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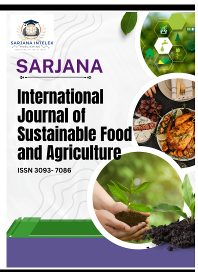

Optimization of Pectin Extraction from Pineapple Peel in Acetic Acid Solution via Microwave-Assisted Method

Muhammad Haidil Rosmin[1], Norfazilah Muhamad[1], Harisun Yaakob[2], Noorazwani Zainol[2], Siti Nor Azlina Abd Rashid[3], Joko Sulistyo[4], Nur Eleena Sadikon[5], Dayang Norulfairuz Abang Zaidel[1,3,*]

1 Department of Chemical and Environmental Engineering, Malaysia-Japan International Institute of Technology, Universiti Teknologi Malaysia, Jalan Sultan Yahya Petra, 54100 Kuala Lumpur, Malaysia
2 Institute of Bioproduct Development, Universiti Teknologi Malaysia, 83100, Skudai, Johor, Malaysia
3 Innovation Centre in Agritechnology for Advanced Bioprocessing, Universiti Teknologi Malaysia, Pagoh Campus,84600 Pagoh, Johor, Malaysia
4 Food Technology Program, Faculty of Tourism, Ciputra University, Citraland, CBD Boulevard, Surabaya 60219, Indonesia
5 Josa Pina Trading Sgn Bhd, Sunway Iskandar, Persiaran Medini 3, Bandar Sunway Iskandar Puteri, 79250 Iskandar Puteri, Johor, Malaysia

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ABSTRACT

Biowastes from food processing industries, such as pineapple peels, cause environmental issues due to their high nitrogen, phosphorus, macronutrients, and water contents. However, these biowastes also contain valuable bioproducts like pectin, which is widely used in the food industry as a thickener, emulsifier, stabilizer, and gelling agent. Conventional pectin extraction method requires prolonged processing at high temperatures, leading to thermal degradation of pectin molecules. To address this, the current study optimized pectin extraction from pineapple peels in acidic solution using microwave-assisted method. Response surface methodology (RSM) was applied to determine the optimal extractions, considering pH of acetic acid solution (2, 3, and 4), extraction time (2, 3, and 4 min), and microwave power (270, 360, and 700 W). The highest pectin yield (0.53 %) was obtained at 3 minutes of extraction, 270 W microwave power, and pH 4 of the acetic acid solution. The extracted pectin was analyzed for its degree of esterification, and its structural characteristics were compared with commercial pectin using FTIR with KBr method, revealing no significant differences. These comparable characteristics confirm that the microwave-assisted method is an efficient alternative for pectin extraction, offering a faster and more sustainable approach with potential applications in the food industry.

1. Introduction

Waste management is a key challenge in mitigating environmental degradation. Biowaste generation has increased significantly worldwide and is expected to continue rising. The food processing sector contributes heavily to this issue, producing large volumes of agricultural and food waste, primarily inedible plant parts that are rich in organic matter and water content [1,2]. Food waste valorization offers a sustainable alternative to landfilling by converting biowaste into value-

* *Corresponding author.*
E-mail address: dnorulfairuz@utm.my

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added products. This approach reduces disposal costs, minimizes environmental impact, and creates economic opportunities. Repurposing food processing waste not only mitigates pollution but also supports the transition to a circular bioeconomy [3]. Therefore, current efforts focus on harnessing biowaste as a resource, promoting its transformation into useful products for environmental remediation.

Among various value-added products, pectin is a widely explored biopolymer extracted from food biomass. It is extensively used in the food industry as a gelling agent, emulsifier, stabilizer, and thickener. Currently, commercial pectin is primarily sourced from citrus peels (85.5%) and apple pomace (14.0%). However, with market demand projected to reach $2.12 billion by 2030, alternative agro-industrial waste is gaining attention as potential pectin sources [4,5]. Notably, Hamidon *et al.,*[6] successfully extracted pectin from sweet potato peel which showed potential as an emulsifier. Similarly, Petkowicz and William [7] have used acid extraction to obtain pectin from waste broccoli stalks. The extracted pectin exhibits a high degree of methyl-esterification and low acetyl content, making it promising thickening and emulsifying agent.

