermolabile constituents and can extract the targeted bioactive compound from raw materials efficiently (Mandal *et al.*, 2007). UAE however required significant longer time of extraction compared to MAE and has less pectin recovery of pectin extraction from dragon fruit peel (Rahmati *et al.*, 2015; Chua *et al.*, 2018). One of the shortcoming in MAE is the loss of active compounds due to thermal degradation from the utilization of high microwave irradiation. However, this problem can be prevented with intermittent microwave extraction. Intermittent extractions can keep out samples from being overheated by balancing the heat and mass transfer besides enhance the efficacy of extraction (Swamy and Muthukumarappan, 2017).

This study was aimed to optimize the pectin extracted from pineapple peel by using MAE method. The extraction method can influence the pectin quality. Hence, characterization of optimum extract from MAE were performed to evaluate the chemical and physicochemical properties of the extract. Pectin extracted at optimum condition was subjected to determination of AUA and DE value and Fourier Transform Infrared Spectroscopy (FTIR) to compare the chemical composition of extracted pectin with commercial pectin. On the other hand, determination of emulsifying properties and viscosity measurement to prove the ability of the extract to serve as an alternative for commercial pectin.

1.2 Problem Statement

Commercialized pectin is usually obtained either from citrus peel or apple pomace after juice extraction. According to May (1990), limited sources for potential food pectin was known due to its standard requirement to contain at least 65 % galacturonic acid of substance. Pectin has been an important food additive in food industry and its demand in world market is currently has reached approximately 45,000 tons annually with 4-5 % growing per annum (Raji *et al.*, 2017). In recent days, many studies have been done to utilize food wastes as an alternative resources for pectin production which are from mango peels (Rehman *et al.* 2004; Berardini *et al.*, 2005), passion fruit peels (Pinheiro *et al.*, 2008; Kliemann *et al.*, 2009; Liew *et al.*, 2014), dragon fruit peels (Mohd Ismail *et al.*, 2012) and jackfruit and chempedak fruit rind (Leong *et al.*, 2016).

Pineapple peel has been said to be an excellent potential in food application due to its composition high in dietary fiber which content was 42.2 % (w/w) to produce pectin (Huang *et al.*, 2011). Several studies on extracting pectin from pineapple peel have been done earlier by Ukiwe and Ainnor (2011), Karim *et al.* (2014) and Sun *et al.* (2014). However, the information of pineapple peel pectin on its purity, physicochemical, structural and functional properties are still unclear. The purity of pectin has become one of the challenges in product market after Food and Agriculture Organization (FAO) has defined the minimum galacturonic acid content required must not be less than 65 % (FAO, 2007). Meanwhile, characterization of pectin is crucial in further assessment to perform an extensive research on its rheological properties and emulsifying properties to evaluate its potential becoming a marketable product. Apart of its high availability from canned processing activity in this country, pineapple peel waste in return would provide an alternative sources for commercial pectin and thus would help to reduce the number of pollutants in our environment.

Conventionally, it took long operating time of six hours to complete the extraction process of pectin from fruit wastes by using the soxhlet method. The hot dilute mineral acid extraction via water bath on the other hand requires one to two hours of operation that can lead to pectin degradation. Longer time of extraction also would causes more energy and cost consumption of production. Various method of extraction have been studied earlier in order to improve the recovery of valuable crude extracts from the biomass wastes such as microwave-assisted extraction (MAE), ultrasonic-assisted extraction (UAE), pressurized solvent extraction (PSE) and super-critical fluid extraction (SFE). The MAE has been said can be a promising as new extraction technique. The attractive features of MAE to its ability of performance of high and fast extraction, low solvent usage and offered protection to the thermolabile compounds become preferable as an alternative to the conventional method.

Optimization of operating condition for pectin extraction by MAE was important in order to obtain the optimum pectin yield and pectin quality. The quality of pectin was according to the AUA content that referred to the purity of extracted pectin. According to Kute *et al.* (2015), several parameters were considered to be optimized in pectin extraction by MAE which were pH, solid to liquid ratio, microwave power and time. These parameters had gave significant effect on the pectin yield. The optimum condition of MAE obtained in literature by other studies however are restricted since it was limited to a specific microwave system and specific scale that can be used solely as a reference for the extraction by using microwave. This was due to the fact that different microwave system have different efficiency in microwave heating and extraction performance (Chan *et al.*, 2014). Hence, optimization of MAE is required to obtain an optimum condition based on the parameters involved.

1.3 Objectives of Research

This study aims to achieve the objectives:

1. To determine the significant parameters for pectin extracted from pineapple peel.

2. To optimize the pectin yield extracted from pineapple peel by using microwave-assisted extraction.

3. To characterize and evaluate the chemical and physicochemical properties of the extracted pineapple peel pectin obtained from the optimized pectin yield.

1.4 Scope of Study

In order to achieve objectives, the scope of this research has been identified and divided into few parts.

a) Preliminary study on best method of pectin extraction from pineapple peel was carried out by using three types of solvents which were citric acid, sulphuric acid, and ammonium oxalate. Different parameters (time, pH value, temperature, microwave power and solid to liquid ratio) were screened out in pectin extraction by using microwave-assisted extraction (MAE).

b) The optimization was performed by using Box-behnken Design (BBD) of Response Surface Methodology (RSM) for three parameters which were microwave power (400, 500 and 600 W), pH value (pH 1.5, 2.0 and 2.5) and solid to liquid ratio (1:10, 1:20 and 1:30 g/mL) in response to pectin yield and anhydrouronic acid (AUA) content.

c) The pectin obtained from the optimal extraction was analysed its characteristics which were degree of esterification (DE) value and AUA content of pectin, pectin flow behaviour, emulsifying properties of pectin and structural analysis by using Fourier Transform Infrared (FTIR). The pectin characterization was also compared to the commercial pectin in terms of DE and AUA value, flow behaviour of pectin solution, emulsifying properties and structural analysis.

1.5 Significant of Study

The data obtained from the optimized extraction of pectin from pineapple peel by using MAE would be very useful because none of pectin extraction from pineapple peel by using MAE method has been carried out to date. MAE can be a promising and alternative extraction method over conventional to improve better pectin yield and quality.

Furthermore, the data obtained from the characterization of pineapple peel pectin would have benefited to food industries, pharmaceuticals and cosmetics. The data would provide information of the pectin properties and its application in product development. In food industry for instance, type of pectin influenced the product preparation and it is crucial to understand the pectin type to ensure that the food product will be developed successfully according to the desired requirements.

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