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Optimization of Pectin Production Parameters from Pineapple Waste

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ABSTRACT

The objective of this study was to optimise the extraction parameters for enhancing the yield of pectin production from pineapple waste. The production of pectin from the pineapple waste was optimised with parameters such as time(120-240s), microwave power (270 W, 360 W, and 700 W), and pH 2-4 to achieve the optimal pectin yield. The Minitab Taguchi approach was used to optimise the parameter to get a high yield of pectin. Additionally, FTIR analysis was utilised to evaluate the quality of the product. The results are a significant yield of 3.7 g and a degree of esterification of 52.4% from 100 g of pineapple waste. These results may be achieved by using the ideal parameters of pH 2, 360 W, and 180 s. To summarise, this study allows for the production of large amounts of high-grade pectin from pineapple waste. The extracted pectin can be utilised in culinary and medical uses as a renewable and eco-friendly component.

1. Introduction

Pineapple, a top fruit globally, ranks third in juice preference. Malaysia, a major producer, primarily exports fresh fruit or canned products [1]. The pineapple industry was an important source of income for many small-scale farmers, and the fruit was also used as an ingredient in the food processing industry. Today, Malaysia remains a significant producer of pineapple, with the majority of production concentrated in Johor and Negeri Sembilan. The industry has seen significant growth in canned pineapple exports and overall demand. However, pineapple processing generates substantial waste, mainly from the peel, core, stem, and crown. Researchers are exploring various uses for pineapple waste, including pectin extraction for food applications. Pectin, a vital food additive, is derived from sources like citrus peel or apple pomace. Recent efforts focus on utilizing fruit waste from mangoes, passion fruit, dragon fruit, sweet potatoes, and chempedak/jackfruit for pectin production.

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Pectin finds extensive use in the food industry as a thickener, stabilizer, and emulsifier, commonly added to products like jams, jellies, fruit juices, and dairy items [2]. Additionally, studies have shown that incorporating pectin into foods and beverages can enhance mouthfeel, increase satiety, and potentially reduce consumption. In pharmaceuticals, pectin has been utilized to lower cholesterol, treat diarrheal diseases, and eliminate toxic metals from the digestive and respiratory systems. However, traditional extraction methods for pectin are time-consuming and may lead to degradation. Microwave-assisted extraction (MAE) [3] and ultrasound-assisted extraction (UAE) [4] have been introduced as efficient alternatives, with MAE showing particular promise due to its shorter extraction time, reduced solvent consumption, and higher extraction rates. Although MAE may cause thermal degradation of active compounds, intermittent microwave extraction can mitigate this issue.

Recent studies have explored various methods for extracting pectin from pineapple leaves, including conventional acidic extraction using inorganic acids like hydrochloric acid, sulfuric acid, and nitric acid, which resulted in pectin yields ranging from 0.8% to 2.4% [5]. This research focuses on extracting pectin from pineapple leaves, a byproduct of the pineapple industry, promoting waste valorization. The study employs innovative extraction methods that enhance yield and quality compared to traditional approaches. Comprehensive analysis of the physicochemical properties of the extracted pectin, providing insights into its potential applications in food and pharmaceutical industries. This study aims to investigate the effects of various factors on the MAE of pineapple peel for pectin production, focusing on irradiation time, pH, temperature, solid-to-solvent ratio, and microwave power, and characterizing the resulting pectin by its degree of esterification (DE).

Section 2 describes the extraction techniques used, including the experimental design, materials, and analytical methods for pectin characterization. Section 3 presents the findings from the extraction process, including yield percentages, physicochemical properties, and implications for practical applications. Section 4 summarizes the key findings, discusses their significance, and outlines potential avenues for further research in pectin extraction and application.

2. Methodology

2.1 Preparation of Pineapple Peel Powder

The pineapple peels were sliced into 1 cm thick slices of roughly 2.5 cm^2 size with a sharp knife. The waste was rinsed in potable water to eliminate any leftover pulp. Then the sample was placed in a conventional oven at 60 °C for 72 h. Then the dried sample was ground into powder [6].