Fruit byproducts often contain bioactive compounds at a higher concentration than the final product [4]. Pineapple, a globally significant fruit crop, known for its distinct flavor derived from a unique blend of sugars and organic acids. It ranks as the third most important tropical fruit worldwide, following bananas and mangoes [8]. The growing production of processed pineapple products proportionally increases pineapple waste, with the peel comprised largest portion (30-42%). Rich in cellulose, hemicellulose, lignin, and pectin, pineapple peel presents a promising pectin source [9].

Pectin extraction from food and agricultural by-products has been practiced for years. However, conventional extraction method faces several limitations, including thermal degradation, undesirable physicochemical properties, and a low degree of esterification due to prolonged direct heating. Additionally, the complexity of plant cell walls, composed of polysaccharides and structural proteins, makes pectin extraction challenging. To address these issues, researchers are refining extraction technologies. Microwave assisted extraction (MAE) offers an efficient and sustainable alternative route to pectin extraction, offering higher yields, shorter extraction times, and lower energy consumption compared to conventional extraction methods [10,11], but requires further technological advancements and optimization for commercial-scale adoption. According to Mao *et al.,* [10] only two reports exist on scale-up of MAE for pectin extraction, with no examples of industrial implementations.

A major challenge is the need for large-scale microwave reactors, which are costly and require significant investment for maintenance. Developing hybrid systems that combine MAE with conventional extraction could help reduce initial costs and optimize energy use. Another challenge is ensuring uniform extraction efficiency, as microwaves may not penetrate large volumes consistently, leading to variations in pectin yield. Implementing continuous-flow microwave reactors with optimized stirring can address this issue. Notably, Arrutia *et al.,* [12] and Garcia-Garcia *et al.,* [13] have developed semi-continuous and circulating batch systems to extract pectin from potato and orange peel, respectively. These studies demonstrate the potential of microwave assisted processes to enhance both pectin yield and quality.

While many studies have investigated MAE for pectin extraction, few have focused on optimizing the system and method. Therefore, this study aims to optimize pectin extraction from pineapple peel using response surface methodology (RSM). Traditionally, pectin extraction relies on strong inorganic acids such as hydrochloric, nitric, and sulfuric acid due to their hydrolyzing properties [14-18]. However, environmental concerns have driven interest in replacing these acids with organic alternatives. Despite this shift, research on organic acid-based extraction remains limited. To address this gap, the current study examines the feasibility of using acetic acid as an extraction solvent for pectin from pineapple waste, with potential applications in the food industry.

2. Methodology

2.1 Material

Ethanol (C_2H_5OH) (95%) and glacial acetic acid (CH_3COOH) were acquired from Chemiz. All the chemicals were used without further purification.

2.2 Sample Preparation

Pineapple waste from Josapina Sdn. Bhd, Pontian, Johor were sorted to separate the peels from the crown, core, and stem. Rotten peels were discarded maximize pectin yield. The selected peels were cut into 1 cm thick pieces, approximately 2.5 cm^2 in size, washed with potable water, and dried in a conventional oven at 60 °C for 72 hours. The dried peels were then finely ground using a food blender and sieved through a 0.60 mm stainless-steel sieve. The processed samples were then stored in an airtight container until further use [19].

2.3 Extraction of Pectin

Glacial acetic acid, as an extraction solvent, was added to the sample at 1:30 S/S ratio. The mixture was stirred homogenous, then heated in a microwave for extraction process. After extraction, the mixture was filtered using Whatman filter paper. Ethanol was added to the filtrate at a 2:1 ratio and left overnight. The mixture then was centrifuged at 3800 rpm for 20 minutes and the supernatant was discarded. The collected pectin underwent 3-4 ethanol washes to remove impurities before being dried in a conventional oven at 50 °C for 16 hours. The dried pectin yield (%) was calculated using Eq. 1 [6]. Previous studies had found that extraction time, microwave power, and pH of extraction solvent significantly affected the pectin yield. Therefore, these three variables have been selected for the current study. Pectin extraction was carried out under the extraction condition as summarized in Table 1.

$$Yield\ (\%) = \frac{M_0}{M} \times 100 \quad (1)$$

where M_0(g) is weight of dried pectin and M(g) is weight of dried pineapple peel powder.