2.2 Microwave-Assisted Extraction of Pectin

An acidic solvent was prepared by using citric acid, and the pH was adjusted to 2. Then 10 g of pineapple peel powder was dissolved in it and was heated up to 75°C at the same time the solvent was spun at 300 rpm for 30 min to break the cell wall and extract out the pectin [7].

Once the solvent completed the heat-up procedure, the solvent was sent to microwave-assisted extraction with 2 min with the microwave power of 270 W (defrost). While in the microwave heating, strong microwave radiation had absorbed by the pineapple waste and resulting in the pectin in the plant cell being released to the solvent [7]. The solvent was then centrifuged for 10 min at 3800 rpm and chilled to 25 °C. Then the supernatant part was collected and pellet part was decomposed. In order to make the pectin float, the pectin-containing supernatant was coagulated with an equivalent volume of 96% ethanol and let to stand for 24 h [8].

After 24 h the solution was centrifuged again to obtain the pure pectin in pellet form. The completed item was combined with distilled water, dried in an oven, and store at room temperature [7].

2.3 Optimisation of Pineapple Peel Pectin Yield and Degree of Esterification by using Taguchi Method

The Taguchi method's emphasis on robustness makes it particularly suitable for addressing the inherent variability in natural raw materials and the sensitivity of the pectin extraction process to temperature and pH variations. Consequently, the Taguchi Method offers a reliable approach to optimizing these crucial parameters for efficient and consistent pectin extraction as shown in the Table 1. It written the details of the three types of method used in Taguchi method [9].

The analysis of variance (ANOVA) method was employed to assess the contribution of various parameters to the extraction of pectin from pineapple waste. By examining the significance levels of each parameter, the most influential factors were identified. This statistical approach aids in determining the optimal conditions for pectin extraction, optimizing resource utilization and enhancing process efficiency [10].

Table 1
Taguchi method on three types of optimisation analysis

Type of method	Explanation
Smaller-the-better (Minimize the deviation from the target):	S/N = -10 * log10(1/n * Σ(yi - T)^2)
Larger-the-better (Maximize the mean or performance):	S/N = 10 * log10(1/n * Σyi)
Nominal-the-best (Maximize the mean and minimize the deviation):	S/N = 10 * log10(1/(n * σ^2) * Σ(yi - T)^2)

Where:
n is the number of experimental runs
yi is the observed value in the ith experimental run
T is the target value
σ is the standard deviation of the observed values
S/N=Signal-to-noise ratio

Using Design Expert (Version 6), 9 trial runs as shown in Table 2 of the sample were produced. The goal of the earlier exploratory research on screening various MAE parameters was to find any significant influences on the range of experimental conditions and their impact on the intended response. Based on the initial research, the solvent's pH (factor 1), the microwave's strength (factor 2), and the microwave's duration (factor 3) were chosen, and their responses to pectin were further optimized the yields as well as DE content. The value of microwave time microwave power and the pH value was varied from 120 to 240 s, 270 to 700 W, and pH 2 to 4.

Table 2
Pectin extraction parameter by using Taguchi method

Run	pH	Microwave power, W	Time, s
1	2	360	180
2	4	700	180
3	2	270	120
4	4	270	240

5	4	360	120
6	3	360	240
7	3	700	120
8	3	270	180
9	2	360	240

2.4 Pineapple Peel Pectin Yield

Pectin yield extracted from pineapple peel can be calculated by using the Eq. (1) below (in the form of dry basis), as W_1 represent weight after extraction and W_2 represent weight of pineapple powder used.

$$Yield\ (\%) = \frac{W_1}{W_2}\ X\ 100\ \% \tag{1}$$

2.5 Structural Analysis by Fourier Transform Infrared (FTIR) Spectroscopy

About 0.10 ± 0.01 g of dried pineapple peel pectin and commercial pectin were subjected for structural analysis by using Fourier Transform Infrared (FTIR) Spectroscopy. The pineapple peel pectin and commercial pectin were mixed and ground with the potassium bromide powder (spectroscopic grade) and pressed to form pellets for the spectra measurements. The infrared data (IR) of both pellets were recorded by using Fourier Transform Infrared Spectrometer at room temperature within the wavelengths range from 4000 to 400 cm^{-1} [11].

2.6 Degree of Esterification (DE) of Pineapple Peel Pectin

From the result of FTIR, the absorbance from wavelengths range from 4000 to 400 cm^{-1} and estimated by using Eq(2) [12] where A_{1630} and A_{1745} represent the absorbance intensity for methyl-esterified and non-methyl-esterified carboxyl groups, respectively, at 1630 and 1745 cm^{-1} [12]. Hence by using Eq. (2), the value of degree of esterification was estimated.

$$DE(\%) = \frac{A_{1745}}{A_{1745}+A_{1630}}\ x\ 100 \tag{2}$$

2.7 Proximate Analysis for Pineapple Peel Pectin

The AOAC method was utilized for assessing moisture, ash, and total protein content [13]. Total dietary fiber (TDF) was determined following AOAC method 991.43 [14]. Soxhlet method was employed to determine fat content [15]. Carbohydrate content (CHO) was calculated using the AOAC method outlined in Eq. (3) [14].

$$Carbohydrate(\%) = 100 - (ash + protein + fat + fibre) \tag{3}$$

3. Results

3.1 Screening of Solvent

Pectin yield from pineapple peels extracted with sodium bicarbonate, citric acid, and acetic acid as extraction solvents is shown in Table 3. As increase the mM the more pectin yield was obtained but for sodium bicarbonate after screening done, it was found it was not pectin. Hence citric acid was the best solution among the acetic acid and sodium bicarbonate.

Table 3
Pectin yield using different screening solvent

For 50 mM

Type of solvent	Weight of Pectin, g	Pectin Yield, %
Sodium Bicarbonate	0.14	1.4
Citric Acid	0.06	0.6
Acetic Acid	0.04	0.4

For 200 mM

Type of solvent	Weight of Pectin, g	Pectin Yield, %
Sodium Bicarbonate	0.26	2.6
Citric Acid	0.07	0.7
Acetic Acid	-	-

For 500 mM

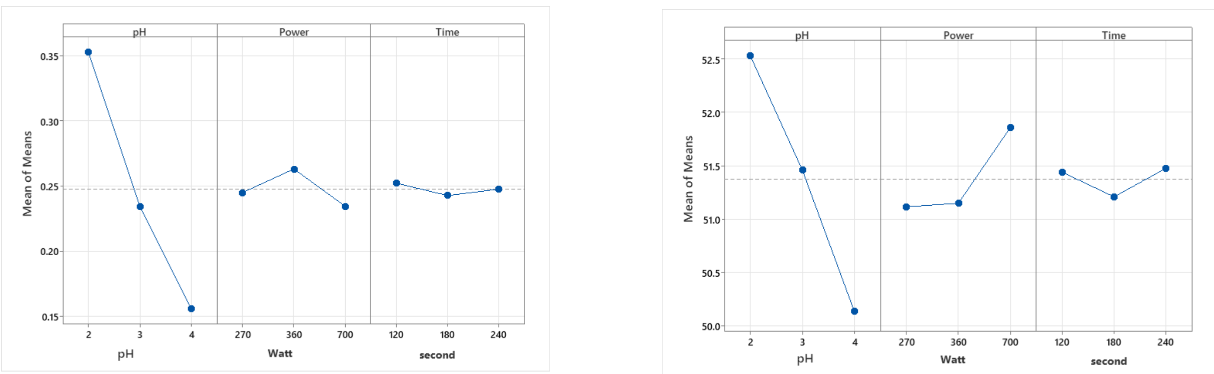
Type of solvent	Weight of Pectin, g	Pectin Yield, %
Sodium Bicarbonate	-	-
Citric Acid	-	-
Acetic Acid	0.09	0.9

For 1000 mM

Type of solvent	Weight of Pectin, g	Pectin Yield, %
Sodium Bicarbonate	-	-
Citric Acid	0.26	2.6
Acetic Acid	0.1	1.0

3.3 Factors Influencing Pineapple Peel Pectin Yield in MAE

Figure 1 indicates the signal-to-noise (S/N) ratio for yield and %DE which represents the dispersion around the intended value, where the desired value is the "signal" and the unwanted value is the "noise." The higher S/N ratio is indicative of a higher quality feature, regardless of the quality attributes. However, while optimizing multi-quality features, a higher-quality feature of one quality characteristic may have an impact on the product quality since a lower-quality feature may be needed for another quality characteristic [16].