Table 1

The design of experiments

Run	Extraction time, min	Microwave power, W	pH
1	2	360	4
2	4	360	2
3	3	270	4
4	3	360	3
5	2	270	3
6	3	360	3
7	3	700	4
8	4	270	3
9	3	270	2
10	2	700	3
11	3	360	3
12	3	360	3
13	3	360	3
14	2	360	2
15	4	360	4
16	4	700	3
17	3	700	2

2.3 Experimental Design

Response surface methodology (RSM) was used to optimize pectin extraction from pineapple peel using Design Expert Software version 6.0. The experimental design (Table 1) was structured to identify the best combinations of controls factors. Pectin yield (%) served as the response variable. The design included 17 runs, comprising 12 factorial points and five central point replicates, allowing for the estimation of pure error. All the experiments were performed randomly to minimize the effect of unexplained variability caused by systematic errors.

2.4 Model Verification

Model adequacy was evaluated using the 'Lack of Fit', the coefficient of determination (R^2), adjusted determination coefficient (Adj R^2), and F-test value from the ANOVA table as performed using Design Expert version 6.0. In the sensory window, the manipulated parameter value and the desired maximum or minimum output were selected to determine the optimal parameter of the experiment.

2.5 Characterization of Pectin
2.5.1 Physicochemical properties

The structural characteristic of extracted pectin was analyzed using Fourier Transform Infrared (FTIR) spectroscopy with the KBr method. To prepare the KBr disk, 2 mg of pectin sample was mixed with 300 mg of KBr powder and finely ground in agate mortar. Meanwhile, the KBr die was assembled with the lower pellet polished face up. The ground mixture was evenly distributed in the cylindrical bore, followed by the insertion of the second pellet and plunger. The assembled die was then placed in a hydraulic press for compression. Once formed, the KBr disk was transferred to the spectrometer holder and mounted in spectrometer. The spectrum was recorded in transmission mode over the wavenumber range of 400 to 4000 cm^{-1}.

2.5.2 Degree of esterification

The degree of esterification (DE) of pectin from pineapple peel was determined using Fourier transform infrared (FTIR) spectroscopy. DE was calculated based on the ratio of the peak intensity at approximately 1740 cm^{-1} to the sum of peaks intensities at around 1740 cm^{-1} and 1630-1600 cm^{-1} (Eq 2) [6].

$$DE(\%) = \frac{Absorbance\ peak\ at\ 1740\ cm^{-1}}{Absorbance\ at\ peak\ around\ (1740cm^{-1}+1630\ to\ \ 1600cm^{-1})} \quad (2)$$

3. Results and Discussion

3.1 Pectin Yield

The yield of extracted pectin is presented in Table 2. According the RSM, model gave the highest pectin yield (0.532) was obtained under the conditions of three minutes extraction, 270 W microwave power, and pH 4 of acetic acid solution. This result is comparable with previous work reported by Ukiwe and Alinnor [20], who extracted pectin from pineapple peel using acetic acid and obtained a yield of 0.3 %. Analysis of variance (ANOVA) was performed to determine the significance of RSM model, and the results summarized in Table 3. The P-value (>0.05) indicates that the model is significant. Based on Table 3, microwave power was identified as the most significant factor influencing pectin yield, followed by extraction time and pH. Among the quadratic terms, only A^2 (extraction time) and C^2 (pH) were significant. Additionally, the interaction term *AC* (*A*: Extraction time, *C*: pH) had the most substantial effect on pectin yield. The difference between the predicted R^2 (0.5830) and the adjusted R^2 (0.8939) exceeds 0.2, which may indicate a significant block effect or potential issues with the data or model. This suggests that model refinement, such as reduction or response transformation, should be considered. However, the 'Lack of Fit' was not significant, suggesting the model adequately fits the data.

Table 2
Pectin yield as a response to different extraction conditions

Run	Extraction time, min	Microwave power, W	pH	Yield, %
1	2	360	4.0	0.310
2	4	360	2.0	0.007
3	3	270	4.0	0.532
4	3	360	3.0	0.425
5	2	270	3.0	0.494
6	3	360	3.0	0.391
7	3	700	4.0	0.212
8	4	270	3.0	0.217
9	3	270	2.0	0.240
10	2	700	3.0	0.133
11	3	360	3.0	0.397
12	3	360	3.0	0.350
13	3	360	3.0	0.320
14	2	360	2.0	0.244
15	4	360	4.0	0.158
16	4	700	3.0	0.141
17	3	700	2.0	0.192