(a) (b)

Fig. 1. (a) S/N ratio for yield (b) S/N ratio for Degree of esterification

In the investigation of the influence of microwave power and duration on pH levels, the analysis of the S/N ratio with the highest value assumes significance as it provides insights into the optimal conditions for achieving desired pH outcomes. By prioritizing the S/N ratio with the highest value, researchers can identify the combination of microwave power and duration that maximizes the quality characteristic under consideration, in this case, pH levels [17].

Initially, the experiment involves varying microwave power levels while keeping the duration constant to isolate the effect of power on pH. Subsequently, microwave duration is manipulated while maintaining a consistent power level to assess its impact on pH. The S/N ratio was calculated for each combination of pH, microwave power, and duration, reflecting the signal (yield and %DE) relative to the noise (variability) [18].

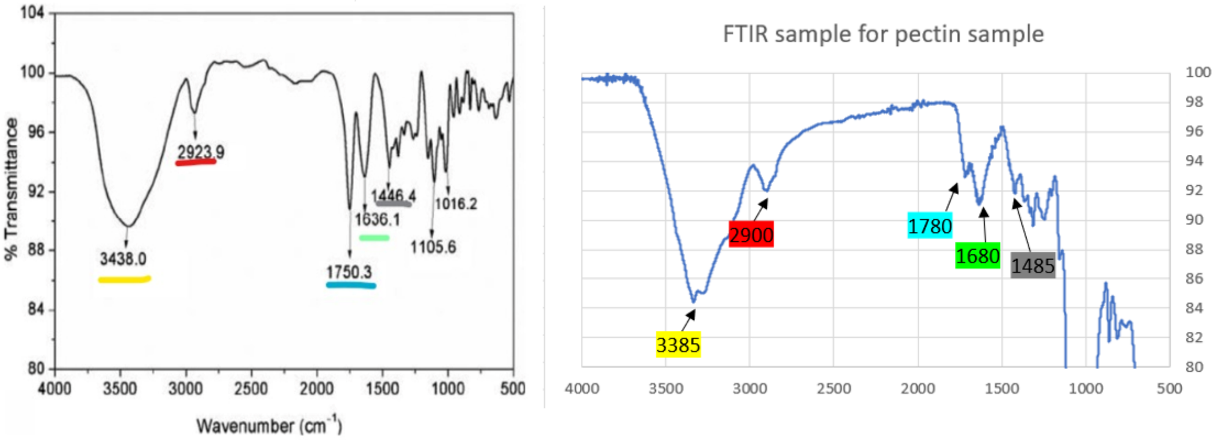
Upon analysis, if the S/N ratio exhibits its highest value in response to a specific combination of pH it indicates that this particular setting yields the most favourable pectin yield outcome. This optimal condition signifies the ideal balance between the mean pH level and its variation, thereby indicating the most efficient operating parameters for the microwave process. Furthermore, the identification of the highest S/N ratio underscores the significance of microwave power as the primary factor influencing %DE, followed by duration. This hierarchical understanding facilitates the refinement of process parameters to enhance pectin stability and reproducibility, thereby contributing to the overall quality improvement of the product or process under investigation.

3.4 Optimization of Pineapple Peel Yield and Degree of Esterification using MAE

From the Design Expert, optimal parameters obtained for extracting pectin from pineapple waste were pH 2, 360 W, and 180 s to obtain 3.7 g of pectin and 52.4% degree of esterification from 100 g of pineapple waste. Figure 2 shows the FTIR spectra of commercial pectin (CP) and pineapple waste pectin (PWP) extracted under optimized conditions (microwave power of 260 W, solvent pH of 2, and microwave time for 180 s).