Table 3

ANOVA table for variance analysis

Source	Sum of Squares	df	Mean Square	F Value	P Value
Model	0.2988	9	0.0332	15.97	0.0007
A-Extraction time	0.0272	1	0.0272	13.08	0.0085
B-Power	0.0810	1	0.0810	38.98	0.0004
C-pH	0.0181	1	0.0181	8.72	0.0213
AB	0.0219	1	0.0219	10.53	0.0142
AC	0.0018	1	0.0018	0.8691	0.3822
BC	0.0128	1	0.0128	6.15	0.0423
A^2	0.0630	1	0.0630	30.30	0.0009
B^2	0.0091	1	0.0091	4.38	0.0747
C^2	0.0234	1	0.0234	11.26	0.0122
Residual	0.0145	7	0.0021		
Lack of Fit	0.0077	3	0.0026	1.49	0.3457
Pure Error	0.0069	4	0.0017		
Cor Total	0.3133	16			
R^2	0.9536				
Adj R^2	0.8939				
Predicted R^2	0.5830				
Adeq Precision	14.9518				

The difference between the predicted R^2 (0.5830) and the adjusted R^2 (0.8939) exceeds 0.2, which may indicate a significant block effect or potential issues with the data or model. This suggests that model refinement, such as reduction or response transformation, should be considered. However, the 'Lack of Fit' was not significant, suggesting the model adequately fits the data.

3.2 Diagnostic Plots

Figure 1a presents the residuals versus predicted values plot. A random scatter pattern indicates that residuals and predicted response values are distributed without any systematic trend. The graph shows that all residual values fall within the predicted outlier range of $\pm$4.81963, confirming that the pectin yield values are reasonable and valid. Figure 1b illustrates the residuals plotted against the experimental order, also represented by a random scatter. Since all data points remain within the red control limits, it can be concluded that there are no systematic errors in the model. While for Figure 1c, the plots of predicted versus the actual response values forms an approximately linear pattern, suggesting a strong correlation between the predicted and observed results. This indicates that model provide a good fit for data.

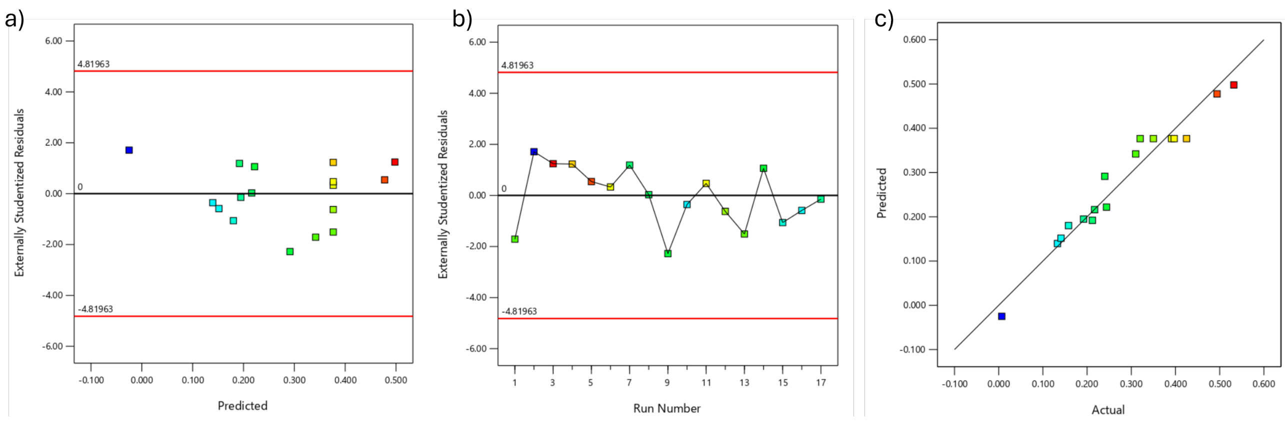

Fig. 1. Analysis of diagnostic plot

3.3. Structural Characteristics of Pectin

Figure 2 presents the FTIR spectrum of pineapple peel pectin. The broad absorption band between 3200 to 3600 cm^{-1} corresponds to O-H stretching vibration, attributed to vibrational modes of inter and intra-molecular hydrogen bonds of the galacturonic acid polymer in pectin samples [21]. The absorption peak at 2923 cm^{-1} is associated with C-H stretching vibrations, while the peaks between 1636 and 1750 cm^{-1} correspond to C=O stretching vibrations. Additionally, the signal at 1446 cm^{-1} is attributed to N-H bending motion of amines [22]. The FTIR spectrum of extracted pectin is resembles that of commercial pectin, as reported by Joel *et al*., [22], conforming the successful extraction of pectin from pineapple peel.

3.4. Degree of Esterification

Degree of esterification (DE) refers to the percentage of esterified carboxyl groups relative to the total carboxyl groups in pectin. Based on DE values, commercial pectin is typically classified into high methoxyl pectin (HMP, DE > 50%) and low methoxyl pectin (LMP, DE < 50%) [23]. The DE of pineapple peel pectin was determined using the peak intensities at around 1740 cm^{-1} corresponding to esterified carboxyl groups, and the sum of the peak intensities at around 1740 cm^{-1} and 1640-1600 cm^{-1} (Figure 2), which corresponded to the free carboxyl groups [24,25]. The calculated DE was greater than 50%, classifying pineapple peel pectin as HMP. This result aligns with previous studies that extracted HMP from watermelon rind [26], sweet potato [6], lemon and mango peels [27] and pomelo peel [28]. Different types of pectin influence gel formation mechanisms. HMP requires high sugar concentrations and low pH conditions to promote hydrophobic interaction between methoxyl groups and cross-linking homogalacturan by hydrogen bonding, facilitating gel formation [29]. Typically, pectin extraction using water, mineral acids or bases results in HMP.

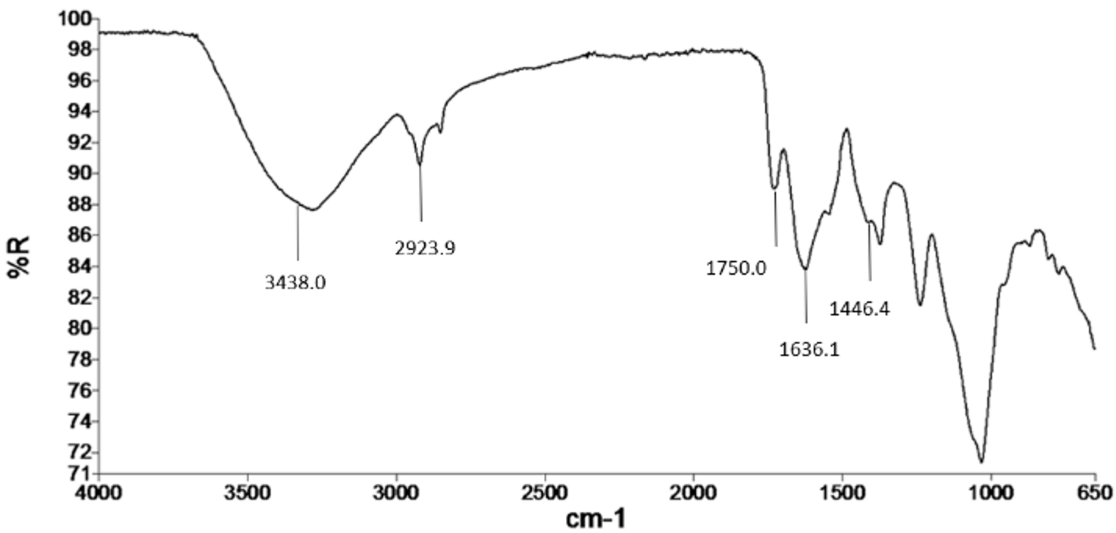

Fig. 2. FTIR spectrum of extracted pineapple peel pectin

4. Conclusions

In conclusion, this study successfully optimized the extraction process of pineapple peel pectin using acetic acid and MAE while characterizing its physicochemical properties. Response surface methodology is a great tool to use for the optimization of pectin extraction conditions. The optimal parameters were determined as a 3-minute extraction time, 270 W microwave power, pH 4 using acetic acid, yielding 0.532%. The extracted pectin showed that its molecular weight distribution, esterification degree, and gel forming ability were comparable to commercial pectin. The findings demonstrate that pineapple peel, an agricultural by-product, can be effectively utilized to produce high quality pectin, offering economic and environmental benefits. The research provides a promising foundation for industrial pectin extraction and further developments in the field.

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