The main functional groups in pectin were identified using the FTIR spectra in the 1000–2000 cm^{-1} range [7]. The pineapple waste pectin (PWP) spectrum (Figure 2) demonstrates that the stretching of the –OH group as a result of moisture adsorption in the sample occurred at the peak frequency of 3438.00 cm^{-1}. Alkane groups (CH, CH_2, and CH_3) were identified as C-H at the peak, which measured 2923.9 cm^{-1}. Around 1750.3 cm^{-1}, the peak was caused by the methyl esterified carboxyl group's CO group stretching.

The carboxylate group is assigned to two peaks, which appear to be weak asymmetrical vibrating at 1636.1 cm^{-1} and weak symmetric vibrating at 1446.4 cm^{-1} [19]. Conversely, the maximum at 1105.6 cm^{-1} and stretching in the glycosidic bond between the sugar monomers was indicated by values of 1016.2 cm^{-1}. As seen in Figure 2, the structure and spectrum of pectin derived from pineapple peel are comparable to those of commercial pectin. The DE value was defined as the carbonyl bands of free and esterified carboxyl groups with absorption of 1636.1 cm^{-1} and 1750.3 cm^{-1}, respectively. Hence in Figure 2(b), 1680 cm^{-1} and 1780 cm^{-1} shown the same chemical properties.



(a) (b)

Fig. 2. Comparison of (a) commercialized pectin and (b) pineapple peel pectin from this study

3.5 Proximate Analysis of Pineapple Peel Pectin

The variance in moisture and ash content between pineapple pectin and commercial pectin in proximate analysis can be attributed to several factors. By comparing in Table 4 pineapple peel pectin, derived from the peels and cores of pineapples, tends to retain more moisture due to its natural origin and processing methods hence giving a huge number of carbohydrate due to its origin when compare to the commercial pectin. During extraction, residual moisture from the fruit may contribute to higher moisture content in pineapple pectin compared to commercial varieties, which often undergo more extensive drying processes to achieve lower moisture levels. The moisture percentages of the pineapple waste pectin varied from 29.23% to 32.98% but there is some moisture increase due to bad packing (leakage to environment) hence the moisture content increase to 41.07% [20]. The functioning and quality of the pectin might be negatively impacted by excessive moisture, thus it's important to keep it correctly in a dry environment to avoid moisture absorption [21]. Additionally, pineapple pectin may contain a higher ash content due to the presence of minerals naturally occurring in the fruit, as well as any soil or environmental contaminants absorbed during cultivation. Commercial pectin, on the other hand, is typically purified and processed to remove impurities, resulting in lower ash content. The pectin that was acceptable generated had an ash level of 6.20 % hence it is still acceptable for 3.26% of ash content [21]. Furthermore, variations in the composition of the raw materials and processing techniques between the two types of pectin can also influence their proximate analysis profiles [22]. Overall, the disparities in moisture and ash content between pineapple pectin and commercial pectin underscore the importance of considering the source and processing of pectin products when interpreting proximate analysis data.

Table 4
Comparison of result for proximate analysis for Pineapple waste pectin and commercial pectin [23]

Parameter	Pineapple peel pectin	Commercial Pectin
Moisture	41.07±0.05	7.02±0.21
DE(%)	52.4±0.10	42.68±0.19
Ash	3.26±0.05	1.16±0.01
Carbohydrate(g/100g)	46.69±0.05	61.83±0.29

4. Conclusions

Microwave-assisted extraction (MAE) proved highly effective in extracting pectin from pineapple waste, achieving the highest yield and degree of esterification under optimized conditions. The Taguchi method was used for optimization, revealing pH as a significant factor affecting pectin yield, while microwave power and pH influenced the degree of esterification. MAE outperformed conventional methods in yield, although conventional methods showed a higher degree of esterification. In conclusion, the optimal parameters for extracting pectin from pineapple waste are pH 2, 360 W, and 180 s to obtain 3.7 g of pectin and 52.4% degree of esterification from 100 g of pineapple waste. Chemical and physicochemical analyses confirmed the quality of MAE-extracted pectin, comparable to commercial pectin, with promising attributes for various applications. Pineapple waste utilization not only yields valuable products but also addresses environmental concerns in Malaysia.

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